

**Viscon Multi Media Evaluation  
(Tier II)**

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# Multi Media Evaluation

## I. Introduction

Viscon is an additive for diesel fuel which is being proposed as a strategy for reducing PM and NOx emissions for diesel engines. The active component of Viscon is an ultra high molecular weight polyisobutylene (UHMWPIB) polymer. It is combined with CARB diesel at 1 part polymer to 99 parts diesel. Viscon is used at less than 5 ppm UHMWPIB to the end use CARB diesel. UHMWPIB is a non-toxic, colorless, tasteless, odorless food grade hydrocarbon polymer, which is insoluble in water. Lower molecular weight PIB is a component in PIB amine keep-clean additives for gasoline. UHMWPIB has been used as a method for controlling releases of diesel fuel, approved for use in California. The use of Viscon as an additive to CARB diesel creates no additional risks to the environmental or to human health when compared to unmodified CARB diesel. The use of Viscon as an additive to diesel fuel can result in a significant reduction in PM and NOx emissions from diesel engines with no significant risk to the air, water and soil environments.

Viscon should be considered by California regulatory authorities as posing no significant risk to human health or to the environment.

## II. Executive Summary

### *II-A. Summary of regulatory approvals.*

Viscon is exempt from the US EPA's requirement for registration of fuel additives under 49CFR Part 79 Subpart A Section 79.2 (e), since it is comprised of only hydrogen and carbon.

No other regulatory regimes under which Viscon may be required to be approved have been identified, either U.S. or international.

### *II-B. Background information on Viscon.*

Viscon is an additive for diesel fuel. It is used at a dose level in diesel fuel of about 500 ppm. Viscon is comprised of 1 part UHMWPIB and 99 parts CARB diesel. UHMWPIB is the active component in Viscon. It has an average molecular weight of about 7 million Daltons. Its chemical formula is C<sub>4</sub>H<sub>8</sub>. UHMWPIB is a food grade material (see Attachment 1, Food-Contact Applications).

### ***II-C. Manufacturing, Transportation and Storage of Fuel and Components***

Viscon is manufactured in Bakersfield, California at Las Palmas Oil and Dehydration. The active component of Viscon, UHMWPIB (Attachment 2, BASF's Oppanol - Polyisobutylenes) is a tough rubber solid which is delivered to the site and stored in a clean dry enclosed room in 44 pound polyethylene bags.

The manufacturing process begins by granulating the UHMWPIB to a specific size. Refer to Attachment 3, Viscon Production Flow for a complete schematic of the production steps. CARB diesel fuel is used to dissolve the UHMWPIB. The CARB diesel fuel is pre-heated to 180-185° F using a low-emissions boiler and transferred to a specially designed insulated column holding 770 gallons of fuel. The granulated UHMWPIB is then combined with the diesel. The UHMWPIB is slowly added to the top of the column, dissolved and continually mixed for 12 hours. There is no reactive chemistry involved. The finished product is transferred via pipeline to bulk holding tanks for eventual transfer to shipment vessel(s). The type of shipment vessel varies depending on customer requirements. Tanker trucks, totes, and 55-gallon drums are typical containers that are transported to customers. All liquid production areas are diked in accordance with EPA/California regulations.

Viscon is added to diesel fuel at the customer's site using equipment customarily used in their business. It is stored on the customer's site in storage tanks customarily used for storage of diesel fuel additives and petroleum products. Attachment 4, Customer-Site Storage provides examples of storage containers. All precautions normally taken for storage of diesel fuel are followed for Viscon storage.

### ***II-D. Historical Use of Viscon's Active Component***

Lower molecular weight PIB is used in a large part of gasoline consumed in the United States. It is a component in one category of keep-clean additive, PIB amines, required by State and Federal regulations. Also refer to Attachment 2, BASF's Oppanol other uses of PIB.

### ***II-E. Information Necessary for Risk Assessment***

Viscon is 1 part UHMWPIB and 99 parts CARB diesel. The diesel fuel used in Viscon poses the same risk as diesel fuel used in other applications. UHMW PIB is a solid rubber before it is added to its diesel fuel carrier. UHMWPIB does not change the chemistry of CARB diesel and does not add to its toxicity. UHMWPIB is colorless, virtually tasteless and odor free. UHMWPIB is insoluble in water. UHMWPIB does change the physical properties of the CARB diesel in which it is blended. The Viscon additive is highly viscous. Refer to Attachment 5, Viscosity Measurements for Viscon viscosity test results. Viscon also has restricted mobility in soil and in water when compared to diesel fuel. A similar combination of UHMWPIB and hydrocarbon liquids

was used as a method for controlling and collecting oil spills. UHMWPIB inhibits dispersion of the base petroleum liquid in water and can be used to immobilize a spill on land. The environmental product was called Elastol and was licensed (see Attachment 6, Issuance of License for Oil Spill Clean-Up Agent) by the State Water Resources Control Board for use on oil spills in California.

The amount of UHMWPIB added to the final diesel product, less than 5 ppm, has only a minor effect on the physical properties of diesel fuel at rest. However, it does have a significant effect on the physical properties of diesel under dynamic conditions. One well known example is the use of this polymer at similar concentrations as a drag reduction additive in pipelines. This phenomenon is viscoelasticity, an immediate and temporary increase in viscosity when the fluid is put under shear stress (see Attachment 7, Extensional Viscosity)

The anticipated use of Viscon in California is in CARB diesel. There would be no significant change in the physical, chemical or toxic properties of CARB diesel in handling, transport and storage caused by addition of less than 5 ppm of UHMWPIB with the exception of changes to CARB diesel's physical properties under conditions of shear stress.

### **III. Risk Assessment**

#### ***III-A. CARB Diesel Containing 5 ppm Viscon***

A risk assessment of CARB diesel treated with Viscon at the effective dose level, about 5 ppm UHMWPIB, would be the same as for CARB diesel without the additive, with the exception of risks associated with diesel exhaust emissions. The purpose for adding Viscon to CARB diesel is to reduce emissions of PM, NOx, HC, and CO. The results of tests conducted under CARB protocols to assess the potential benefit of Viscon as a strategy for reducing harmful exhaust emissions from diesel engines is attached. This testing includes data related to the effect of Viscon treatment on toxic emissions (see Attachment 8, CARB-Protocol Emissions Test Results).

A risk analysis of CARB diesel treated with Viscon for potential environmental and resource impacts that may result from likely release scenarios would be the same as a risk analysis of untreated CARB diesel. There are no additional risks created by addition of about 5 ppm of UHMWPIB to CARB diesel. UHMWPIB is a non toxic polymer which is insoluble in water. It is dissolved at 1 part UHMWPIB to 99 parts CARB diesel to produce Viscon. UHMWPIB is a pure hydrocarbon, food grade substance.

UHMWPIB does not change the chemical properties of CARB diesel nor does the concentration of UHMWPIB used in treating CARB diesel change CARB diesel's physical properties at rest. It does change the physical properties of CARB diesel under conditions of shear stress, causing an immediate and temporary increase in CARB

diesel's viscosity. The increase in CARB diesel's viscosity could reduce vapor loss from a release under certain conditions and could restrict travel of the fuel in soil and dispersion in water. These conclusions are based upon work done with the same polymer as an environmental technology used to reduce the risk of spills of liquid hydrocarbons.

### ***III-B. The Viscon Additive.***

The Viscon fuel additive is comprised of 1 part UHMWPIB and 99 parts CARB diesel. The risk of a potential environmental or resource impact resulting from a release of Viscon is significantly less than from a release of CARB diesel. Viscon is highly viscous.. The viscosity of Viscon increases further when put under conditions of shear stress. Penetration of Viscon in soil and transport in soil are minimal when compared to CARB diesel. Viscon does not disperse in water, and maintains its integrity on the surface of water.

UHMWPIB has been used as a method for reducing the hazard from releases of hydrocarbon liquids. The product was called Elastol Elastol was used as a fine powder which dissolved in a spill to form a substance similar to Viscon. Refer to Attachments 9, 10, & 11, Oil Spill Recovery Using Elastol, Reports 1-3. A laboratory investigation of the properties of Elastol in solution was carried out at BASF (see Attachment 12, Toxicity and Soil Penetration). UHMWPIB was also used as a 1% solution in a hydrocarbon liquid to restrict the spread, dispersion and emulsion of heavy oil.

Toxicity tests on Elastol were carried out by U.S. Testing Labs in New Jersey (see Attachment 13, Oil Dispersant Toxicity Test). Abalone Larval Development Short Term Toxicity tests were carried out in California to support a request for a license to use Elastol in California. Data from the abalone test is missing from company files, however a memorandum explaining the results of the test is Attachment 14, Abalone Larval Development Test - Memorandum. The State Water Resources Control Board issued the license on May 1, 1992 (see Attachment 6, Issuance of License for Oil Spill Clean-Up Agent). The UHMWPIB used in the Elastol product is the same as the active component of Viscon.

### ***III-C. Release Scenarios***

#### **III-C.1 CARB diesel treated with Viscon**

The release scenarios for Viscon treated CARB diesel are the same as for CARB diesel. Viscon is added to CARB diesel at a fuel distributor's site or a user's site. Once added to CARB diesel the Viscon treated fuel follows the same pathway as the CARB diesel would normally follow. As noted above any release occurring during the storage, transportation and transfer of Viscon treated CARB diesel would have the same risk

factors as for neat CARB diesel, except to the extent that added viscosity under conditions of shear stress may help mitigate the hazard from such a release.

### **III-C.2 Viscon Additive**

Viscon is produced at GTAT California's facility in Bakersfield, California. The production facility has several dissolving units each with a capacity of 770 gallons. Dissolving units and storage tanks for Viscon are contained within diked areas in accordance with US EPA and California regulations for facilities storing quantities of diesel fuel. Any accident resulting in a tank rupture or valve failure would be limited to the diked area. Vapor release to the air would be somewhat less than from a release of an equal amount of CARB diesel. The addition of the UHMWPIB to diesel limits its ability to penetrate the ground and the walls of the dike. Recovery is enhanced by the viscoelastic properties of the liquid (see Attachment 12, Toxicity and Soil Penetration).

Viscon is transported to the customer via tanker trucks, totes, and 55-gallon drums. A release resulting from an accident during transportation would have limited mobility on the ground and could easily be controlled by standard spill containment equipment. Runoff of a spill of Viscon into an adjacent waterway would be inhibited by product viscosity, resulting in a significantly reduced hazard when compared to a spill of diesel fuel.

In the event that a Viscon release found its way into a creek or river the spill would have limited ability to spread on the surface and there would be no dispersion into the body of water. Standard oil spill booms could contain the spill and the effectiveness of recovery equipment would be significantly enhanced compared to its use with a diesel spill (see Attachments 9, 10, 11, Oil Spill Recovery Using Elastol, Reports 1-3, and Attachment 12, Toxicity and Soil Penetration).

It is not anticipated that Viscon would be stored in underground storage tanks. However, if Viscon is stored underground and there is a breach in the tank due to a catastrophic event, or slow leakage or spillage around the fill port of the tank the spilled liquid would have minor mobility in the soil.

Potential air releases in manufacture of Viscon occur from the handling and storage of the CARB diesel which comprises 99% of the final product. The Viscon production facility is operated in accordance with all appropriate regulations governing the storage and handling of diesel fuel. The completed Viscon product has significantly less potential for release of vapor to the air than CARB diesel.

Air releases from Viscon during transfers and accidental releases would be reduced when compared to CARB diesel. (Attachment 9) In addition, any release in transfers or accidents is more easily contained and removed than would be the case of a similar size release of CARB diesel reducing the time the spill is open to the environment.

### ***III-D. Exposure Pathways***

#### **III-D.1 CARB diesel treated with Viscon**

The exposure pathways for CARB diesel treated with Viscon are the same as for CARB diesel, except to the extent that the addition of Viscon may reduce exposure as a result of increased viscosity under conditions of shear stress.

The polymer molecules in the Viscon treated CARB diesel are biodegradable like the fuel molecules. The pure hydrocarbon PIB molecules do not raise transformation issues when combined with CARB diesel. UHMWPIB is a non-toxic substance cleared for use in packaging exposed to food (see Attachment 1, Food-Contact Applications). Exposure to UHMWPIB does not raise chronic or acute exposure or other issues related to human exposure in any media (see Attachment 2, BASF's Oppanol - Polyisobutylenes).

#### **III-D.2 Viscon Additive**

The potential exposure pathways for Viscon in relation to both human and ecological receptors are reduced compared to those for CARB diesel. Viscon is used at a concentration of less than 500 ppm in CARB diesel. The volume of product which may be exposed to human or ecological receptors is 1/20<sup>th</sup> of 1% of CARB diesel volume, if it is assumed that all CARB diesel is treated with Viscon. The viscosity of Viscon is 57.97 Centipoise (cP) at 1,800 reciprocal seconds (D(1/s)) at 90° F compared to 2.33 Centipoise (cP) at 1,800 reciprocal seconds (D(1/s)) for CARB diesel. The higher viscosity of Viscon further limits the exposure pathways for human and ecological receptors when compared to neat CARB diesel because of reduced migration in soil, and reduced dispersion in water. Viscoelastic properties of Viscon, an immediate and temporary increase in viscosity under shear stress, further limit the spread of Viscon in an active water system such as a river or creek, where the action of the water causes the spill to consolidate and not break up and spread on the surface. The cohesiveness of Viscon also reduces potential pathways to human and ecological receptors because of the significantly increased capabilities of spill containment and recovery equipment to control and recover an accidental spill when compared to a spill of CARB diesel (see Attachment 9, Oil Spill Recovery Using Elastol, Reports 1-3 and Attachment 12, Toxicity and Soil Penetration).

### ***III-E. Risk Determination***

#### **III-E.1 Waste Management**

A release of CARB diesel treated with Viscon would respond to standard petroleum cleanup strategies and technologies the same as untreated CARB diesel. A release of

CARB diesel treated with Viscon would be no harder or easier to cleanup than CARB diesel without Viscon.

The disposal of soils contaminated with CARB diesel treated with Viscon would be handled the same as soils contaminated with CARB diesel without Viscon.

The only potential waste associated with the production of Viscon is product which does not meet product specification. This waste would be 99% CARB diesel and 1% PIB and would be handled in the same manner as CARB diesel contaminated with pure hydrocarbon substance.

Discarded Viscon or CARB diesel treated with Viscon would be subject to the same disposal regulations as discarded CARB diesel.

### **III-E.2 Risk/Benefit Assessment**

There is no significant risk to human health or to the environment caused by the production, transportation, handling, storage or use of Viscon as an additive to CARB diesel in addition to risk normally associated with the use of untreated CARB diesel. On the other hand, the use of Viscon as an additive to CARB diesel could significantly reduce the impact on human health and the environment associated with the combustion of CARB diesel in diesel engines due to demonstrated reductions in exhaust emissions of NO<sub>x</sub>, PM, CO and HC.

**Attachment 1**

**Food-Contact Applications**

OCT 27 '00 05:33PM BASF CORP

P.1/2

BASF Corporation



October 27, 2000

GTA Technologies, Inc..  
Attn. Jerry Trippe  
7400 Gallerher Road  
Gainesville, VA 20155

Fax: 703 753 9476

RE: Oppanol Approvals for Food Contact

Dear Jerry,

As per your request, please find attached the requested information on Oppanol in contact with food.

This is for informational purposes only. In case you intend to use a specific Oppanol grade in an application with food contact, I will ask Product Stewardship to issue a statement covering that particular grade.

If you have any questions or comments, please do not hesitate to call me at 973 426 2531.

Best regards,

A handwritten signature in black ink, appearing to read "Uli Eichenauer", is written over the typed name.

Uli Eichenauer  
Marketing Manager

Attachment

## **Food-Contact Applications**

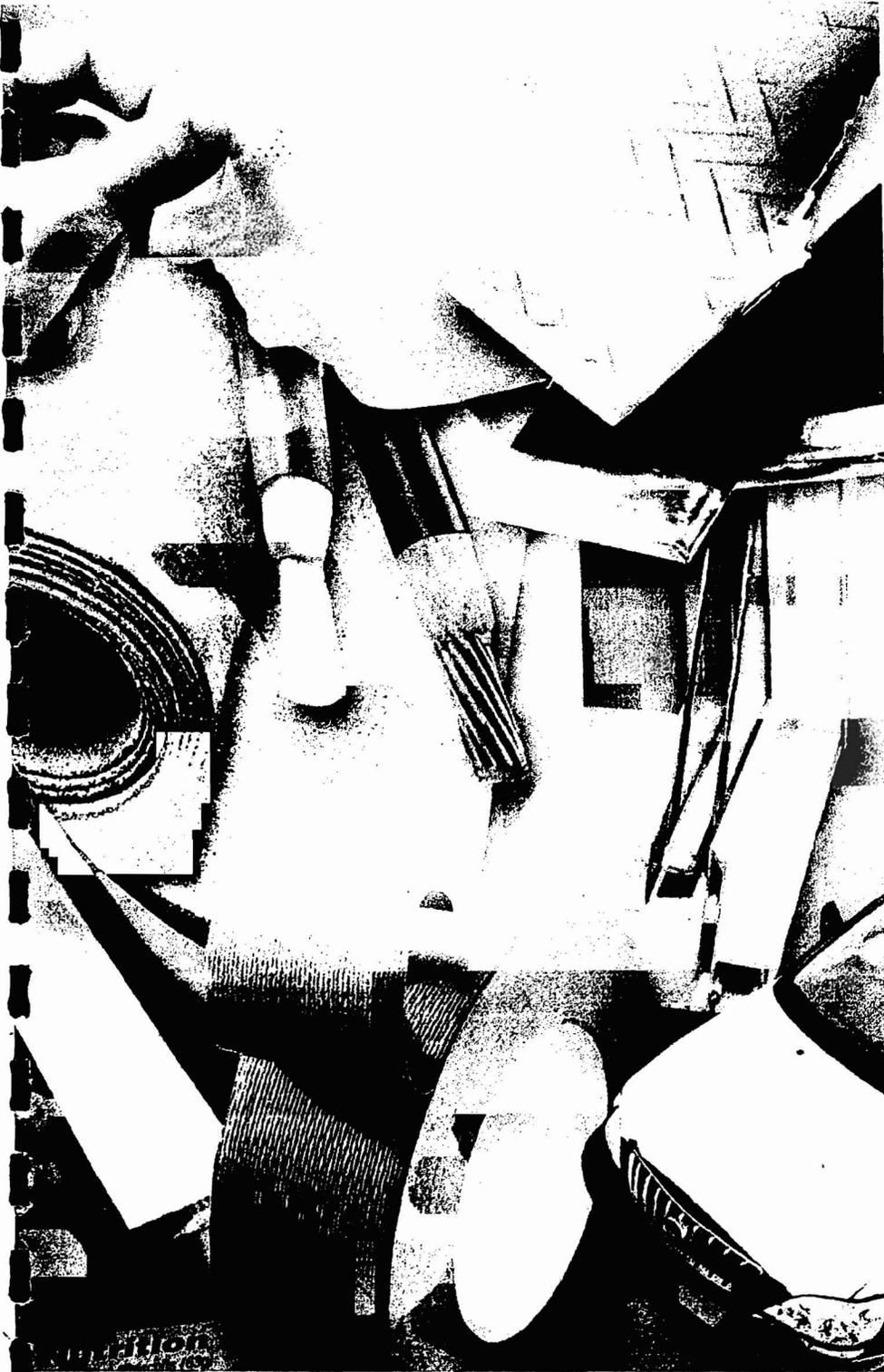
**Oppanol fulfil the requirements of:**

- **US FDA 21 CFR 172.615 Chewing gum base**
- **American Food Chemical Codex of 1996 concerning chewing gum**
- **German legislation regarding chewing gum**
- **US FDA 21 CFR 175.105 Adhesives**
- **US FDA 21 CFR 175.125 Pressure-sensitives adhesives**
- **US FDA 21 CFR 175.300 Resinous and polym. Coatings**
- **US FDA 21 CFR 176.189 Component and paperboard in contact with dry food**
- **US FDA 21 CFR 177.1420 Polyisobutene polymers (Oppanol B 100 and higher molar mass products)**
- **US FDA 21 CFR 178.3570 Lubricants with incidental food contact**
- **US FDA 21 CFR 178.3910 Surface lubricant used in the manufacture of metallic articles**
- **German Health authorities recommendation on polyisobutene(Empfehlung XX, Polybuten, 167. Mitteilung Bundesgesundheitsblatt 27, 289, 1984)**
- **German regulation concerning use for toys or for consumer articles that come into contact with food (Lebensmittel-und Bedarfsgegenständegesetz, Paragraph 5, No.1 and No 5)**

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**Attachment 2**

**BASF's Oppanol - Polyisobutylenes**



**BASF INVENTED PIB. NOW WE'VE MADE IT BETTER.**

**PIB TO HANDLE IT**

**HELPING MAKE PRODUCTS BETTER™**

**BASF'S OPPANOL®**

**POLYISOBUTYLENES**

**BASF**

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## 1. Portfolio

Oppanol® products are classified into two groups according to molecular weight:

### **MM-Oppanol:**

Medium molecular weight (Mv 40,000 - 85,000)

### **HM-Oppanol:**

High molecular weight (Mv 20,0000 - 4,000,000)

### **Products**

#### **MM-Oppanol:**

B 10 SFN, B 12 SFN, B 13 SFN, B 15 SFN

#### **HM-Oppanol:**

B 30 SF, B 50, B 50 SF, B 80, B 100, B 150, B 200

## 2. Chemical Nature

The Oppanol types consist of polyisobutylenes with different molar masses. They are mainly used in the production of adhesives, sealants, lubricants, coatings and chewing gum.

## 3. General Information

### 3.1. Properties

#### **Purity**

Oppanol is pure polyisobutylene with an ash content of less than 100 ppm and a heavy metal content of less than 3 mg/kg (except for iron < 10 mg/kg).

#### **Food-Contact Applications**

The Oppanol polymers fulfill the requirements of German legislation regarding chewing gum and the regulations concerning chewing gum contained in the American Food Chemical Codex of 1996 and FDA Paragraph 21 CFR 172.615 "Chewing gum base".

In addition the use of Oppanol products is governed by the following FDA regulations:

*21 CFR 175.105*  
Adhesives

*21 CFR 175.125*  
Pressure-sensitive adhesives

*21 CFR 175.300*

Resinous and polymeric coatings

*21 CFR 176.180*

Components of paper and paperboard in contact with dry food

*21 CFR 177.1420*

Polyisobutylene polymers (Oppanol B 100 and products with a higher molar mass)

*21 CFR 178.3570*

Lubricants with incidental food contact

*21 CFR 178.3910*

Surface lubricants used in the manufacture of metallic articles

## Appearance, Color, Odor

Oppanol is virtually colorless and very tacky. Its tack decreases substantially with increasing molar mass. Oppanol is virtually tasteless and odor-free.

## Water Resistance

Oppanol is fully resistant to water, including boiling water. It is completely insoluble in water.

## Permeability to Gases and Water Vapor

Oppanol has very low permeability to gases such as Argon, water or aqueous solutions. Oppanol is resistant to the following substances at room temperature:

- ☒ Diluted and concentrated acids (hydrochloric acid, sulfuric acid, phosphoric acid, chlorosulphonic acid, phenolsulphonic acid, formic acid, acetic acid)
- ☒ Diluted and concentrated ammonia
- ☒ Diluted and concentrated solutions of sodium hydroxide and potassium hydroxide
- ☒ Aqueous quicklime, aqueous hydrogen sulfite, copper sulfate solution, hydrogen peroxide, potassium permanganate solution, chromic acid, potassium dichromate solution

Oppanol has adequate resistance to:

- ☒ Concentrated nitric acid and mixed acid

Oppanol is not resistant to:

- ☒ Chlorine and bromine in liquid, aqueous or gaseous form

## Solvent Resistance

Oppanol is insoluble in:

- ☒ Methanol, ethanol, ethylene glycol, glycerin

- ☒ Acetone, cyclohexanone

- ☒ Methyl acetate, ethyl acetate

Oppanol is solvated by:

- ☒ Ethyl ether

- ☒ Butyl acetate

- ☒ Oils and fats of animal and vegetable origin

Oppanol is soluble in:

- Paraffin, mineral oil (gasoline, diesel oil, lubricating oil, bitumen)

- ☒ Benzene, toluene, xylene, cyclohexane, poly- $\alpha$ -olefins, oil soluble synthetic esters

- ☒ Methylene chloride, tetra chloromethane, chlorobenzene

- ☒ Carbon disulfide

## Resistance to Low Temperatures

Oppanol remains elastic at temperatures down to  $-50^{\circ}\text{C}$ . It gradually becomes harder at lower temperatures and eventually becomes brittle.

## Heat Resistance

- ☒ The mechanical properties of high molecular weight Oppanol remain virtually unchanged up to a temperature of about  $100^{\circ}\text{C}$ . It gradually becomes plastic at higher temperatures, and it starts to flow at  $180 - 200^{\circ}\text{C}$  (under nitrogen). The polymer starts to degrade rapidly at this temperature in the presence of air.

- ☒ Medium molecular weight Oppanol starts to flow at about  $100^{\circ}\text{C}$ . It starts to degrade at  $160 - 180^{\circ}\text{C}$ .

## Resistance to Sunlight and Oxidation

Unstabilized Oppanol undergoes hardly any discoloration in diffuse light, and it does not undergo any other significant

physical changes. It begins to degrade in direct sunlight, and especially when irradiated with UV light, which is indicated by a decrease in strength and elasticity and increased tackiness. The resistance of Oppanol to sunlight can be improved substantially by adding pigments and fillers such as carbon black or by adding antioxidants and UV stabilizers.

## Affinity for Fillers

Oppanol has a high affinity for all types of fillers. There is no difficulty in preparing sheets consisting of 1 part of Oppanol and 10 parts of fillers such as china clay, chalk or talc, etc.

## Compatibility with Rubber, Resins, Waxes, etc.

Oppanol can be mixed with rubber and rubber substitutes.

## Dielectric Properties

Oppanol is a nonpolar, hydrocarbon polymer. It is an excellent insulator, and its dielectric properties are not affected by prolonged immersion in water.

## Dimensional Stability

Oppanol displays cold flow, and undergoes permanent deformation under a permanent load.

## Reactivity

Oppanol is an almost completely saturated paraffinic polymer, and therefore practically inert.

## Cross-Linking

The methods that are usually used to cross-link saturated ethylene polymers, such as applying high-energy radiation or adding peroxide and allowing it to decompose, cause Oppanol to break down.

Because Oppanol is saturated, it cannot be vulcanized with sulfur. Peroxides need to be added to Oppanol before it can be vulcanized.

## Typical properties

Density at 20°C	0.92	g/cm <sup>3</sup>
Glass transition temperature, T <sub>g</sub> (DSC)	-62	°C
Specific heat, c	2.0	kJ*kg <sup>-1</sup> *K <sup>-1</sup>
Thermal conductivity, λ	0.19	W*K <sup>-1</sup> *m <sup>-1</sup>
Refractive index, n <sup>20</sup> D	1.51	
Dielectric constant, ε <sub>r</sub> (50 Hz, 23°C)	2.2	
Dissipation factor, tan δ (50 Hz, 23°C)	<=5*10 <sup>-4</sup>	
Specific resistance	1,016	Ω*cm
Coefficient of permeability to water vapor	2.5*10 <sup>-7</sup>	g*m <sup>-1</sup> *h <sup>-1</sup> *mbar <sup>-1</sup>

### Note:

The details presented here on the properties and processing of Oppanol<sup>®</sup> and its applications are for information purposes only. They do not constitute a specification or a guarantee of specific properties.

### 3.2. Processing

Oppanol is usually packaged in 20 kg cardboard boxes, 20 kg bags, and 100 lbs drums.

Oppanol is a thermoplastic and can be processed with conventional machinery used in the rubber industry, such as kneaders, roll mills, calendars, single- and twin-screw extruders, and extrusion presses. Recently, press mixers have also been used.



### 3.3. Removing Packaging

Cardboard box (Fig. 1 - Fig. 4)

BASF recommends opening the box on both sides longitudinally by hand (Fig. 2), pulling out the flaps (Fig. 3), and folding down the side walls (Fig. 4).



Figure 1



Figure 2

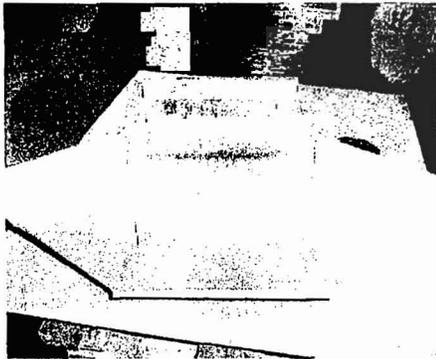


Figure 3

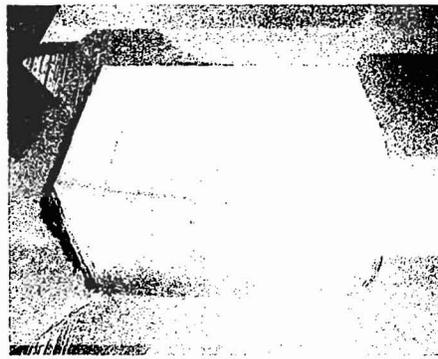


Figure 4

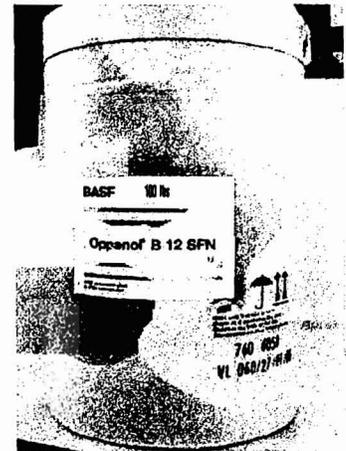


Figure 5

Drum (Fig. 5)

Remove lid from the top of the drum. Place drum upside down to remove the material.



Figure 6



Figure 7



Figure 8

Plastic bag (Fig. 6 - Fig. 8)

BASF recommends cutting open the liner at the front and the sides (Fig. 7). If the bag sticks to the contents and is difficult to remove, then the bag can be inflated with compressed air (Fig. 8) before it is cut open.

### 3.4. Applications

#### Formulations that Contain Fillers

##### *Building industry:*

For sealing membranes used to seal buildings from standing water; roofing membranes with or without standing water, waterproofing for tunnels and cellars in combination with bitumen

##### *Corrosion protection:*

Linings for chemical reactors, other vessels, and underbody sealants for automobiles

##### *Electrical industry:*

Conductive films filled with graphite and magnetic films filled with barium ferrite

##### *Electrical insulation:*

Extremely high flashover voltage

##### *Inorganic fillers:*

Carbon black, graphite, chalk, shale, quartz, china clay and talc

##### *Thermoplastic additives:*

Thermoplastics such as EVA, copolymers, PP, HDPE, LDPE and LLDPE act as plasticizers at temperatures above their melting point. They reduce shear, resulting in less mechanical degradation when Oppanol is processed. At temperatures below their melting point, these thermoplastics act as fillers at low deformation, resulting in a higher modulus of elasticity and a higher elongation at break.

#### Rubber Industry

##### *Coating compounds:*

Additive for compounds of natural and synthetic rubber

##### *Regeneration:*

Used in the regeneration of waste vulcanized rubber

##### *Cable sheathing:*

Good insulator with self sealing properties to prevent short circuits if sheathing is punctured

##### *Ebonite compounds:*

Increases the impact resistance of ebonite

#### Mixtures with Waxes

Mixtures of this type are mainly used to coat, impregnate and laminate paper, plastic film, and metal foil.

Advantages: High melt viscosity, low permeability to water vapor, very pliable film, better adhesion, improved sealability

#### Mixtures with Bitumen

Advantages: Increased melt viscosity, a more pliable bitumen film, improved resistance to changes in temperature (in bridge construction), higher weathering resistance, better adhesion, very high water resistance

#### Adhesives

Oppanol® is used on account of its resistance to aging and to attack by chemicals in pressure sensitive adhesives for technical and medical applications, such as surgical plasters, sealing tapes, insulating tapes, masking tapes and anticorrosion tapes.

#### Permanently Elastic Sealants

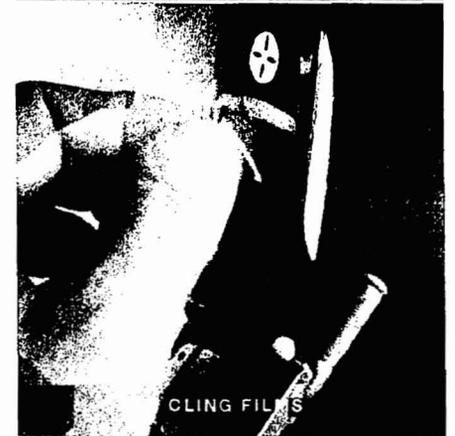
Permanently elastic sealants (mainly for double-glazed windows) can be manufactured by blending different Oppanol® types and using fillers such as chalk, zinc oxide, china clay or barites. Bitumen may also be included in the formulation.

#### Chewing Gum Base

Chewing gum base is manufactured by mixing Oppanol with chalk, corn oil, paraffin wax and other waxes and fats, etc. The chewing gum itself is made by adding icing sugar, dextrose, glycerin, citric acid and flavoring to the chewing gum base.

#### Mixtures with Mineral Oil and Lubricants

Oppanol can be used to increase the viscosity and adhesion of oils and chain lubricants, or as a thickener for lubricating grease, and a drag reducer in oil pipelines.



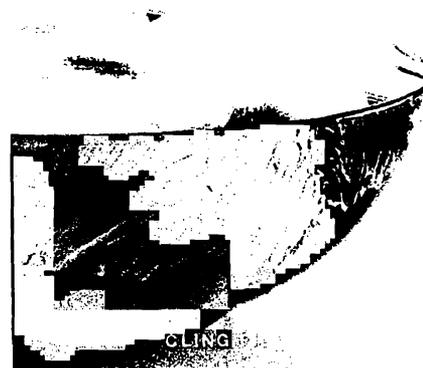
## 4. Other Possible Applications

### - Oppanol®

- Disposal of solid and liquid waste
- Disposal of metal dust, asbestos dust, and toxic sludge
- Removing oil pollution from water
- Cleaning up oil slicks after tanker accidents
- Recycling plastic waste (in the automotive sector)
- Can be processed chemically into lubricant additives, copolymers, and sealants with enhanced adhesion

### - Blends of Oppanol® and other polymers

- Sterile tubes for medical suction equipment
- Infusion tubes
- Seals for disposable syringes
- Medical metering equipment
- Automotive films
- Decorative films, including films laminated with textile fibers
- Household films
- Cling film for food packaging
- Peelable film for yogurt containers, etc.
- Cosmetic packaging
- Sealants for bottle caps
- Non-slip tablemats
- Protective panels for vacuum cleaners



Note:

The details presented here on the properties and processing of Oppanol and its applications are for information purposes only. They do not constitute a specification or a guarantee of specific properties.

## 5. Specifications, Technical Literature, Safety Data Sheets, and Product Range

### Product range:

Oppanol	Molecular Weight		Consistency
	Mw (GPC)	Mv	
B 10 SFN	36,000	40,000	Soft, resinous
B 12 SFN	51,000	55,000	Soft, resinous
B 13 SFN	60,000	65,000	Soft, resinous
B 15 SFN	75,000	85,000	Soft, resinous
B 30 SF	200,000	200,000	Soft, resinous
B 50 / B 50 SF	340,000	400,000	Soft, resinous
B 80	750,000	800,000	Rubbery
B 100	1,100,000	1,110,000	Rubbery
B 150	2,500,000	2,600,000	Rubbery
B 200	4,100,000	4,000,000	Rubbery

The C of A lists Staudinger index (Jo) measured by a BASF method. Specifications, technical literature, and safety data sheets are available from your local BASF representatives.

## 6. Markings

### 6.1. Oppanol® B 10 SFN, B 12 SFN, B 13 SFN, B 15 SFN, B 30 SF, B 50, B 50 SF

These products are packed in 20 kg (44 lb) corrugated cardboard boxes with an inner silicone release coating and in 100 lb paper/fiber drums lined with a silicone coated nylon liner.

The lid is labeled as follows:

BASF Oppanol B XX XXX 20 kg  
BASF Aktiengesellschaft D-67056 Ludwigshafen  
MADE IN GERMANY

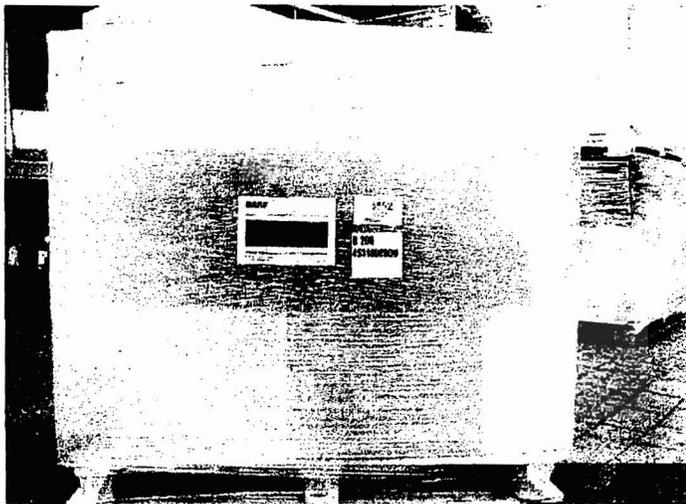
The cardboard boxes are labeled on only one side of the box.

The symbols for "this side up" and "protect from moisture" also appear.



On one pallet there are 30 cardboard boxes or 9 drums.

A large, colored sticker is attached to one of the boxes on each pallet in order to draw attention to the designation of the product. Attached to the same box is a stamped label, which gives details of the total net weight of the pallet load and the number of the pallet. This serial number is important for tracing back the pallet and for processing shipment related requests.



The pallet is covered with shrink wrap to protect it from moisture.



## 6.2. Oppanol® B 80, B 100, B 150, B 200

These products are supplied in 20 kg LDPE bags.

Each bag is labeled as follows:

BASF Oppanol B XX XXX 20 kg  
BASF Aktiengesellschaft D-67056 Ludwigshafen Germany

The bags are also labeled with the code number of the packaging and a recycling symbol.

The grade of product is printed on the label.

The bags are supplied on pallets in units of 30 (Oppanol B 150 / B 200) or 40 (Oppanol B 80 / B 100). A large corrugated cardboard cover is placed over the bags on the pallet in order to ensure that the stack remains stable when the bags at the bottom are deformed under the weight of the bags on top as the result of cold flow.

A large sticker is attached to the cardboard cover of each pallet in order to draw attention to the designation of the product. A label is also attached which gives details of the total net weight of the pallet load and the number of the pallet. This pallet number is important for tracing back for processing complaints.

The cardboard cover is then shrink wrapped to protect it from moisture.



## 7. Dispatch

When the product is ready to be shipped, labels are attached providing details of the internal work order number and customer specific information.

These labels appear on the outside of the shrink wrap.

## 8. Traceability

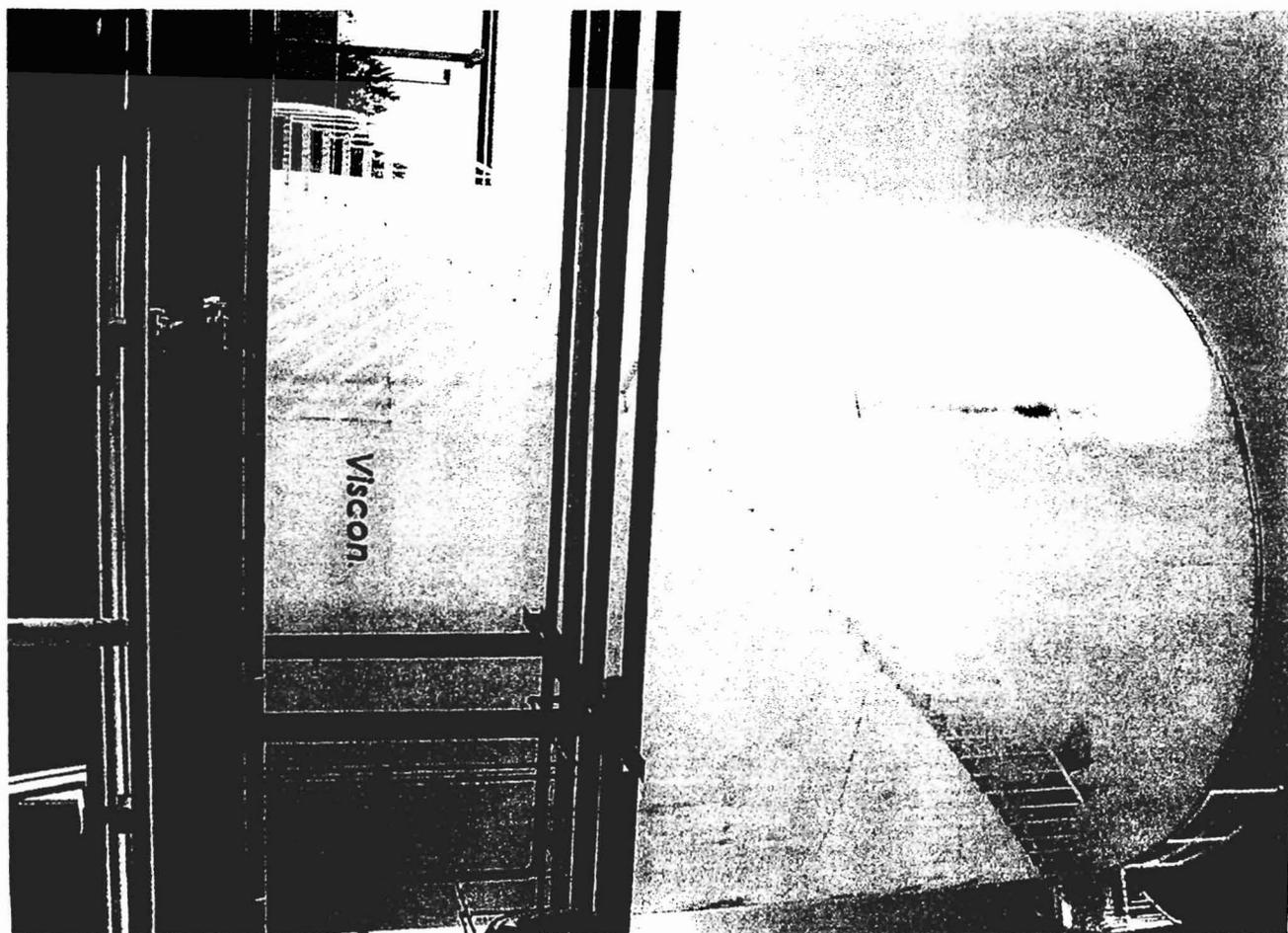


The pallet is covered with shrink wrap to protect it from moisture.



**Attachment 3**

**Viscon Production Flow**

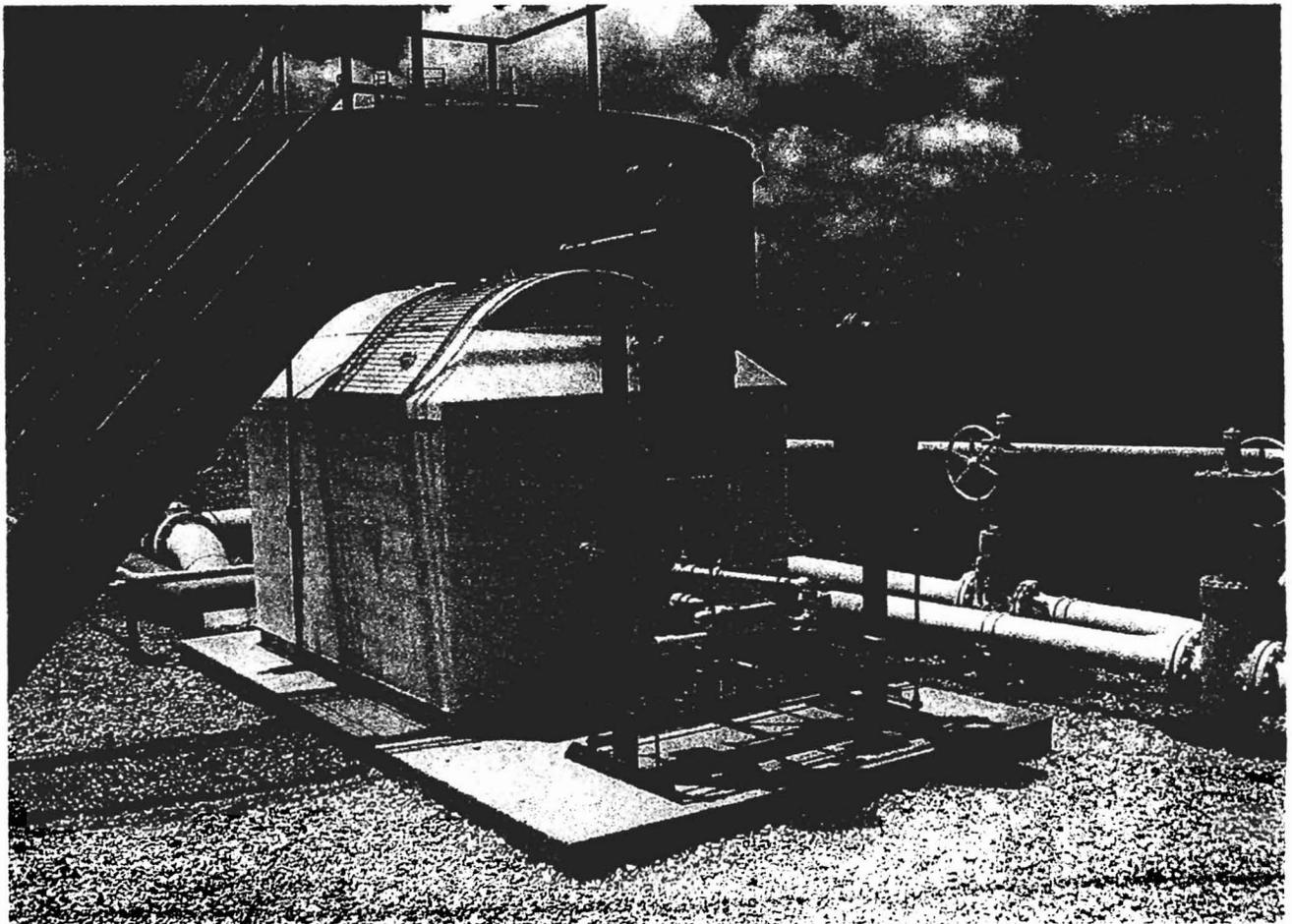
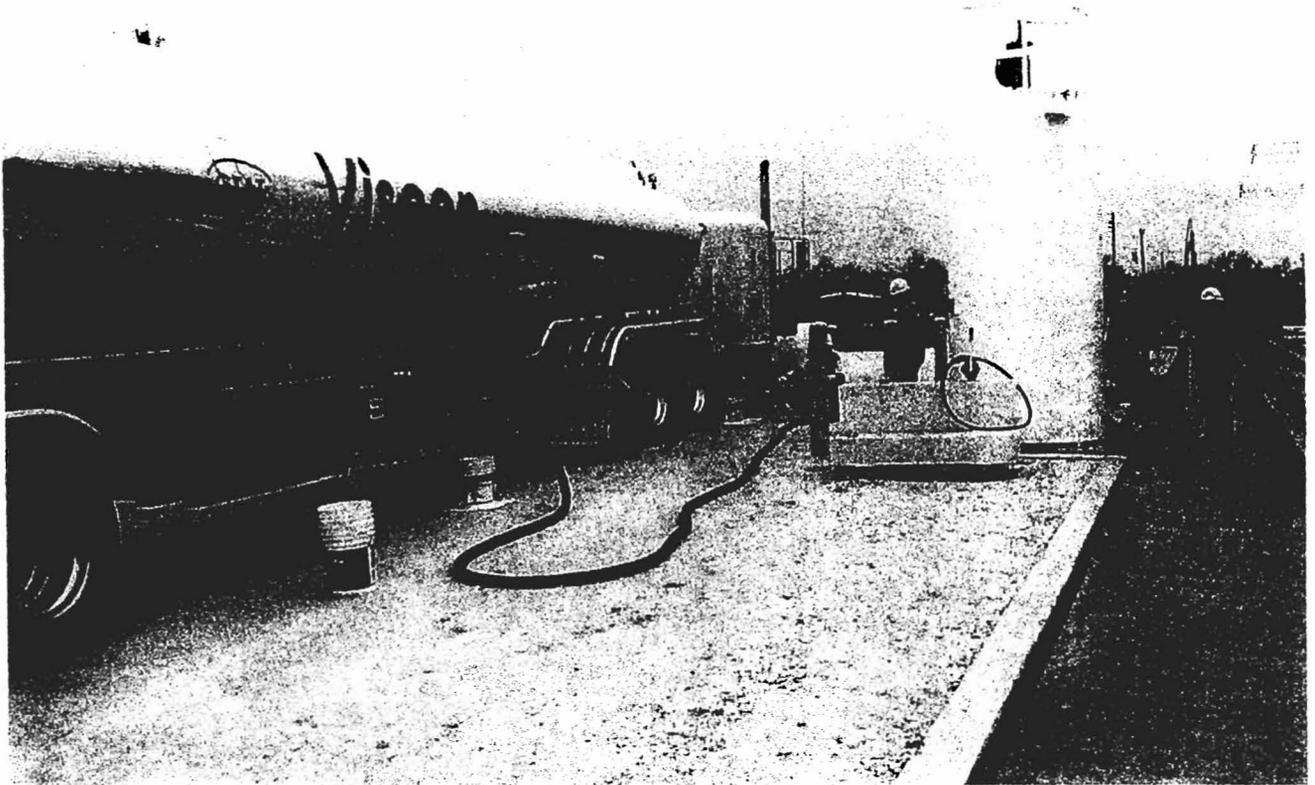


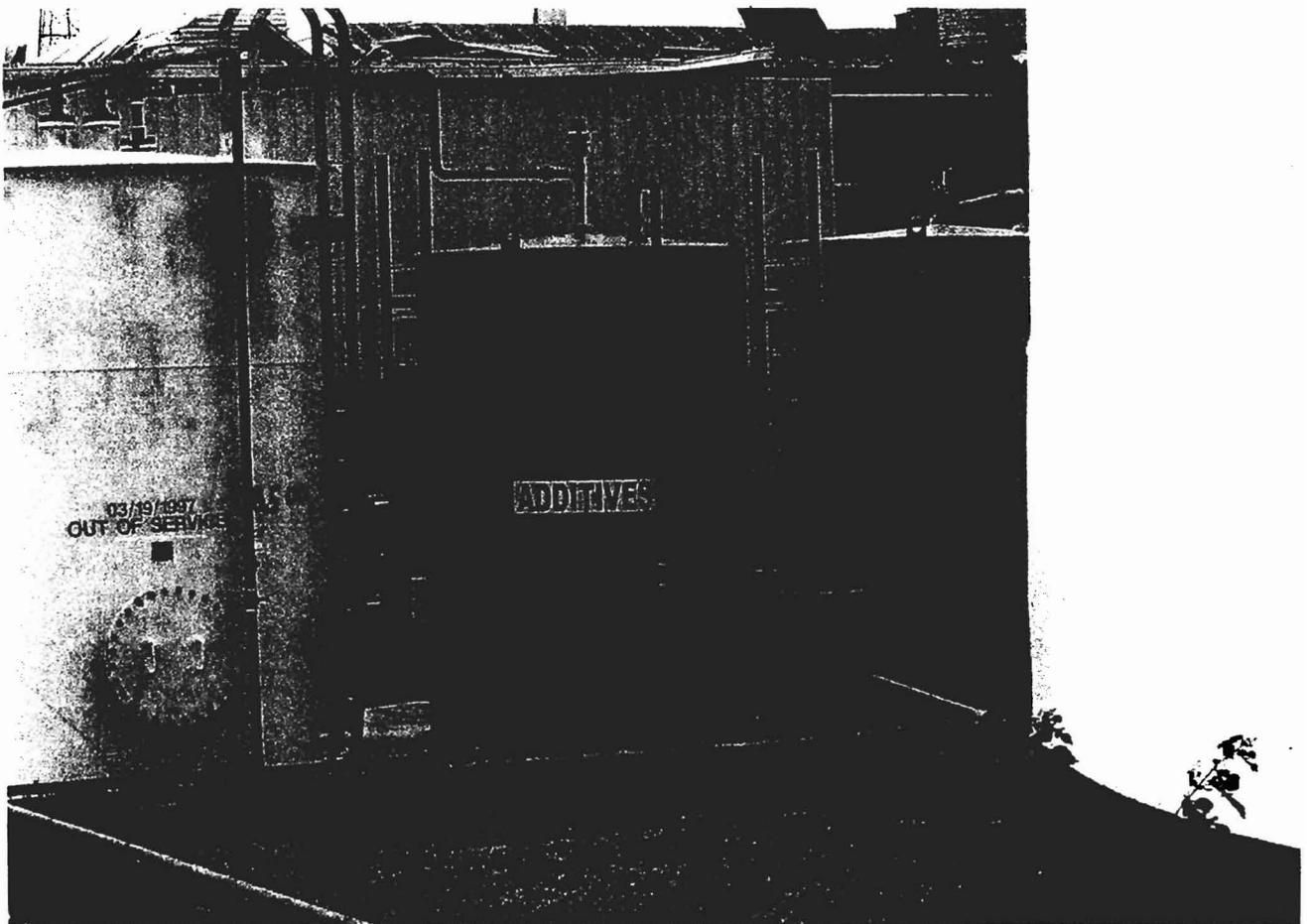


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**Attachment 4**

**Customer-Site Storage**





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**Attachment 5**

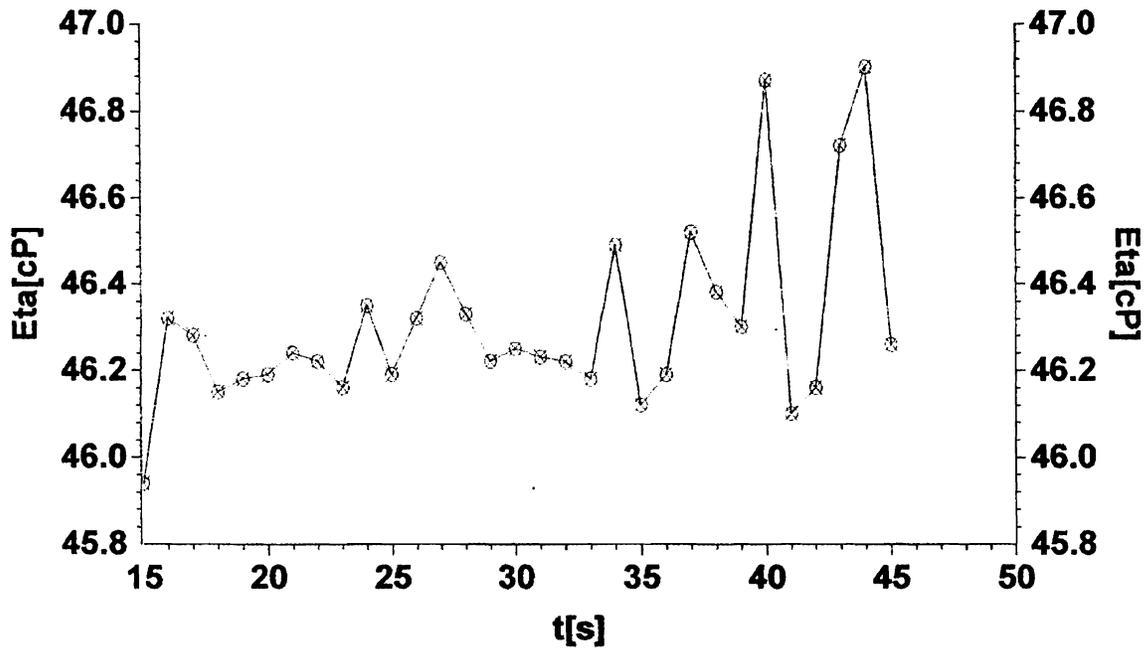
**Viscosity Measurements  
(Quality Control)**

Customer: Quality Control  
 Measuring-Ident: Batch # 222  
 Medium-Ident: Tower 3  
 Operator: Misty  
 Comment:

Batch was made 10.09.06

Program filename: C:\RHEO28\PROG\Viscon Sequence.seq  
 Prog.mode: sequence  
 Date: 06:58 10/10/06  
 Block termination: Block end  
 Block 2 of 2:  
 measuring-system: CC48 DIN  
 Rheometer: R/S+ Rheometer Ver.:9.00 from 15.02.05, Serialnumber: #303117

filters active  
 t[s]>=15  
 data-graph:



- Eta[cP]=f(t[s]):Batch # 222.dat, Block 2
- ×—× Eta[cP]=f(t[s]):Batch # 222.dat, Block 2
- 
- ×—×
- 
- ×—×
- 
- ×—×

Data-grid: Batch # 222.dat Block:2

Datafilter is active!

MP#	t[s]	Eta[mPas]	D[1/s]	Tau[Pa]	T[°C]	M[%.]	n[rpm]	Step
15	15	45.94	250	11.484	32	60.44	48.62	1
16	16	46.32	250	11.581	32	60.95	48.62	1
17	17	46.28	250	11.569	32	60.89	48.62	1
18	18	46.15	250	11.538	32	60.72	48.62	1
19	19	46.18	250	11.545	32	60.76	48.62	1
20	20	46.19	250	11.547	32	60.77	48.62	1
21	21	46.24	250	11.56	32	60.84	48.62	1
22	22	46.22	250	11.556	32	60.82	48.62	1
23	23	46.16	250	11.539	32	60.73	48.62	1
24	24	46.35	250	11.587	32	60.98	48.62	1
25	25	46.19	250	11.548	32	60.78	48.62	1
26	26	46.32	250	11.581	32	60.95	48.62	1
27	27	46.45	250	11.613	32	61.12	48.62	1
28	28	46.33	250	11.583	32	60.97	48.62	1
29	29	46.22	250	11.554	32	60.81	48.62	1
30	30	46.25	250	11.563	32	60.86	48.62	1
31	31	46.23	250	11.557	32	60.83	48.62	1
32	32	46.22	250	11.555	32	60.82	48.62	1
33	33	46.18	250	11.546	32	60.77	48.62	1
34	34	46.49	250	11.623	32	61.17	48.62	1
35	35	46.12	250	11.531	32	60.69	48.62	1
36	36	46.19	250	11.547	32	60.77	48.62	1
37	37	46.52	250	11.629	32	61.21	48.62	1
38	38	46.38	250	11.594	32	61.02	48.62	1
39	39	46.3	250	11.576	32	60.93	48.62	1
40	40	46.87	250	11.718	32	61.67	48.62	1
41	41	46.1	250	11.525	32	60.66	48.62	1
42	42	46.16	250	11.539	32	60.73	48.62	1
43	43	46.72	250	11.679	32	61.47	48.62	1
44	44	46.9	250	11.726	32	61.72	48.62	1
45	45	46.26	250	11.564	32	60.86	48.62	1

Analysis-results:

filter activated: t[s]&gt;=15

step1: average/mean of Eta[Pa]=0.046, S=0.0002

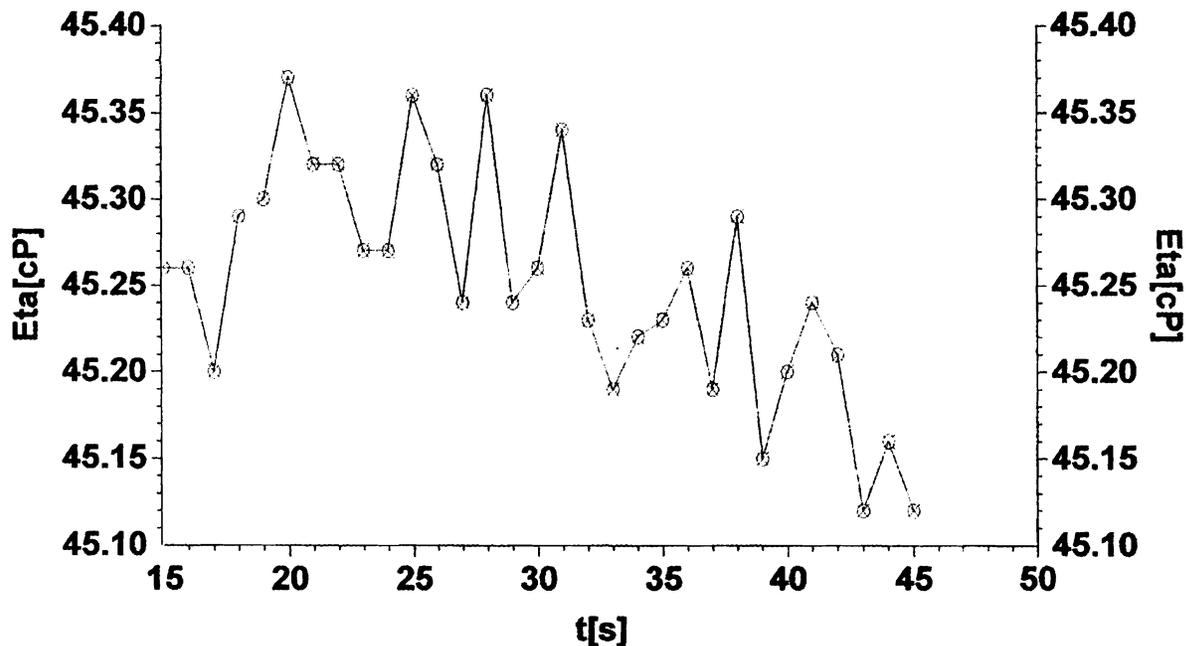
End of report

Customer: Quality Control  
Measuring-Ident: Batch # 329  
Medium-Ident: Tower 2  
Operator: Preston  
Comment:

Batch was made 12.8.06

Program filename: C:\RHEO28\PROG\Viscon Sequence.seq  
Prog.mode: sequence  
Date: 06:44 11/12/06  
Block termination: Block end  
Block 2 of 2:  
measuring-system: CC48 DIN  
Rheometer: R/S+ Rheometer Ver.:9.00 from 15.02.05, Serialnumber: #303117

filters active  
t[s]>=15  
data-graph:



- Eta[cP]=f(t[s]):Batch # 329.dat, Block 2
- ×—× Eta[cP]=f(t[s]):Batch # 329.dat, Block 2
- 
- ×—×
- 
- ×—×
- 
- ×—×

Data-grid: Batch # 329.dat Block:2

Datafilter is active!

MP#	t[s]	Eta[mPas]	D[1/s]	Tau[Pa]	T[°C]	M[%.]	n[rpm]	Step
15	15	45.26	250	11.314	31.4	59.55	48.62	1
16	16	45.26	250	11.316	31.4	59.56	48.62	1
17	17	45.2	250	11.3	31.4	59.47	48.62	1
18	18	45.29	250	11.322	31.4	59.59	48.62	1
19	19	45.3	250	11.325	31.4	59.6	48.62	1
20	20	45.37	250	11.342	31.4	59.69	48.62	1
21	21	45.32	250	11.33	31.4	59.63	48.62	1
22	22	45.32	250	11.329	31.4	59.63	48.62	1
23	23	45.27	250	11.318	31.4	59.57	48.62	1
24	24	45.27	250	11.318	31.4	59.57	48.62	1
25	25	45.36	250	11.341	31.4	59.69	48.62	1
26	26	45.32	250	11.33	31.4	59.63	48.62	1
27	27	45.24	250	11.309	31.4	59.52	48.62	1
28	28	45.36	250	11.341	31.4	59.69	48.62	1
29	29	45.24	250	11.311	31.4	59.53	48.62	1
30	30	45.26	250	11.315	31.4	59.55	48.62	1
31	31	45.34	250	11.334	31.4	59.65	48.62	1
32	32	45.23	250	11.308	31.4	59.52	48.62	1
33	33	45.19	250	11.298	31.4	59.46	48.62	1
34	34	45.22	250	11.305	31.4	59.5	48.62	1
35	35	45.23	250	11.307	31.4	59.51	48.62	1
36	36	45.26	250	11.315	31.4	59.55	48.62	1
37	37	45.19	250	11.298	31.4	59.47	48.62	1
38	38	45.29	250	11.322	31.4	59.59	48.62	1
39	39	45.15	250	11.287	31.4	59.41	48.62	1
40	40	45.2	250	11.3	31.4	59.48	48.62	1
41	41	45.24	250	11.311	31.4	59.53	48.62	1
42	42	45.21	250	11.303	31.4	59.49	48.62	1
43	43	45.12	250	11.279	31.4	59.36	48.62	1
44	44	45.16	250	11.289	31.4	59.42	48.62	1
45	45	45.12	250	11.28	31.4	59.37	48.62	1

Analysis-results:

filter activated: t[s]&gt;=15

step1: average/mean of Eta[Pa]=0.045, S=0.0001

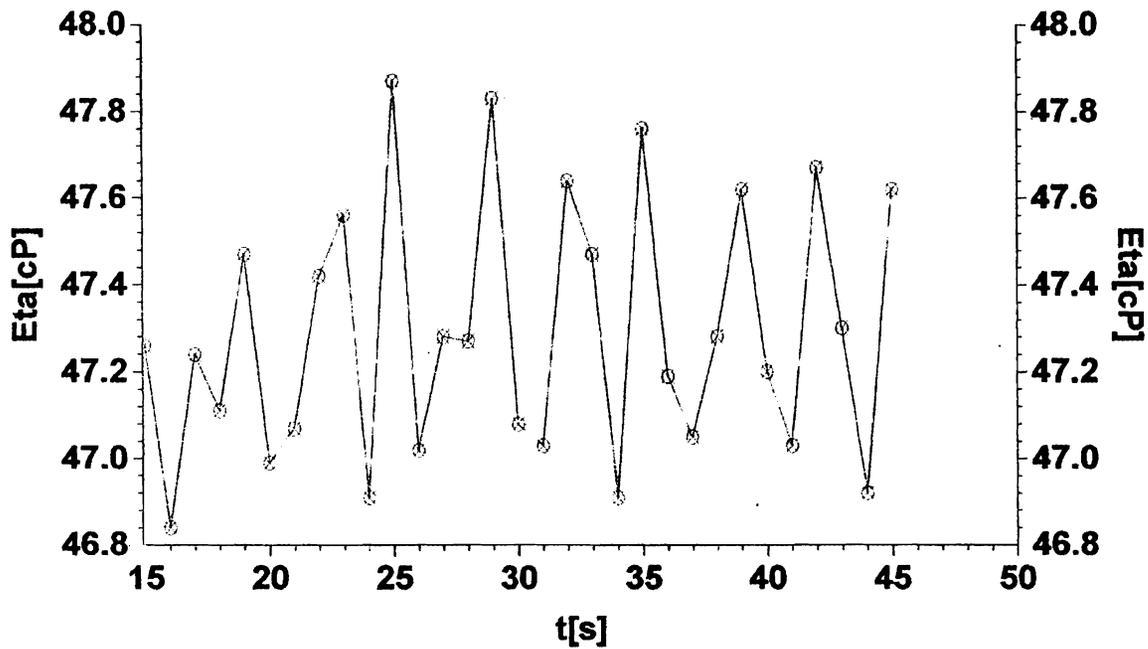
End of report

Customer: Quality Control  
Measuring-Ident: Batch # 313  
Medium-Ident: Tower 1  
Operator: Misty  
Comment:

Batch was made 11.07.06

Program filename: C:\RHEO28\PROG\Viscon Sequence.seq  
Prog.mode: sequence  
Date: 06:02 08/11/06  
Block termination: Block end  
Block 2 of 2:  
measuring-system: CC48 DIN  
Rheometer: R/S+ Rheometer Ver.:9.00 from 15.02.05, Serialnumber: #303117

filters active  
t[s]>=15  
data-graph:



○—○ Eta[cP]=f(t[s]):Batch # 313.dat, Block 2  
x—x Eta[cP]=f(t[s]):Batch # 313.dat, Block 2  
○—○  
x—x  
○—○  
x—x  
○—○  
x—x

Data-grid: Batch # 313.dat Block:2

Datafilter is active!

MP#	t[s]	Eta[mPas]	D[1/s]	Tau[Pa]	T[°C]	M[%.]	n[rpm]	Step
15	15	47.26	250	11.816	31.9	62.19	48.62	1
16	16	46.84	250	11.71	31.9	61.63	48.62	1
17	17	47.24	250	11.81	31.9	62.16	48.62	1
18	18	47.11	250	11.778	31.9	61.99	48.62	1
19	19	47.47	250	11.868	31.9	62.47	48.62	1
20	20	46.99	250	11.747	31.9	61.82	48.62	1
21	21	47.07	250	11.768	31.9	61.94	48.62	1
22	22	47.42	250	11.856	31.9	62.4	48.62	1
23	23	47.56	250	11.891	31.9	62.59	48.62	1
24	24	46.91	250	11.728	31.9	61.73	48.62	1
25	25	47.87	250	11.968	31.9	62.99	48.62	1
26	26	47.02	250	11.754	31.9	61.86	48.62	1
27	27	47.28	250	11.819	31.9	62.2	48.62	1
28	28	47.27	250	11.817	31.9	62.2	48.62	1
29	29	47.83	250	11.958	31.9	62.94	48.62	1
30	30	47.08	250	11.769	31.9	61.94	48.62	1
31	31	47.03	250	11.758	31.9	61.88	48.62	1
32	32	47.64	250	11.909	31.9	62.68	48.62	1
33	33	47.47	250	11.867	31.9	62.46	48.62	1
34	34	46.91	250	11.728	31.9	61.73	48.62	1
35	35	47.76	250	11.941	31.9	62.85	48.62	1
36	36	47.19	250	11.797	31.9	62.09	48.62	1
37	37	47.05	250	11.763	31.9	61.91	48.62	1
38	38	47.28	250	11.82	31.9	62.21	48.62	1
39	39	47.62	250	11.905	31.9	62.66	48.62	1
40	40	47.2	250	11.799	31.9	62.1	48.62	1
41	41	47.03	250	11.757	31.9	61.88	48.62	1
42	42	47.67	250	11.918	31.9	62.73	48.62	1
43	43	47.3	250	11.824	31.9	62.23	48.62	1
44	44	46.92	250	11.731	31.9	61.74	48.62	1
45	45	47.62	250	11.905	31.9	62.66	48.62	1

Analysis-results:

filter activated: t[s]&gt;=15

step1: average/mean of Eta[Pas]=0.047, S=0.0003

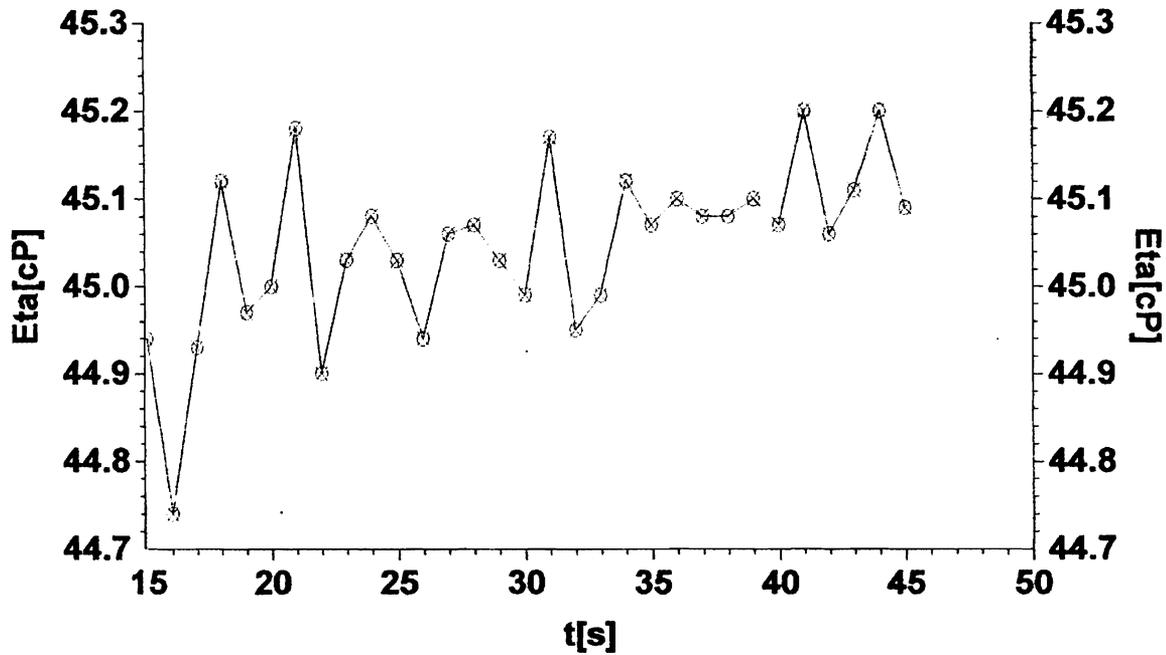
End of report

Customer: Quality Control  
Measuring-Ident: 7,000 Gallons  
Medium-Ident: Tank 351  
Operator: Misty  
Comment:

7,000 gallons going to Texas pulled from Tank 351 01.02.07

Program filename: C:\RHEO28\PROG\Viscon Sequence.seq  
Prog.mode: sequence  
Date: 12:23 02/01/07  
Block termination: Block end  
Block 2 of 2:  
measuring-system: CC48 DIN  
Rheometer: R/S+ Rheometer Ver.:9.00 from 15.02.05, Serialnumber: #303117

filters active  
t[s]>=15  
data-graph:



- Eta[cP]=f(t[s]):7,000 Gal. 01.02.07.dat, Block 2
- ×—× Eta[cP]=f(t[s]):7,000 Gal. 01.02.07.dat, Block 2
- 
- ×—×
- 
- ×—×
- 
- ×—×

Data-grid: 7,000 Gal. 01.02.07.dat Block:2

Datafilter is active!

MP#	t[s]	Eta[mPas]	D[1/s]	Tau[Pa]	T[°C]	M[%.]	n[rpm]	Step
15	15	44.94	250	11.234	32.2	59.13	48.62	1
16	16	44.74	250	11.186	32.2	58.88	48.62	1
17	17	44.93	250	11.232	32.2	59.12	48.62	1
18	18	45.12	250	11.281	32.2	59.38	48.62	1
19	19	44.97	250	11.243	32.2	59.18	48.62	1
20	20	45	250	11.25	32.2	59.21	48.62	1
21	21	45.18	250	11.294	32.2	59.44	48.62	1
22	22	44.9	250	11.225	32.2	59.08	48.62	1
23	23	45.03	250	11.257	32.2	59.25	48.62	1
24	24	45.08	250	11.269	32.2	59.31	48.62	1
25	25	45.03	250	11.258	32.2	59.26	48.62	1
26	26	44.94	250	11.234	32.2	59.13	48.62	1
27	27	45.06	250	11.265	32.2	59.29	48.62	1
28	28	45.07	250	11.267	32.2	59.3	48.62	1
29	29	45.03	250	11.257	32.2	59.25	48.62	1
30	30	44.99	250	11.247	32.2	59.2	48.62	1
31	31	45.17	250	11.293	32.2	59.44	48.62	1
32	32	44.95	250	11.237	32.2	59.14	48.62	1
33	33	44.99	250	11.248	32.2	59.2	48.62	1
34	34	45.12	250	11.279	32.2	59.36	48.62	1
35	35	45.07	250	11.268	32.2	59.3	48.62	1
36	36	45.1	250	11.274	32.2	59.34	48.62	1
37	37	45.08	250	11.269	32.2	59.31	48.62	1
38	38	45.08	250	11.271	32.2	59.32	48.62	1
39	39	45.1	250	11.275	32.2	59.34	48.62	1
40	40	45.07	250	11.268	32.2	59.31	48.62	1
41	41	45.2	250	11.299	32.2	59.47	48.62	1
42	42	45.06	250	11.265	32.2	59.29	48.62	1
43	43	45.11	250	11.277	32.2	59.36	48.62	1
44	44	45.2	250	11.301	32.2	59.48	48.62	1
45	45	45.09	250	11.272	32.2	59.32	48.62	1

Analysis-results:

filter activated: t[s]&gt;=15

step1: average/mean of Eta[Pas]=0.045, S=0.0001

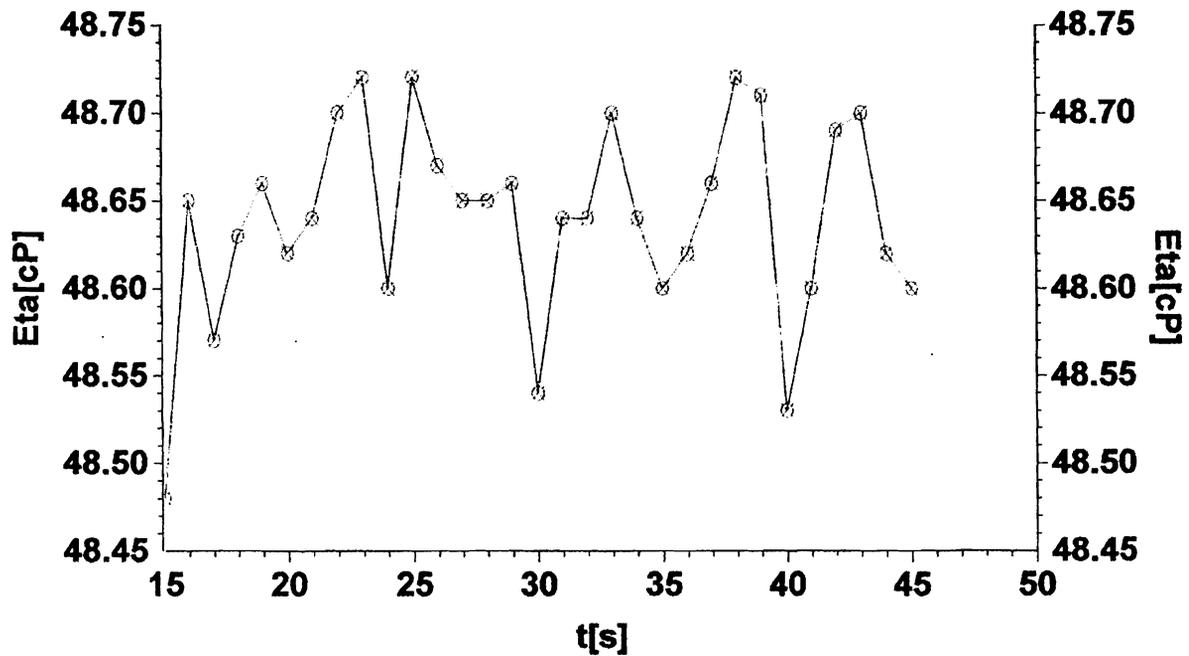
End of report

**Customer:** Quality Control  
**Measuring-Ident:** Batch # 165  
**Medium-Ident:** Tower # 2  
**Operator:** Preston  
**Comment:**

Batch made 8.31.06

**Program filename:** C:\RHEO28\PROG\Viscon Sequence.seq  
**Prog.mode:** sequence  
**Date:** 10:27 06/09/06  
**Block termination:** Block end  
**Block 2 of 2:**  
**measuring-system:** CC48 DIN  
**Rheometer:** R/S+ Rheometer Ver.:9.00 from 15.02.05, Serialnumber: #303117

filters active  
t[s]>=15  
data-graph:



Data-grid: Batch # 165.dat Block:2

Datafilter is active!

MP#	t[s]	Eta[mPas]	D[1/s]	Tau[Pa]	T[°C]	M[%.]	n[rpm]	Step
15	15	48.48	250	12.12	31.8	63.79	48.62	1
16	16	48.65	250	12.163	31.8	64.01	48.62	1
17	17	48.57	250	12.142	31.8	63.91	48.62	1
18	18	48.63	250	12.158	31.8	63.99	48.62	1
19	19	48.66	250	12.166	31.8	64.03	48.62	1
20	20	48.62	250	12.155	31.8	63.97	48.62	1
21	21	48.64	250	12.161	31.8	64	48.62	1
22	22	48.7	250	12.174	31.8	64.08	48.62	1
23	23	48.72	250	12.181	31.8	64.11	48.62	1
24	24	48.6	250	12.15	31.8	63.95	48.62	1
25	25	48.72	250	12.179	31.8	64.1	48.62	1
26	26	48.67	250	12.168	31.8	64.04	48.62	1
27	27	48.65	250	12.163	31.8	64.02	48.62	1
28	28	48.65	250	12.162	31.8	64.01	48.62	1
29	29	48.66	250	12.166	31.8	64.03	48.62	1
30	30	48.54	250	12.134	31.8	63.86	48.62	1
31	31	48.64	250	12.159	31.8	64	48.62	1
32	32	48.64	250	12.161	31.8	64.01	48.62	1
33	33	48.7	250	12.175	31.8	64.08	48.62	1
34	34	48.64	250	12.159	31.8	63.99	48.62	1
35	35	48.6	250	12.149	31.8	63.94	48.62	1
36	36	48.62	250	12.154	31.8	63.97	48.62	1
37	37	48.66	250	12.166	31.8	64.03	48.62	1
38	38	48.72	250	12.18	31.8	64.1	48.62	1
39	39	48.71	250	12.178	31.8	64.1	48.62	1
40	40	48.53	250	12.132	31.8	63.85	48.62	1
41	41	48.6	250	12.15	31.8	63.95	48.62	1
42	42	48.69	250	12.172	31.8	64.06	48.62	1
43	43	48.7	250	12.174	31.8	64.07	48.62	1
44	44	48.62	250	12.154	31.8	63.97	48.62	1
45	45	48.6	250	12.149	31.8	63.94	48.62	1

Analysis-results:

filter activated: t[s]&gt;=15

step1: average/mean of Eta[Pas]=0.049, S=0.0001

End of report

**Attachment 6**

**Issuance of License for Oil Spill Clean-Up Agent**

STATE OF CALIFORNIA

State Water Resources Control Board

**LICENSE for Oil Spill Cleanup Agent**

**Issued to:** GENERAL TECHNOLOGIES APPLICATIONS, INC.

PRODUCT NAME: "ELASTOL"

PRODUCT  
CLASSIFICATION: COLLECTING AGENT

DATE ISSUED: MAY 1, 1992

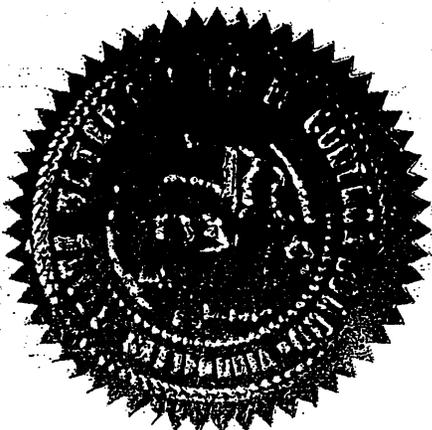
EXPIRATION DATE: MAY 1, 1997

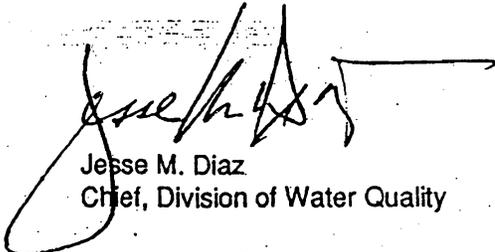
*The applicant having followed the procedure as outlined in Title 23, Chapter 3, Subchapter 10, of the California Administrative Code, and having submitted proper applications which was reviewed by interested agencies and is found in order for approval, this license is hereby issued.*

*Use of the product must be in strict compliance with Section 2332 of referenced Administrative Code.*

*The term of this license shall be for a period of five (5) years unless revoked prior to that date for good cause.*

*This license does not denote endorsement of the product by the State Water Resources Control Board or any other State agency.*



  
Jesse M. Diaz  
Chief, Division of Water Quality

**Attachment 7**

**Extensional Viscosity**

**Extensional Viscosity and a Single  
Phenomenological Basis for the  
GTA Fuel Additive**

1. Approach

We begin with the equations for Newtonian viscosity as a function of concentration and molecular weight. Then we look at the parameters influencing the extensional viscosity. Finally, we look at the parameters governing the evaporation of a liquid from the surface of an evaporating drop. The extensional viscosity relationship is then overlaid on the equation for evaporation of a liquid drop. Calculated values will be given in the next discussion paper.

2. Resting Viscosity of a Dilute Polymer Solution

The Newtonian (resting) viscosity of a very dilute polymer solution is close to that of the solvent. This is a consequence of the following two equations:

$$\begin{aligned} \text{(I)} \quad & [\eta] = KM^{1/2} \quad \text{and} \\ \text{(II)} \quad & \eta_{sp} = [\eta]c + k'[\eta]^2c^2 \end{aligned}$$

where  $[\eta]$  is the intrinsic viscosity of the polymer - solvent system,  $M$  is molecular weight, and  $K$  is the constant of proportionality. Note that  $[\eta]$  is weakly dependent on molecular weight, thus the specific viscosity of a given solution concentration is also little increased by  $M$ . This is especially the case when the polymer concentration,  $c$ , is also small as it is for fuel additive at 10 ppm, or 0.0010 grams per deciliter.  $c$  is  $1 \times 10^{-3}$  g/dl and  $c^2$  is  $1 \times 10^{-6}$  g<sup>2</sup>/dl<sup>2</sup>. Thus the treated fuel is not very different from untreated fuel at rest. References for this are 1) Billmeyer and 2) Flory.

3. Extensional Viscosity

The ratio of viscosity of stretched, elongated or extended polymer solution to the viscosity of the unperturbed solution is given by

$$\text{(III)} \quad \eta/\eta_0 = 3 + \epsilon c \eta_0 K^2 M^{(1+2\alpha)}/RT$$

The extensional viscosity,  $\eta$ , is strongly dependent on the molecular weight. The term  $M^{(1+2\alpha)}$  becomes  $M^2$  for the case of

worst solvent for the polymer, and  $M^3$  for the best possible solvent. Polyisobutylene and fuel are ideally compatible and the term more closely approaches  $M^3$  than  $M^2$ . Despite the strong dependencies of  $\eta/\eta_0$  on the molecular weight,  $\eta/\eta_0$  approaches the low limiting value of 3 when  $c$  approaches 0. This is why the ratio can be "tuned" to a particular type of engine or burning condition, (further discussions will be postponed to the next section). The rate of strain,  $\dot{\epsilon}$  is a measure of elongation per unit length per second, or  $\Delta L/L\Delta t$ . This parameter changes when the solution is sheared, stretched, or deformed. Physically the large polymer chains are deformed from a random coil conformation to a stretched chain conformation. In the absence of the strain, the chains thermally relax to the random coils. Often the strain induces a stress which breaks some of the extended molecules in the middle (mid-point break theory). However, in undergoing a break, the fluid is also most rigid. In other words  $\Delta L/L\Delta t$  was very large and  $\eta/\eta_0$  was also maximized. Regions where  $\Delta L/L\Delta t$  are large include turbulent liquid flow regions (drag reduction effect), injection from a nozzle (cohesive effect), the interaction of a liquid stream or droplets with high velocity air. These conditions exist in most modern engines, especially when conditions are changing in the millisecond to microsecond domain.  $K$  in this equation is obtained in the laboratory by plotting the log of the intrinsic viscosity against the log of the molecular weight and taking the slope of the line.  $K$  is constant for a particular polymer solvent system. The  $K$  for polyisobutylene in Benzene is  $1.07 \times 10^{-3}$  at 297°K.  $T$  enters the expression for  $\eta/\eta_0$  in the term  $1/RT$  where  $R$  is the gas constant.  $T$  has little effect on  $\eta/\eta_0$  since there is usually little displacement from 298 degrees (25°C).  $T$  could be 273 (0°C) or perhaps 325 degrees, but the effect on  $\eta/\eta_0$  is very small.

#### 4. Effects on Fuel Properties

Under some conditions  $\eta/\eta_0$  can approach 10,000. When this is too large for an optimal effect on combustion or volumetric efficiency, or delayed vaporization, or suppression of vapor-like particles, the concentration can be decreased toward zero. This it is always possible to tune the fuel for 2 cycle spark ignition, 4 cycle spark ignition, compression ignition, turbine or other combustion scenarios.  $c$  can also be changed for different carburetor or fuel injection systems. High molecular weight is an advantage because it generates large  $\eta/\eta_0$  values at low concentrations where "at rest" fuel properties are virtually unaffected. There is no doubt that 5 to 10 ppm concentrations of  $7.2 \times 10^6$  M PIB decreases energy loss in turbulently flowing fuels, resulting in flow rate increase of more than 20%. There is also no doubt that the formation of vapor-like particles is suppressed

at low concentrations of high molecular weight PIB. These two effects are related to  $\eta/\eta_0$ .

However,  $\eta/\eta_0$  is also related to transient increases in the "solidity" and surface tension of particles produced by shear at surfaces or in turbulent air. The following relationships have been derived in "Physical Chemistry", by Moelwyn - Hughes p. 1213. The derivation will not be repeated here. Consider a sphere of isolated liquid in an inert atmosphere at constant temperature and pressure. Let the radius be larger than  $10^{-6}$  cm. The number of molecules vaporizing from the surface of the sphere per second is given by

$$(IV) \quad dN/dt = 4\pi r D n^\circ$$

$r$  is the radius of the droplet and  $D$  is the diffusion coefficient of molecules within the droplet.  $n^\circ$  is the saturation vapor pressure at the interface of the drop with the gaseous atmosphere.  $r_m$  is the molecular radius. The link between  $D$  and  $\eta$  is via the Stokes relationship or

$$(V) \quad D = RT/n_0 6\pi n r_m$$

accumulating the constants and the radius of a molecule equation V becomes

$$D = k/\eta, \text{ or for extensional viscosity } \eta$$

$$(VI) \quad D = k/\eta$$

Substitution of VI into V we get

$$dN/dt = 4\pi r k n^\circ / \eta$$

Thus during periods close to those where  $D\Delta L/L\Delta t$  is large, the rate of vaporization,  $dN/dt$  is very depressed. Simultaneously the droplet is very rigid and solid like, and the surface is equally tense or hardened. When  $\eta$  subsequently approaches  $3\eta_0$  the rate of evaporation increases sharply. During the "hardened" phase both the surface tension and bulk viscosity of the droplet favor transport without coating or premature vaporization. This leads to improved performance in gasoline and diesel engines.

Another equation incorporates  $D$  and  $\gamma$ , or surface tension into the expression for the lifetime of a spherical droplet. This equation is,

$$(VII) \quad t = (r^2 n_L / 2 D n^\circ) (1 - 4\gamma / n_L r k_c T)$$

where  $n_L$  is the number of molecules per  $\text{cm}^3$  in the liquid and  $r$  is the radius of the droplet.

again replacing  $D$  by  $k/\eta$  we get

$$(VIII) \quad t = (r^2 \eta n_L) / (2kn^0) (1 - 4\gamma/n_L r k_c T)$$

In equation (VIII) the surface tension approaches 0 when saturated vapor is present above the droplet interface and  $\gamma$  approaches 0. The droplet lifetime equation (VIII) then becomes proportional to  $\eta$ .

#### Relaxation of PIB and $\eta$

$\eta$  becomes larger as  $\dot{\epsilon}$  increases. Similarly  $\eta$  decreases as  $\dot{\epsilon}$  approaches zero. When the stretching force is released the extensional or elongational viscosity decreases. The increase or decrease in  $\eta$  depends on the product of the strain  $\dot{\epsilon}$  and the reciprocal of the time it takes for the molecule to thermally relax to the unstretched conformation. The units of the stretching rate or elongation rate are reciprocal seconds and the units of polymer relaxation times are seconds. The product of the two factors is dimensionless. The following equation links  $\eta$  to  $\dot{\epsilon}$  and  $\theta$ .

$$\eta = 3\eta_0 + (3cRT/M) \sum_p (\theta_p^{-1} ((1 - 2\dot{\epsilon}\theta_p) (1 + \dot{\epsilon}\theta_p)))$$

$\theta_p$  is the relaxation time of the  $p$ th normal mode of chain response. Note that as  $\dot{\epsilon} \rightarrow 1/2\theta_1$  where  $\theta_1$  is the longest relaxation time, the  $(1 - 2\dot{\epsilon}\theta_1)$  term approaches zero. As the denominator approaches zero,  $\eta$  approaches infinite elongational viscosity. The relaxation time  $\theta_1$  is proportional to

$$[\eta] M \eta_0 / RT$$

where  $[\eta]$  is itself equal to  $KM^\alpha$  and  $\alpha$  is between 0.5 ( $\theta$  solvent = poor solvent) and 1.0 (perfect solvent). Thus the relaxation time is proportional to  $M^2 \eta_0 / RT$  in a good solvent. Relaxation time increases with  $M^2$  and therefore a state of high  $\eta$  will exist longer than it would for the case of a lower molecular weight. The mist particles would retain a high  $\eta$  for a desirable period during engine operation. Since  $\eta$  approaches  $\infty$  when  $\dot{\epsilon}$  approaches  $1/2\theta$  a high molecular weight polymer in a good solvent will develop an extensional viscosity that approaches infinity at

lower extension rates than a low molecular weight polymer in a poor solvent. The former is the case for the GTA fuel additive in gasoline and diesel fuels. Concentration then provides the final tuning for the desired  $\eta$  at a particular  $\epsilon$ .

#### Summary

1. The large  $\eta/\eta_0$  possible using low concentrations of  $7.2 \times 10^6$  M PIB in gasoline and diesel fuels influences pre-combustion events and thus affects combustion.
2. Through  $c$ ,  $\eta/\eta_0$  can be tuned to improve the operation of several types of engines.
3.  $\eta/\eta_0$  changes are linearly dependent on concentration and  $\Delta L/L\Delta t$  and this provides another dimension for engine tuning.
4. Through  $c$ ,  $\eta/\eta_0$  can be tuned to:
  - a) eliminate vapor-like burning
  - b) promote diffusive burning (diesel)
  - c) develop a uniform cloud mix for improved combustion (diesel)
  - d) improve volumetric efficiency by slightly delaying vaporization (2-stroke and 4-stroke spark ignition engines)
  - e) negate undesirable surface coating effects through momentary increases in surface and bulk rigidity
  - f) promote diesel fuel jet penetration prior to ignition and diffusive burning
  - g) decrease the extent of vapor explosion before TDC in a diesel
  - h) prevent droplet agglomeration and surface wetting after TDC in a diesel.

## **Attachment 8**

### **CARB Protocol Emissions Test Results**

## Summary of Data

Baseline Data No Viscon	Average .....Grams/BHP-HR.....							
	HP	HC	CO	Nox	NO	CO2	PM	Fuel
<b>Baseline</b>	136.362	1.379	1.695	5.79	5.144	561.255	0.265	178.507
<b>Pre Durability with Viscon</b>	136.332	1.504	1.827	6.036	5.084	556.206	0.202	177.106
<b>Post Durabilitywith Viscon</b>	131.05	1.43	1.62	5.89	5.43	552.58	0.197	175.80
<b>Average of Pre &amp; Post</b>	133.691	1.467	1.7235	5.963	5.257	554.393	0.1995	176.453
<b>% Change</b>	<b>-1.96%</b>	<b>6.38%</b>	<b>1.68%</b>	<b>2.99%</b>	<b>2.20%</b>	<b>-1.22%</b>	<b>-24.72%</b>	<b>-1.15%</b>



ISO 9001:2000 Registered

## **FINAL REPORT**

### **Engine and Fuel Testing to Measure Exhaust Emissions from a Model 3306 Caterpillar Diesel Engine**

When Operating Over the ISO 8178 8-Mode Off-Road  
Emission Test Cycle and When Operating With  
Viscon Treated Ultra Low Sulfur Diesel Fuel  
Meeting California ULSD Specifications

**Prepared for  
Mr. Mike Porter  
Viscon California  
Formerly GTAT California  
Bakersfield, CA**

**December 1, 2006  
Revised August 8, 2007  
Revised October 22, 2008**

**Prepared by  
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## **Introduction and Background**

This emission testing project is a follow-on project for Viscon California, formerly GTAT (Las Palmas Oil and Dehydration Company) using the same diesel engine that has already completed standard controlled substance emission testing in accordance with the CARB protocol entitled *Verification Procedure, Warranty and In-Use Strategies to Control Emissions from Diesel Engines as Described in the Final Regulation Order, Title 13, California Code of Regulations, Sections 2700 through 2710*.

Original emission testing and 1000 hours of durability operation with the same test engine was completed in 2004 with Viscon fuel treatment resulting in significant reductions of NOx and Particulates compared to original baseline emission measurements before fuel treatment with Viscon.

Subsequent to conclusion of the above testing project, CARB has specified that the Viscon treated diesel fuel must additionally be tested as an alternative fuel with detailed exhaust emission measurements of uncontrolled substances, including toxic hydrocarbons, PAH's and Carbonyls. This new emission report provides all of the CARB required data after operating the test engine and measuring emissions in exact accordance with CARB pre-approved testing and analysis protocols as described in this detailed report.

Since all of the previous emission and durability testing was conducted over two years ago the then available test fuel is no longer available. Accordingly, the same engine was operated and tested on commercially available ultra low sulfur diesel fuel that meets California ULSD specifications. Baseline emissions were again measured after 50 hours of stabilizing operation with the new ULSD test fuel. Subsequent stabilizing operation for 125 hours with the same fuel treated with Viscon was conducted and emissions were again measured for comparison to the new baseline data.

## **Test Engine**

The test engine for this project was a Model 3306 six cylinder Caterpillar diesel engine rated at 265 HP. This is the same engine (without modification) used in the prior emission testing to measure changes in exhaust emissions with the Viscon polymer additive admixed to the baseline no. 2 diesel fuel. The previous engine testing is described in an ETS (Olson Engineering, Inc.) summary report dated August 19, 2003 providing the data comparisons before durability testing and a second summary report (Olson-EcoLogic) dated November 26, 2004 covering the emission/fuel economy measurements after 1000 hours of engine durability operation with the Viscon treated fuel. Both reports are available upon request.



## Test Fuel

Previous testing with the specified test engine was done in 2003 and 2004 using commercially available no. 2 diesel fuel for the baseline and Viscon treated test fuel. This fuel met California fuel specifications that existed in the earlier time frame, but such fuel is no longer available in the California commercial market. Accordingly, all of the test work described in this report was done with one batch of commercially available no. 2 ultra low sulfur diesel fuel. This test fuel has a sulfur content of less than 15 ppm and other composition differences compared to the test fuel used in earlier years with this engine. Fuel specifications are provided in the Appendix.

## Test Protocol

As in all previous testing with the specified test engine the official test protocol was the ISO 8178 8-mode steady-state test as required for certification of heavy-duty diesel engines in off-road applications. This operating test protocol involves the collection and analysis of emissions from a hot-start while operating the engine over 8-modes of warmed up operation at specific loads and speeds under stabilized conditions. The detailed testing cycle is as follows:

Mode No.	Weighting Factor, %	RPM	Torque, %
1	15	Rated	100
2	15	Rated	75
3	15	Rated	50
4	10	Rated	10
5	10	Max torque, rpm	100
6	10	Max torque	75
7	10	Max torque	50
8	15	Idle	0

The engine was operated and emissions were recorded every second of each mode for five minutes to stabilize emissions. The last two minutes of each mode were recorded second-by-second and averaged to provide the stabilized steady-state emission/fuel economy results.

All engine exhaust for all modes passed through the Horiba Olson-Ecologic dilution tunnel. The dilution tunnel was fixed to operate at a total constant flow of 70 cubic meters per minute, providing variable exhaust dilution for each mode as a function of actual exhaust flow rates. Primary air was measured through the initial critical flow venturi (dilution air) and the total temperature corrected volume ( $V_{mix}$ ) of dilutant air plus exhaust was measured continuously at the second critical flow venturi. The calculated dilution ratio mode-by-mode is simply the temperature corrected composite total ( $V_{mix}$ ) divided by the exhaust flow ( $V_{mix}$  – dilution air). The measured dilution ratio is expected to be essentially a constant for the same mode for all tests, but the



actual dilution ratios were used as measured for each individual test to allow for test-to-test variance.

Gaseous emissions for each test mode were measured every tenth of a second for each five minutes of mode operation. Only the last two minutes of each mode were averaged to provide the stabilized diluted mode data. Dilute data from each mode was then multiplied by the specific mode dilution ratio to obtain the raw modal data and then multiplied by the appropriate mode weighting factor before adding all eight modes together. Finally, the total sum of the weighted modal data was divided by the weighted horsepower and by the test time to provide the weighted grams per bhp-hr for direct data comparisons to the official emission standards for controlled substances.

Particulate sampling involved initial calculation of exhaust mass flow per mode from an actual full 8-mode test. Secondary dilution ratios in the AVL Smart Sampler calculation were programmed for each mode and time of sampling was adjusted to properly compensate for specified modal weighting factors. Since the AVL PM Sampler draws its sample from the exhaust dilution tunnel for each mode the dilution ratio existing for each mode was included in the sample calculation to ultimately determine the total raw exhaust volume for proper calculation of the generated particulates in grams/bhp-hr. All particulate sampling resulted in the capture of measured and weighted particulates on a single filter media for each mode of each 8-mode test (eight complete particulate samples per test).

Exhaust sampling and subsequent gas chromatography analysis for uncontrolled toxic hydrocarbons (C2-C12) followed the detailed Standard Operating Procedure (S.O.P.) described in the Appendix. Modal sample collection was done for a total of 20 minutes. Modes 1, 2, 3 and 8 were collected for three minutes of the five minute mode, while modes 4, 5, 6 and 7 were collected for two minutes. Samples were drawn from each bag and analyzed by gas chromatography. The overall dilution ratio for the 8-modes (20 minutes of testing time) was used to correct and calculate the resulting G.C. data in grams/bhp-hr.  $((CONC^{ppb}/10^{-9})(V_{mix}, ft^3)(Density, g/ft^3)/Work, bhp-hr)$ . Simultaneously samples were drawn and captured for PAH and Carbonyl analysis at an off-site location (Desert Research Institute) as described in the Appended S.O.P. for this procedure.

### **Tasks Descriptions**

The specific tasks and order of operation were as follows:

1. Modified engine/dyno coupling and adapted engine to dynamometer.
2. Instrumented and MAP engine, verified test cycle and all calibrations – initiated engine testing.
3. Operated engine over a representative load cycle for 50 hours on the ULSD test fuel.



4. Conducted nine standard ISO 8178 8-mode baseline emission tests for PM and gaseous emissions simultaneously capturing dilute exhaust gas samples from three of the tests for in house measurement of toxic hydrocarbons by gas chromatography and for off-site (Desert Research Institute) analysis of PAH's and Carbonyls.
5. Added the Viscon product to the baseline ULS diesel fuel in the client specified concentrations and operated engine over the same load schedule as used in Task no. 3 for 125 hours to stabilize emissions.
6. Conducted several unofficial ISO 8178 8-mode tests during emission stabilizing operation to assure effectiveness and stabilization of the Viscon product. No samples were captured for toxic hydrocarbons, PAH's or Carbonyls during this task.
7. Conducted nine ISO 8178 8-mode standard emission tests with the Viscon treated fuel, again capturing triplicate samples for toxic hydrocarbons, PAH's and Carbonyls exactly as in Task no. 4.
8. Conducted quality audit functions for all data, calculated and reported changes in all measured emission caused by the Viscon treated fuel compared to the baseline fuel at a 95% confidence level. Prepared final report.

### **Test Facility, Equipment and Capability**

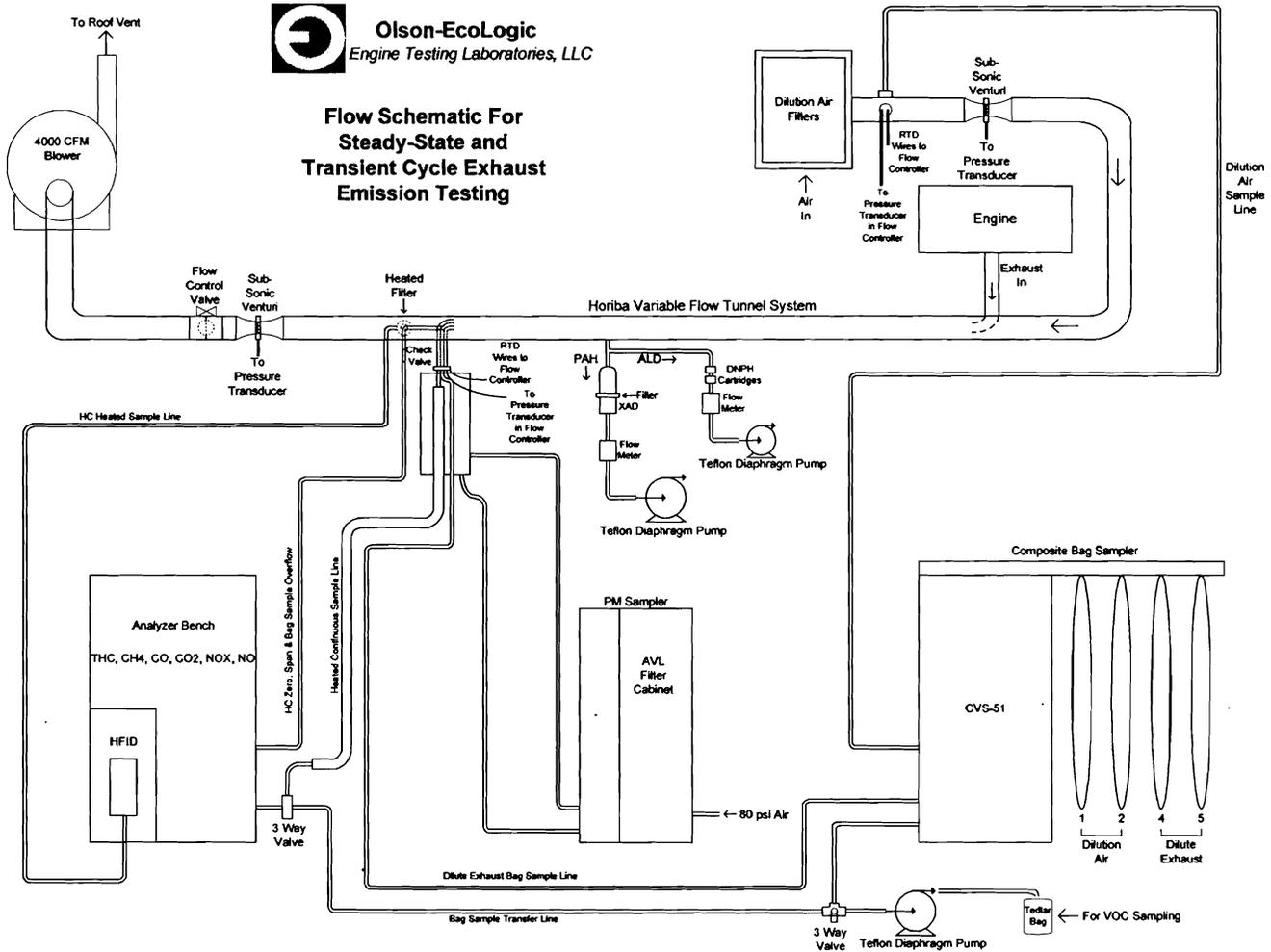
All testing discussed in this report was conducted at the Olson-EcoLogic heavy-duty engine test facility located in Fullerton, California. All initial engine operation and emission testing was conducted in the Olson-EcoLogic test cell 1. The test cell is equipped with a 450 horsepower full electric dynamometer, air conditioning and conventional sensors for continuous measurement of pressures, temperatures and air mass. State-of-the-art Horiba analyzers were used for dilute gas measurement of THC, CO, CO<sub>2</sub>, NO<sub>x</sub> and NO. An AVL Smart Particulate Sampler was used to capture particulates and fuel consumption was measured gravimetrically. The test cell operation is controlled by a fully integrated Labview based custom software program.

Heated sample lines were used to transport the exhaust gases to HC, NO<sub>x</sub> and NO analyzers. Calibrations with certified gases were routed to the analysers through the same sample lines used to sample the exhaust gases. In all cases the requirements of CFR 40 were followed. The particulate filters were conditioned for temperature and humidity before and after accumulation of particulates. Air mass to the engine was measured by a temperature corrected Sierra Air mass sensor as a secondary backup calibrated by the dilution tunnel venturi measurements of diluent air and total V<sub>mix</sub>. Engine intake air was controlled between 68 and 86 degrees F and fuel temperature was controlled to 100 degrees F +/- 10 degrees F as specified in CFR 40. A schematic of the complete exhaust gas sampling system is shown in the following figure.



**Olson-EcoLogic**  
Engine Testing Laboratories, LLC

### Flow Schematic For Steady-State and Transient Cycle Exhaust Emission Testing



The quality management system of Olson-EcoLogic is ISO 9001:2000 registered. The Company is officially recognized by EPA and CARB as a capable emission test facility for the protocols used in this project. All test activity was under the direction and responsibility of Donel R. Olson. Mr. Olson is a registered professional mechanical engineer with 50 years of experience in the measurement and analysis of engine emissions. Over twenty engine emission test laboratories in the United States and Germany have been under his ownership and direction during the past 35 years. Details of the Olson-EcoLogic emission test facility capability can be viewed and obtained on the Olson-EcoLogic website ([www.ecologiclabs.com](http://www.ecologiclabs.com)).

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[www.ecologiclabs.com](http://www.ecologiclabs.com)



## Other

Olson-EcoLogic Engine Testing Laboratories, LLC was acting as an independent contractor and not as agents or employees of the client in the conduct of work related to this project.

The Company does not guarantee the results of any test protocol, however, if repeat tests are not within acceptable variances Olson-EcoLogic on their own decision repeats the tests at no additional cost to the client.

## Test Results

All of the exhaust emission tests reported here are listed chronologically in Table 1.

After several preliminary tests to verify the engine emissions stability, "official" testing starting with test no. 8MLP14 using the reference fuel and finished with the ninth test, no. 8MLP22. This set of tests and the following sets are listed in Table 1.

Viscon additive at the normal concentration of 1oz per 20 gallons was admixed in the same fuel used to measure baseline emissions and several additional (unofficial) 8-mode tests were conducted during a 125 hour period of typical engine operation. This was intended to provide stability of exhaust emissions with the Viscon treated reference fuel.

Beginning with Viscon treated test no. 8MLP35, nine official tests were completed to compare to the reference fuel baseline data.

The third test series, starting with test no. 8MLP46, was a set of three heavy dosage tests with Viscon additive admixed to the reference fuel at ten times the normal dosage (10oz. per 20 gallons).

The fourth and final set of five tests, with reference fuel only (no Viscon) started with test no. 8MLP49, immediately after the data set with 10 times dosage of Viscon. Other than engine warm-up and fuel flushing, there was no significant engine operation prior to start of this data set. The purpose was to see any residual effect of the Viscon additive.

Concurrently with the above tests, as shown in Tables 2 and 3, data were collected for toxic compounds analysis including VOC's, PAH's and Carbonyls. Triplicate sets of data were obtained with the baseline reference fuel (test nos. 17, 18, and 19) and for comparison, triplicate sets of data were obtained for the Viscon treated reference fuel (test nos. 39, 40, and 41). In addition, VOC analyses (only) were conducted for test nos. 46 and 47, which were tests conducted with 10 times the normal Viscon dosage.



Table 2 provides the results of all VOC, PAH and Carbonyl data for the triplicate baseline sets compared to the triplicate sets of data obtained with the Viscon treated reference fuel.

Table 3 only provides the VOC data for duplicates of the Viscon heavy dosage tests (test nos. 46 and 47). No analysis of these data have been made for PAH and Carbonyl results.

### **Discussion of Results**

The measured effect of the Viscon additive on all controlled emissions can be seen in Tables 1 and 1A.

The product as demonstrated for the Caterpillar Model 3306 engine under steady-state operating conditions will reduce exhaust particulates to satisfy level 1 CARB criteria (25-50%). This result is consistent with earlier data accumulated on the same engine.

Additional data collection required by CARB involved two major expansions of the project.

- 1) Exhaust particulates were sampled for each mode of the 8-mode test protocol instead of composite sample collection of all 8 modes on one filter media and
- 2) Integrated samples over the 8-modes of testing were collected and analyzed for toxic hydrocarbons (VOC's), PAH's and Aldehydes . These specific testing protocols are described in the appendix.

### **Particulate Sampling and Analysis**

The PM sampling for each mode resulted in measurement of PM for eight specific conditions for each 8-mode test. To be consistent with the ISO 8-mode test protocol, the specific sampling for each mode was weighted by dilution ratio selection and time of sampling capture to match the ISO specific weighting factors. Accordingly, modes 1, 2, 3 and 8 were weighted 15% each and modes 4, 5, 6 and 7 were weighted 10% each to total 100% for all eight modes.

The analysis of the PM data was accomplished by weighing the captured PM for each mode and dividing that weight by sampling time and the horsepower generated during the mode to provide the weighted PM result in grams/hp-hr. For the idle mode, (mode 8) this calculation was not possible because hp at idle is zero. Therefore, the idle PM data is reported in grams/hour only. However, the idle PM weight does go into the calculation of total weighted grams/bhp-hr. The PM values from all 7 of the other operating modes



have been summed and tabulated in Tables 1 and 1A to show the total PM generated in grams/bhp-hr.

The PM measurements illustrate some very interesting reductions in PM caused by the Viscon additive. At the rated speed of 2200 RPM and the intermediate speed of 1400 RPM there is a clear relationship with load as shown below:

<b>Percent PM Reduction caused by Viscon</b>		
<b>Engine Load</b>	<b>2200 RPM</b>	<b>1400 RPM</b>
100%	15.3%	4.5%
75%	29.6%	17.3%
50%	28.0%	31.8%
10%	35.6%	--

Simultaneously idle PM (mode 8) was reduced 56% with the Viscon additive. This discovery can have specific importance in vehicle and engine operations that involve a significant amount of idling operation such as school busses, delivery vehicles, etc. In addition to the PM reduction, idle NOx emissions were also reduced over 10% as can be seen in Table 1.

The VOC, PAH and Carbonyl comparisons shown in Tables 2 and 3 for the same tests are somewhat of a mixed bag. For example, 1,3-butadiene is beyond the measurable data in the exhaust by the Viscon additive. As expected by the technical theory, the PAH's also show significant reductions with the Viscon fuel treatment. However, some increases were demonstrated for some VOC's and some Carbonyls, which may also be expected.

**Table 1**  
**Viscon California Data Summary Table**  
**Caterpillar 3306 Diesel Engine**  
 Testing Conducted by Olson-Ecologic Engine Testing Laboratories  
 Testing Conducted October 2006

FUEL	TEST NO.	Absolute			GRAMS PER BHP-HR							GRAMS PER BHP-HR							WTD. AVG. Total PM	GRAMS/HR (Idle PM Mode 6)	GRAMS/HR (Mode 8 NoG)
		Humidity	Wtd. Avg. BHP-HR	HC	CO	NOx	NO	PM	CO2	NMHC	PM Mode 1	PM Mode 2	PM Mode 3	PM Mode 4	PM Mode 5	PM Mode 6	PM Mode 7				
ULSD	8MLP14	58.2 - 60.1	135.15	1.33	1.68	5.80	5.26	179.42	564.32	0.96	0.129	0.183	0.232	3.094	0.342	0.237	0.234	0.289	20.54	27.31	
ULSD	8MLP15	50.8 - 56.1	138.21	1.41	1.72	5.88	5.20	178.66	561.61	0.70	0.122	0.145	0.251	2.106	0.373	0.261	0.248	0.250	2.06	28.49	
ULSD	8MLP16	48.1 - 51.0	138.84	1.41	1.89	5.90	5.12	179.05	562.91	0.72	0.108	0.127	0.251	2.209	0.355	0.261	0.241	0.240	2.08	17.11	
ULSD	8MLP17	54.7 - 58.4	136.55	1.34	1.71	5.26	4.82	178.53	561.42	0.48	0.122	0.143	0.263	2.221	0.427	0.318	0.292	0.284	14.39	18.36	
ULSD	8MLP18	57.5 - 59.1	136.93	1.38	1.70	5.75	5.11	178.67	561.75	0.72	0.121	0.145	0.254	2.430	0.392	0.281	0.314	0.297	30.85	25.37	
ULSD	8MLP19	52.7 - 55.3	136.62	1.40	1.71	5.76	5.05	178.71	561.78	0.72	0.127	0.135	0.207	2.211	0.410	0.265	0.226	0.276	28.72	23.95	
ULSD	8MLP20	51.4 - 53.3	136.77	1.36	1.63	5.85	5.11	178.17	560.36	0.71	0.118	0.153	0.241	2.322	0.352	0.236	0.241	0.273	26.70	17.65	
ULSD	8MLP21	55.8 - 58.4	135.71	1.36	1.73	5.93	5.27	177.44	557.85	0.71	0.112	0.141	0.228	2.108	0.348	0.241	0.243	0.248	12.34	27.76	
ULSD	8MLP22	60.9 - 64.5	136.68	1.42	1.68	5.98	5.36	177.92	559.30	0.79	0.102	0.161	0.210	1.917	0.312	0.208	0.124	0.231	18.51	26.47	
<b>AVERAGES (BASELINE)</b>			<b>136.362</b>	<b>1.379</b>	<b>1.695</b>	<b>5.790</b>	<b>5.144</b>	<b>178.507</b>	<b>561.255</b>	<b>0.722</b>	<b>0.118</b>	<b>0.148</b>	<b>0.237</b>	<b>2.291</b>	<b>0.368</b>	<b>0.256</b>	<b>0.240</b>	<b>0.265</b>	<b>17.128</b>	<b>23.630</b>	

Viscon	8MLP35	33.9 - 36.7	134.86	1.52	1.81	5.79	4.94	176.69	554.88	0.74	0.111	0.097	0.141	1.444	0.332	0.179	0.146	0.190	6.16	18.35
Viscon	8MLP36	37.3 - 41.9	136.04	1.53	1.82	5.87	5.00	176.84	554.67	0.73	0.087	0.087	0.141	1.404	0.320	0.163	0.131	0.170	2.06	17.74
Viscon	8MLP37	34.0 - 36.6	136.67	1.51	1.87	6.05	5.14	177.54	557.48	0.74	0.090	0.107	0.188	1.478	0.366	0.241	0.139	0.200	2.06	19.91
Viscon	8MLP38	32.5 - 35.3	137.01	1.52	1.86	6.27	5.19	176.43	553.96	0.77	0.100	0.100	0.178	1.625	0.352	0.224	0.124	0.197	2.05	16.07
Viscon	8MLP39	31.0 - 41.9	136.03	1.53	1.82	6.05	5.09	178.53	560.65	0.74	0.103	0.115	0.182	1.247	0.328	0.200	0.168	0.208	16.45	23.96
Viscon	8MLP40	34.5 - 43.2	136.77	1.47	1.84	6.20	5.09	177.76	558.39	0.72	0.097	0.102	0.175	1.439	0.382	0.228	0.182	0.211	10.26	27.18
Viscon	8MLP41	46.7 - 50.2	136.77	1.46	1.87	5.86	5.02	176.97	555.84	0.72	0.109	0.120	0.204	1.805	0.389	0.257	0.256	0.246	16.43	18.48
Viscon	8MLP42	47.7 - 53.3	136.92	1.47	1.81	6.17	5.22	177.19	556.80	0.71	0.096	0.094	0.163	1.513	0.391	0.224	0.183	0.210	10.28	17.51
Viscon	8MLP43	56.2 - 58.9	135.92	1.53	1.74	6.06	5.07	176.20	553.38	0.78	0.106	0.117	0.166	1.330	0.301	0.192	0.146	0.186	2.06	31.18
<b>AVERAGES (WITH VISCON)</b>			<b>136.332</b>	<b>1.504</b>	<b>1.827</b>	<b>6.036</b>	<b>5.084</b>	<b>177.106</b>	<b>556.206</b>	<b>0.737</b>	<b>0.100</b>	<b>0.105</b>	<b>0.171</b>	<b>1.476</b>	<b>0.351</b>	<b>0.212</b>	<b>0.164</b>	<b>0.202</b>	<b>7.534</b>	<b>21.153</b>

10X Viscon	8MLP46	61.4 - 63.2	136.95	1.49	1.77	6.01	5.00	176.22	553.50	0.71	0.104	0.122	0.181	1.856	0.373	0.269	0.197	0.236	18.48	22.64
10X Viscon	8MLP47	57.4 - 61.6	137.03	1.46	1.77	6.09	5.12	175.98	552.82	0.72	0.093	0.127	0.213	1.809	0.349	0.281	0.168	0.232	14.39	26.29
10X Viscon	8MLP48	56.2 - 59.7	136.88	1.46	1.77	6.06	5.09	175.93	552.67	0.73	0.102	0.122	0.169	1.331	0.350	0.294	0.219	0.235	24.68	25.89
<b>AVERAGES (WITH 10X VISCON)</b>			<b>136.953</b>	<b>1.471</b>	<b>1.769</b>	<b>6.053</b>	<b>5.070</b>	<b>176.043</b>	<b>552.997</b>	<b>0.720</b>	<b>0.099</b>	<b>0.124</b>	<b>0.188</b>	<b>1.599</b>	<b>0.357</b>	<b>0.281</b>	<b>0.195</b>	<b>0.234</b>	<b>19.183</b>	<b>24.940</b>

ULSD	8MLP49	54.4 - 60.1	136.99	1.44	1.66	6.06	5.09	176.99	556.30	0.75	0.084	0.124	0.150	1.364	0.345	0.216	0.139	0.210	22.61	40.75
ULSD	8MLP50	60.6 - 66.4	136.49	1.49	1.72	6.03	5.04	176.64	554.95	0.68	0.124	0.183	0.232	3.098	0.343	0.237	0.234	0.289	20.54	25.22
ULSD	8MLP51	63.1 - 64.6	136.33	1.50	1.76	6.12	5.01	176.32	553.80	0.68	0.108	0.140	0.235	2.029	0.392	0.306	0.226	0.279	30.83	32.35
ULSD	8MLP52	63.5 - 64.9	135.64	1.47	1.78	5.65	4.95	177.10	556.37	0.64	0.109	0.117	0.222	2.138	0.402	0.294	0.438	0.282	22.61	19.81
ULSD	8MLP53	59.3 - 64.5	136.11	1.48	1.82	6.04	5.01	177.42	557.28	0.68	0.111	0.137	0.194	2.067	0.402	0.310	0.255	0.278	30.82	32.71
<b>AVERAGES (BL AFTER VISCON)</b>			<b>136.312</b>	<b>1.476</b>	<b>1.748</b>	<b>5.980</b>	<b>5.020</b>	<b>176.894</b>	<b>555.740</b>	<b>0.686</b>	<b>0.107</b>	<b>0.140</b>	<b>0.207</b>	<b>2.139</b>	<b>0.376</b>	<b>0.272</b>	<b>0.258</b>	<b>0.268</b>	<b>25.483</b>	<b>30.168</b>



**Table 2**  
**Percent change of various emissions comparing ULSF to ULSF w/ Viscon - Caterpillar 3306 Diesel Engine (Units in g/bhp-hr)**  
 Testing Conducted by Olson-Ecologic Engine Testing Laboratories for Viscon California October 2006

	ULSF				ULSF w/ Viscon				Change
	8MLP17	8MLP18	8MLP19	Average	8MLP39	8MLP40	8MLP41	Average	
<b>VOC's</b>	(Analyzed via Gas Chromatography by Olson-EcoLogic Engine Testing Laboratories)								
1,3-butadiene	0.000245	0.000212	0.000300	<b>0.000252</b>	<RL	<RL	<RL	<RL	<b>-100%</b>
benzene	0.008692	0.008825	0.009560	<b>0.009026</b>	0.011935	0.014013	0.012897	<b>0.012948</b>	<b>43%</b>
toluene	0.004514	0.004058	0.004111	<b>0.004228</b>	0.005860	0.006042	0.006219	<b>0.006040</b>	<b>43%</b>
ethylbenzene	0.002038	0.001774	0.001621	<b>0.001811</b>	0.002025	0.002119	0.002183	<b>0.002109</b>	<b>16%</b>
m&p-xylene	0.004081	0.003440	0.004175	<b>0.003898</b>	0.002775	0.002875	0.003084	<b>0.002911</b>	<b>-25%</b>
o-xylene	0.002292	0.002320	0.002320	<b>0.002311</b>	0.001828	0.001766	0.001810	<b>0.001801</b>	<b>-22%</b>
<b>PAH's</b>	(Analyzed via Gas Chromatography/Mass Spectrometry by Desert Research Institute)								
naphthalene	0.0000125192	0.0000071081	0.0000119718	<b>0.0000105330</b>	0.0000051731	0.0000046456	0.0000039993	<b>0.0000046060</b>	<b>-56%</b>
2-methylnaphthalene	0.0000112961	0.0000084828	0.0000095142	<b>0.0000097644</b>	0.0000024317	0.0000027416	0.0000025569	<b>0.0000025767</b>	<b>-74%</b>
acenaphthylene	0.0000002048	0.0000001814	0.0000001930	<b>0.0000001930</b>	0.0000001683	0.0000001606	0.0000001573	<b>0.0000001621</b>	<b>-16%</b>
acenaphthene	0.0000005852	0.0000006578	0.0000007354	<b>0.0000006595</b>	0.0000000615	0.0000000706	0.0000000610	<b>0.0000000644</b>	<b>-90%</b>
fluorene	0.0000005526	0.0000005425	0.0000005102	<b>0.0000005351</b>	0.0000001376	0.0000001311	0.0000001238	<b>0.0000001308</b>	<b>-76%</b>
phenanthrene	0.0000013504	0.0000013821	0.0000013007	<b>0.0000013444</b>	0.0000004623	0.0000005479	0.0000004993	<b>0.0000005032</b>	<b>-63%</b>
anthracene	0.0000001898	0.0000001834	0.0000001699	<b>0.0000001810</b>	0.0000000647	0.0000000816	0.0000000780	<b>0.0000000748</b>	<b>-59%</b>
fluoranthene	0.0000000719	0.0000000720	0.0000000579	<b>0.0000000673</b>	0.0000000559	0.0000000599	0.0000000350	<b>0.0000000503</b>	<b>-25%</b>
pyrene	0.0000002207	0.0000002166	0.0000002438	<b>0.0000002270</b>	0.0000001584	0.0000001207	0.0000000982	<b>0.0000001258</b>	<b>-45%</b>
benz(a)anthracene	0.0000000074	0.0000000084	0.0000000070	<b>0.0000000076</b>	0.0000000025	0.0000000015	0.0000000006	<b>0.0000000015</b>	<b>-80%</b>
chrysene-triphenylene	0.0000000108	0.0000000105	0.0000000141	<b>0.0000000118</b>	0.0000000073	0.0000000045	0.0000000042	<b>0.0000000054</b>	<b>-55%</b>
benzo(b+j)fluoranthene	0.0000000016	0.0000000006	0.0000000006	<b>0.0000000010</b>	0.0000000000	0.0000000000	0.0000000010	<b>0.0000000003</b>	<b>-67%</b>
benzo(k)fluoranthene	0.0000000005	0.0000000002	0.0000000003	<b>0.0000000003</b>	0.0000000002	0.0000000000	0.0000000003	<b>0.0000000002</b>	<b>-50%</b>
BeP	0.0000000013	0.0000000006	0.0000000006	<b>0.0000000009</b>	0.0000000005	0.0000000005	0.0000000006	<b>0.0000000005</b>	<b>-37%</b>
BaP	0.0000000002	0.0000000000	0.0000000010	<b>0.0000000004</b>	0.0000000000	0.0000000000	0.0000000000	<b>0.0000000000</b>	<b>-100%</b>
perylene	0.0000000006	0.0000000000	0.0000000002	<b>0.0000000003</b>	0.0000000000	0.0000000000	0.0000000000	<b>0.0000000000</b>	<b>-100%</b>
indeno[123-cd]pyrene	0.0000000004	0.0000000000	0.0000000000	<b>0.0000000001</b>	0.0000000002	0.0000000000	0.0000000000	<b>0.0000000001</b>	<b>-50%</b>
dibenzo(ah+ac)anthracene	0.0000000002	0.0000000000	0.0000000000	<b>0.0000000001</b>	0.0000000000	0.0000000000	0.0000000007	<b>0.0000000002</b>	<b>299%</b>
benzo(ghi)perylene	0.0000000000	0.0000000000	0.0000000000	<b>0.0000000000</b>	0.0000000000	0.0000000000	0.0000000004	<b>0.0000000001</b>	

**Table 2 (cont.)**

	<i>ULSF</i>			<i>Average</i>	<i>ULSF w/ Viscon</i>			<i>Average</i>	<i>Change</i>
	<i>8MLP17</i>	<i>8MLP18</i>	<i>8MLP19</i>		<i>8MLP39</i>	<i>8MLP40</i>	<i>8MLP41</i>		
<b>Carbonyls</b>	(Analyzed via High Performance Liquid Chromatography by Desert Research Institute)								
formaldehyde	0.027876	0.026773	0.027121	<b>0.027257</b>	0.035915	0.032348	0.032137	<b>0.033467</b>	<b>23%</b>
acetaldehyde	0.007410	0.007211	0.007535	<b>0.007385</b>	0.009786	0.009169	0.008887	<b>0.009281</b>	<b>26%</b>
acetone	0.002020	0.001754	0.001827	<b>0.001867</b>	0.002895	0.002536	0.002490	<b>0.002640</b>	<b>41%</b>
acrolein	0.000813	0.001405	0.000857	<b>0.001025</b>	0.002133	0.001474	0.001846	<b>0.001817</b>	<b>77%</b>
propionaldehyde	0.001346	0.001304	0.001346	<b>0.001332</b>	0.001651	0.001444	0.001443	<b>0.001512</b>	<b>14%</b>
crotonaldehyde	0.000904	0.001060	0.001027	<b>0.000997</b>	0.001260	0.001122	0.001175	<b>0.001186</b>	<b>19%</b>
methyl ethyl ketone	0.000527	0.000410	0.000448	<b>0.000462</b>	0.000484	0.000437	0.000518	<b>0.000480</b>	<b>4%</b>
methacrolein	0.000474	0.000491	0.000377	<b>0.000447</b>	0.000635	0.000500	0.000552	<b>0.000562</b>	<b>26%</b>
butyraldehyde	0.004892	0.004370	0.005015	<b>0.004759</b>	0.004111	0.003893	0.004090	<b>0.004031</b>	<b>-15%</b>
benzaldehyde	0.000631	0.000617	0.000652	<b>0.000633</b>	0.001054	0.000536	0.000583	<b>0.000724</b>	<b>14%</b>
glyoxal	0.000404	0.000134	0.000429	<b>0.000322</b>	0.000644	0.000631	0.000731	<b>0.000669</b>	<b>108%</b>
valeraldehyde	0.000770	0.000515	0.000567	<b>0.000617</b>	0.000590	0.000424	0.000475	<b>0.000497</b>	<b>-20%</b>
m-tolualdehyde	0.000012	0.000025	0.000012	<b>0.000016</b>	0.000126	0.000101	0.000101	<b>0.000110</b>	<b>570%</b>
hexanaldehyde	0.000350	0.000292	0.000305	<b>0.000316</b>	0.000453	0.000382	0.000270	<b>0.000368</b>	<b>17%</b>

**Table 3**

**Percent change of various emissions comparing ULSF to ULSF w/ 10x Viscon - Caterpillar 3306 Diesel Engine (Units in g/bhp-hr)**

Testing Conducted by Olson-Ecologic Engine Testing Laboratories for Viscon California October 2006

	<i>ULSF</i>			<i>Average</i>	<i>ULSF w/ 10x Viscon</i>		<i>Average</i>	<i>Change</i>
	<i>8MLP17</i>	<i>8MLP18</i>	<i>8MLP19</i>		<i>8MLP46</i>	<i>8MLP47</i>		
<b>VOC's</b>	(Analyzed via Gas Chromatography by Olson-EcoLogic Engine Testing Laboratories)							
1,3-butadiene	0.000245	0.000212	0.000300	<b>0.000252</b>	<RL	<RL	<RL	<b>-100%</b>
benzene	0.008692	0.008825	0.009560	<b>0.009026</b>	0.011786	0.010998	<b>0.011392</b>	<b>26%</b>
toluene	0.004514	0.004058	0.004111	<b>0.004228</b>	0.005157	0.005194	<b>0.005176</b>	<b>22%</b>
ethylbenzene	0.002038	0.001774	0.001621	<b>0.001811</b>	0.001969	0.001515	<b>0.001742</b>	<b>-4%</b>
m&p-xylene	0.004081	0.003440	0.004175	<b>0.003898</b>	0.002573	0.002435	<b>0.002504</b>	<b>-36%</b>
o-xylene	0.002292	0.002320	0.002320	<b>0.002311</b>	0.001691	0.001706	<b>0.001699</b>	<b>-26%</b>



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**OLSON-ECOLOGIC ENGINE TESTING LABORATORY  
GTAT ISO-8178 8-MODE TESTING OF VISCON POLYMER**

**8MLP17 (MULTI-FILTER)**

**BASELINE**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P In-Hga	W/CAL FACTOR	KNOx PPM	Fuelln degF	KNO PPM	Methane ppm	NMHC ppm	Humidy %	Oil P paig	nHnC RATIO
1	2200.0	604.9	253.4	5.80	183.08	610.16	557.56	406.71	741	500.4	911.3	54.7	78.6	30.00	0.027	579.54	103.4	529.58	322.98	83.72	37.74	49.66	1.8
2	2200.0	469.6	196.7	5.49	109.75	550.34	513.57	442.73	583.5	426.0	850.1	56.0	79.8	30.00	0.026	524.46	106.0	489.41	282.88	159.85	37.26	48.55	1.8
3	2200.0	314.7	131.8	4.98	118.62	424.16	389.13	537.02	413.5	346.9	747.7	56.7	79.8	30.00	0.024	404.88	107.3	371.42	279.48	257.54	37.59	50.18	1.8
4	2200.0	61.8	25.9	3.16	228.07	161.15	112.34	792.96	170	258.2	516.7	56.7	79.7	30.00	0.015	153.85	107.7	107.24	415.55	377.42	37.78	52.67	1.8
5	1399.9	789.6	210.4	7.74	912.76	708.90	665.50	169.01	568.5	272.9	1033.4	56.9	79.5	30.00	0.036	677.05	104.8	635.60	169.01	95.85	38.18	36.02	1.8
6	1400.0	578.6	154.2	7.58	657.07	689.29	650.04	291.77	413.5	218.6	962.0	57.2	79.7	30.00	0.035	658.80	104.8	621.29	291.77	53.88	38.16	37.19	1.8
7	1400.0	382.2	101.9	6.86	262.26	594.44	536.12	434.65	279.5	177.2	806.2	57.4	79.7	30.00	0.032	568.36	104.9	512.60	388.81	45.84	38.19	40.04	1.8
8	590.5	0.7	0.0	5.12	287.57	307.97	290.65	706.42	16.25	54.3	338.1	58.4	79.8	30.00	0.024	295.19	98.8	278.60	706.42	535.76	38.70	20.11	1.8

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	311.05	281.45	1463.88	1329.9143	44460	1687536	140113.88	64.4	0.09
15.00	2	281.88	140.45	1102.66	1026.3731	35010	1401200	110343.47	102.3	0.07
15.00	3	266.25	118.21	662.83	609.5737	24810	1086266	77956.07	128.4	0.08
10.00	4	249.76	144.39	160.02	113.71427	10200	678946	31456.06	120.4	0.30
10.00	5	74.02	803.57	979.25	914.64182	34110	983296	107092.87	42.0	0.06
10.00	6	95.12	430.57	709.23	667.78798	24810	730829	78005.74	17.6	0.06
10.00	7	105.97	128.53	457.60	413.62956	16770	543164	52850.70	11.2	0.10
15.00	8	13.31	10.89	18.36	17.563971	975	41333	3044.77	10.1	0.06

**WTD AVG BHP = 136.53      KW = 101.81**

	HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC
WTD AVG GM/H =	183.36	233.36	717.74	658	24377	926074	76659	64.91
<b>AVG GM/BHPH =</b>	<b>1.34</b>	<b>1.71</b>	<b>5.26</b>	<b>4.82</b>	<b>178.54</b>		<b>561.47</b>	<b>0.48</b>
AVG GM/KWH =	1.80	2.29	7.05	6.47	239.43		752.94	0.64

W.F.	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.15
MODE =	1	2	3	4	5	6	7	8
PARTIC. WT, MG =	1.20	0.87	0.84	0.60	1.83	0.78	0.40	0.07
Exh.Probe Mass Flow Rate (g/Sec.)=	1.087	0.868	0.682	0.441	0.859	0.671	0.567	0.137
Sample Time (Sec.)=	180	180	180	120	120	120	120	180
Dil.Exh.Sample Mass-Part.Filter (kg) =	0.196	0.156	0.123	0.053	0.103	0.081	0.068	0.025
Dil.Tunnel Tot Flow Temp.Corr. (scmm)=	65.04	65.31	65.31	65.37	65.20	65.24	65.24	65.34
Dil.Tunnel Tot Mass Flow (kg/Hr.)=	5045.96	5066.95	5067.04	5071.25	5058.16	5061.63	5061.23	5068.85
Partic.Mass Flow Rate (g/Hr.)=	30.9473	28.2146	34.6718	57.4971	89.7985	49.0322	29.7544	14.3884
<b>GM/BHPH=</b>	<b>0.122</b>	<b>0.143</b>	<b>0.263</b>	<b>2.221</b>	<b>0.427</b>	<b>0.318</b>	<b>0.292</b>	

WTD AVG GM/H = 38.842

WT AVG NO2/NOX RATIO = 64.90954311

**WEIGHTED AVG GM/BHPH = 0.284**

WTD AVG GM/KWH = 0.381





**OLSON-ECOLOGIC ENGINE TESTING LABORATORY  
GTAT ISO-8178 8-MODE TESTING OF VISCON POLYMER  
8MLP20 (MULTI-FILTER)**

**BASELINE**

Mode	EngSpd RPM	DynTrq lb-R	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	f/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Humidy %	Oil P psig
1	2200.0	607.7	254.6	5.80	170.27	674.00	581.66	399.96	744.5	504.6	906.8	52.1	73.8	30.00	0.027	636.12	101.5	549.0	208.5	191.4	42.3	52.4
2	2200.0	469.8	196.8	5.45	105.04	601.26	534.63	440.22	581.5	430.8	838.1	51.8	74.5	30.00	0.028	567.07	103.9	504.2	179.1	281.1	41.0	49.9
3	2200.1	314.9	132.0	4.93	119.05	480.74	422.35	534.65	413.5	352.0	741.3	51.4	74.8	30.00	0.023	452.94	105.4	397.9	182.9	351.8	40.3	50.2
4	2200.0	62.1	26.0	3.17	234.83	185.73	142.53	781.19	172	259.5	511.5	51.5	74.6	30.00	0.015	175.04	104.9	134.3	281.4	499.7	40.7	53.1
5	1400.0	789.4	210.4	7.59	867.19	800.42	701.27	198.73	565	273.9	1020.2	51.5	74.2	30.00	0.038	754.35	100.0	660.9	194.9	3.8	41.2	36.1
6	1399.9	578.7	154.2	7.47	599.19	798.58	708.81	305.83	412	218.9	953.8	51.4	74.5	30.00	0.035	752.33	101.2	667.8	247.2	58.7	40.7	37.2
7	1399.9	382.2	101.9	6.71	240.74	686.22	618.37	449.19	278	175.8	799.9	51.4	74.6	30.00	0.031	646.50	100.5	582.6	268.9	180.3	40.6	39.7
8	592.3	0.9	0.1	5.99	292.71	343.90	268.33	830.5	16.75	53.8	376.8	53.3	75.2	30.00	0.028	325.52	98.1	254.0	749.0187	81.4	41.2	19.2

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	307.59	263.21	1615.48	1385.9814	44670	1696251	140822.01	147.7	0.14
15.00	2	281.24	134.89	1196.35	1061.047	34890	1404738	109972.20	167.4	0.11
15.00	3	267.70	119.82	748.90	659.5425	24810	1095767	77948.96	176.8	0.12
10.00	4	248.77	150.31	184.07	143.93756	10320	885974	31831.90	160.5	0.23
10.00	5	88.23	773.85	1105.90	964.32358	33900	994629	106426.17	1.7	0.12
10.00	6	100.82	397.05	819.02	725.94047	24720	737748	77753.88	19.4	0.11
10.00	7	111.39	120.00	529.43	478.28989	16680	551283	52560.46	44.9	0.10
15.00	8	13.79	9.77	17.85	14.115439	1005	36735	3140.52	1.4	0.22

<b>WTD AVG BHP =</b>	<b>136.77</b>	<b>KW =</b>	<b>101.99</b>																			
	HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC														
WTD AVG GM/H =	185.47	223.27	800.63	699	24368	931987	76640	96.64														
<b>AVG GM/BHPH =</b>	<b>1.36</b>	<b>1.63</b>	<b>5.85</b>	<b>5.11</b>	<b>178.17</b>		<b>560.36</b>	<b>0.71</b>														
AVG GM/KWH =	1.82	2.19	7.85	6.86	238.93		751.45	0.95														

WT AVG NO2/NOX RATIO = 0.145129416

W.F.	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10	0.15
MODE =	1	2	3	4	5	6	7	8	
PARTIC. WT, MG =	1.16	0.93	0.77	0.63	1.51	0.58	0.33	0.13	
Exh.Probe Mass Flow Rate (g/Sec.)=	1.087	0.868	0.682	0.441	0.859	0.671	0.567	0.137	
Sample Time (Sec.)=	180	180	180	120	120	120	120	180	
Dil.Exh.Sample Mass-Part.Filter (kg) =	0.20	0.16	0.1228	0.05	0.10	0.08	0.07	0.02	
Dil.Tunnel Tot Flow Temp.Corr. (scmm)=	65.09	65.30	65.36	65.36	65.21	65.25	65.25	65.29	
Dil.Tunnel Tot Mass Flow (kg/Hr.)=	5049.5	5066.3	5070.8	5070.7	5058.7	5082.4	5082.2	5085.2	
Partic.Mass Flow Rate (g/Hr.)=	29.937	30.156	31.806	60.366	74.103	36.466	24.552	26.702	
<b>GM/BHPH=</b>	<b>0.118</b>	<b>0.153</b>	<b>0.241</b>	<b>2.322</b>	<b>0.352</b>	<b>0.236</b>	<b>0.241</b>		
WTD AVG GM/H =	37.339								
<b>WEIGHTED AVG GM/BHPH =</b>	<b>0.273</b>								
WTD AVG GM/KWH =	0.366								









**OLSON-ECOLOGIC ENGINE TESTING LABORATORY  
GTAT ISO-8178 8-MODE TESTING OF VISCON POLYMER  
8MLP37 (MULTI-FILTER)**

Mode	w/Viscon											KNOX PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Humidy %	Oil P psig	nH/nC RATIO	Exh. P psig				
	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F										ABSHUM GR/LB	Air In degF	Baro P In-Hga	l/aCAL FACTOR
1	2200.0	606.6	254.1	5.82	219.44	735.47	608.13	440.35	744.5	482.7	923.3	36.6	77.2	30.10	0.027	668.74	96.6	553.0	240.0	200.3	26.7	53.1	1.8	0
2	2200.0	469.9	196.8	5.49	121.71	674.84	572.11	488.09	578	405.8	863.3	36.6	79.7	30.10	0.026	613.66	99.1	520.2	223.8	264.3	24.6	51.0	1.8	0
3	2200.0	314.8	131.9	4.93	132.16	516.12	449.27	607.41	408.5	334.1	760.7	36.6	80.6	30.10	0.023	469.24	99.6	408.5	226.1	381.3	23.9	51.1	1.8	0
4	2200.0	61.9	25.9	3.06	256.47	185.14	139.11	868.51	166.5	250.3	516.0	36.2	80.5	30.10	0.015	168.16	100.0	126.4	343.4	525.1	23.7	52.7	1.8	0
5	1400.0	789.5	210.4	7.56	900.41	824.72	710.87	228.23	568.5	261.3	1056.5	36.3	80.7	30.10	0.035	749.31	97.6	645.9	192.6	35.7	23.6	36.4	1.8	0
6	1400.1	578.1	154.1	7.43	722.96	834.44	731.05	348.52	413.5	210.7	982.8	34.8	80.9	30.10	0.035	755.47	98.4	661.9	307.3	41.2	22.5	38.9	1.8	0
7	1400.0	382.0	101.8	6.66	272.33	714.93	653.33	505.30	275.5	172.2	818.7	34.0	80.1	30.10	0.031	645.99	98.1	590.3	329.8	175.5	22.5	41.1	1.8	0
8	583.7	1.3	0.1	4.34	435.78	314.61	259.84	1122.590008	15.75	52.8	387.7	34.4	79.5	30.10	0.021	284.55	91.2	235.0	1045.3	77.3	23.2	17.7	1.8	0

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	337.12	337.68	1690.66	1390.7494	44670	1736437	140611.74	154.0	0.17
15.00	2	307.14	153.94	1275.15	1079.2746	34680	1423277	109191.96	167.0	0.15
15.00	3	299.93	131.17	765.16	668.71307	24510	1111259	76874.27	189.2	0.13
10.00	4	275.46	163.50	176.12	135.32772	9990	701778	30676.32	168.4	0.25
10.00	5	102.27	810.99	1108.77	951.48078	34110	1031981	106992.00	16.0	0.14
10.00	6	115.68	482.34	828.05	724.83233	24810	762078	77859.47	13.7	0.12
10.00	7	124.96	135.38	527.55	483.74381	16530	563588	52015.91	43.8	0.09
15.00	8	23.79	18.56	19.91	16.862798	945	47421	2904.10	1.7	0.17

<b>WTD AVG BHP =</b>	<b>136.67</b>	<b>KW =</b>	<b>101.92</b>	W.F.	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10	0.15				
				MODE =	1	2	3	4	5	6	7	8					
				<b>PARTIC. WT, MG =</b>	0.89	0.65	0.60	0.40	1.57	0.59	0.19	0.01					
				Exh.Probe Mass Flow Rate (g/Sec.)=	1.087	0.868	0.682	0.441	0.859	0.671	0.567	0.137					
				Sample Time (Sec.)=	180	180	180	120	120	120	180						
				Dil.Exh.Sample Mass-Part.Filter (kg) =	0.20	0.16	0.1228	0.05	0.10	0.08	0.07	0.02					
WTD AVG GM/H =	207.03	255.42	826.68	703	24265	953702	76192	100.94	Dil.Tunnel Tot Flow Temp.Corr. (scmm)=	65.06	65.32	65.35	65.36	65.18	65.23	65.24	65.34
<b>AVG GM/BHPH =</b>	<b>1.51</b>	<b>1.87</b>	<b>6.05</b>	<b>5.14</b>	<b>177.54</b>	<b>557.48</b>	<b>0.74</b>		Dil.Tunnel Tot Mass Flow (kg/Hr.)=	5047.19	5067.19	5069.71	5070.63	5056.95	5060.45	5061.65	5069.22
									Partic.Mass Flow Rate (g/Hr.)=	22.958	21.081	24.779	38.327	77.022	37.080	14.135	2.056
AVG GM/KWH =	2.03	2.51	8.11	6.90	238.09	747.60	0.99		<b>GM/BHPH=</b>	0.090	0.107	0.188	1.478	0.366	0.241	0.139	
									WTD AVG GM/H =	27.287							
WT AVG NO2/NOX RATIO =	0.154022224								<b>WEIGHTED AVG GM/BHPH =</b>	<b>0.200</b>							
									WTD AVG GM/KWH =	0.268							























**OLSON-ECOLOGIC ENGINE TESTING LABORATORY  
GTAT ISO-8178 8-MODE TESTING OF VISCON POLYMER  
8MLP51 (MULTI-FILTER)**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	f/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Humidy %	Oil P psig
1	2200.0	801.4	251.9	5.61	189.00	879.15	539.90	418.00	725	489.4	908.7	83.5	74.5	30.20	0.026	659.44	95.4	524.24	248.9	169.1	50.6	52.9
2	2200.0	470.0	196.9	5.27	104.41	622.08	509.48	457.53	576.5	413.4	851.6	64.6	76.1	30.20	0.025	605.75	97.6	496.10	220.8	236.7	48.7	50.5
3	2200.0	314.4	131.7	4.71	114.20	478.77	391.61	562.81	408	337.8	748.0	63.9	76.4	30.20	0.022	463.41	98.4	380.64	224.2	338.6	47.7	51.5
4	2200.0	82.1	28.0	2.83	225.99	175.24	125.14	805.65	167	254.9	509.0	63.5	76.4	30.20	0.014	170.17	98.7	121.52	348.4	457.2	47.4	54.1
5	1400.1	789.1	210.4	7.35	911.36	731.41	629.62	241.34	566.5	266.7	1036.4	63.1	76.2	30.20	0.034	709.50	95.9	610.76	200.6	40.8	47.4	38.4
6	1399.9	578.3	154.1	7.08	626.44	742.21	620.91	339.94	411.5	214.0	963.0	63.6	76.8	30.20	0.033	720.80	96.3	603.00	311.7	28.2	46.8	40.1
7	1400.0	382.3	101.9	6.29	225.08	635.84	545.28	456.98	276	173.6	802.2	63.2	77.0	30.20	0.030	616.92	96.1	529.06	319.7	137.3	46.2	42.8
8	576.3	1.2	0.1	3.07	267.58	334.14	247.38	1147.15	16	52.3	385.3	64.0	77.4	30.20	0.015	324.87	91.2	240.51	982.9	164.3	46.3	18.6

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	323.43	293.94	1684.94	1332.2618	43500	1706412	136999.00	131.4	0.21
15.00	2	299.26	137.27	1308.38	1069.1714	34590	1438496	108956.51	155.5	0.18
15.00	3	289.43	118.05	786.96	648.52335	24360	1125152	76450.55	175.0	0.18
10.00	4	277.84	156.65	193.78	141.46758	10020	741838	30775.08	159.6	0.29
10.00	5	110.80	841.02	1075.65	921.93577	33990	1028610	106535.86	18.8	0.14
10.00	6	117.92	436.81	825.71	690.07195	24690	774978	77541.90	9.8	0.16
10.00	7	119.93	118.73	534.64	459.80621	16560	582225	52153.47	36.2	0.14
15.00	8	34.59	16.22	32.35	24.821474	960	65333	2921.45	5.1	0.26

WTD AVG BHP =

136.33 KW = 101.66

WTD AVG GM/H =

204.65 HC 240.14 CO 834.87 KNOX 683 KNO 24038 FUEL 963074 EXHAUST 75500 CO2 92.48 NMHC

AVG GM/BHPH =

1.50 1.76 6.12 5.01 176.32 553.80 0.68

AVG GM/KWH =

2.01 2.36 8.21 6.71 236.45 742.66 0.91

WT AVG NO2/NOX RATIO = 0.196737998

W.F.	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10	0.15
MODE =	1	2	3	4	5	6	7	8	
PARTIC. WT, MG =	1.06	0.85	0.75	0.55	1.68	0.75	0.31	0.15	
Exh.Probe Mass Flow Rate (g/Sec.)=	1.09	0.87	0.6820	0.44	0.86	0.67	0.57	0.14	
Sample Time (Sec.)=	180	180	180	120	120	120	120	180	
Dil.Exh.Sample Mass-Part.Filter (kg) =	0.20	0.16	0.1228	0.05	0.10	0.08	0.07	0.02	
Dil.Tunnel Tot Flow Temp.Corr. (scmm)=	65.04	65.24	65.39	65.37	65.17	65.22	65.21	65.34	
Dil.Tunnel Tot Mass Flow (kg/Hr.)=	5045.73	5061.35	5072.67	5071.56	5055.83	5060.13	5059.29	5069.18	
Partic.Mass Flow Rate (g/Hr.)=	27.34	27.54	30.99	52.71	82.40	47.13	23.05	30.83	
GM/BHPH=	0.108	0.140	0.235	2.029	0.392	0.306	0.226		
WTD AVG GM/H =	38.034								
WEIGHTED AVG GM/BHPH =	0.279								
WTD AVG GM/KWH =	0.374								

**OLSON-ECOLOGIC ENGINE TESTING LABORATORY  
GTAT ISO-8178 8-MODE TESTING OF VISCON POLYMER  
8MLP52 (MULTI-FILTER)**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMes scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	f/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Humidy %	Oil P psig
1	2200.0	590.0	247.2	5.64	186.42	643.11	537.92	423.07	720	479.4	905.7	64.2	77.7	30.20	0.027	625.61	99.4	523.27	259.5	163.6	45.9	53.6
2	2200.0	469.8	196.8	5.37	109.45	573.52	507.17	461.02	578	409.3	857.2	64.2	78.8	30.20	0.025	557.91	101.6	493.37	233.5	227.5	44.3	51.3
3	2200.0	315.3	132.1	4.87	129.14	444.01	396.87	572.82	409	333.3	754.9	64.9	79.9	30.20	0.023	432.60	102.2	386.88	240.2	332.6	43.1	51.9
4	2200.0	62.0	28.0	3.02	244.49	155.37	124.79	828.69	167	252.7	512.8	64.1	79.9	30.20	0.015	151.10	102.1	121.36	361.5	467.2	42.7	53.4
5	1400.0	789.0	210.3	7.55	914.79	708.34	636.93	239.95	567.5	264.6	1045.4	63.6	79.5	30.20	0.035	687.92	99.2	618.57	183.8	56.1	42.8	38.1
6	1400.0	577.7	154.0	7.34	668.65	700.70	640.72	326.97	413	212.3	972.8	63.7	80.1	30.20	0.034	680.63	99.8	622.38	318.8	8.1	42.0	39.8
7	1400.0	382.2	101.9	6.54	234.50	604.31	563.76	467.79	275.5	170.4	809.2	63.5	80.4	30.20	0.031	586.82	100.5	547.44	333.6	134.1	41.6	42.1
8	574.6	1.2	0.1	3.96	291.61	288.59	236.68	946.53	15.5	50.5	387.7	64.0	80.6	30.20	0.019	261.10	95.2	230.09	946.5	0.0	41.5	18.3

WT. FAC %	Mode No	GRAMS/HOUR				GRAMS/HOUR				NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	323.26	286.31	1578.52	1313.4707	43200	1685740	136056.48	125.6	0.16
15.00	2	295.69	141.10	1181.84	1042.7405	34560	1411710	108866.26	146.5	0.12
15.00	3	286.60	129.87	714.74	640.99105	24540	1096429	77013.94	167.2	0.11
10.00	4	267.28	158.51	180.94	132.0557	10020	895116	30805.49	152.5	0.20
10.00	5	107.48	823.68	1017.60	911.11791	34050	1005615	106764.58	25.2	0.10
10.00	6	109.76	451.17	754.50	689.12641	24780	751660	77831.63	2.7	0.09
10.00	7	117.86	118.76	488.24	456.80825	16530	560215	52064.45	34.0	0.07
15.00	8	21.74	13.46	19.81	17.860163	930	49914	2870.84	0	0.12

WTD AVG BHP =

**135.64      KW =    101.15**

WTD AVG GM/H =

199.33    240.82    766.34    671    24023    937830    75468    87.34

AVG GM/BHPH =

**1.47    1.78    5.65    4.95    177.10      556.37    0.64**

AVG GM/KWH =

1.97    2.38    7.58    6.64    237.50      746.10    0.86

WT AVG NO2/NOX RATIO =

0.120860396

W.F.	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.15
MODE =	1	2	3	4	5	6	7	8
PARTIC. WT, MG =	1.04	0.71	0.71	0.58	1.72	0.72	0.60	0.11
Exh.Probe Mass Flow Rate (g/Sec.)=	1.09	0.87	0.6820	0.44	0.86	0.67	0.57	0.14
Sample Time (Sec.)=	180	180	180	120	120	120	180	180
Dil.Exh.Sample Mass-Part.Filter (kg) =	0.20	0.16	0.12	0.05	0.10	0.08	0.07	0.02
Dil.Tunnel Tot Flow Temp.Corr. (scmm)=	65.07	65.23	65.35	65.34	65.23	65.21	65.25	65.35
Dil.Tunnel Tot Mass Flow (kg/Hr.)=	5048.02	5060.38	5069.91	5069.41	5060.63	5058.61	5062.34	5069.51
Partic.Mass Flow Rate (g/Hr.)=	26.83	23.00	29.32	55.56	84.44	45.23	44.64	22.61
GM/BHPH=	0.109	0.117	0.222	2.138	0.402	0.294	0.438	
WTD AVG GM/H =	38.252							
WEIGHTED AVG GM/BHPH =	0.282							
WTD AVG GM/KWH =	0.378							

**OLSON-ECOLOGIC ENGINE TESTING LABORATORY  
GTAT ISO-8178 8-MODE TESTING OF VISCON POLYMER  
8MLP53 (MULTI-FILTER)**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	t/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Humidy %	Oil P paig	nH/nC RATIO
1	2200.0	598.1	250.6	5.71	206.90	664.74	538.36	405.81	734	479.4	922.7	64.5	80.4	30.20	0.027	647.00	100.4	524.00	246.5	159.3	42.2	53.4	1.8
2	2200.0	469.9	196.8	5.38	113.12	611.36	512.42	469.03	578.5	408.9	864.6	61.9	81.9	30.20	0.025	591.17	103.0	495.49	226.3	242.7	38.6	51.1	1.8
3	2200.1	314.6	131.8	4.82	119.44	478.56	400.73	572.66	407	331.7	759.7	61.8	81.7	30.20	0.023	462.66	103.3	387.42	224.4	348.2	38.7	51.7	1.8
4	2200.0	62.0	26.0	2.92	227.00	177.40	131.79	831.10	166	253.2	516.3	59.3	81.0	30.20	0.014	170.43	103.4	126.62	337.9	493.2	38.1	53.2	1.8
5	1400.0	788.5	210.2	7.41	910.86	761.73	637.99	227.06	568	266.3	1050.6	59.8	79.5	30.20	0.035	732.69	101.1	613.67	177.4	49.7	40.3	37.3	1.8
6	1400.0	578.0	154.1	7.15	661.36	761.36	648.33	328.07	412	213.4	974.7	59.2	79.0	30.20	0.034	731.36	101.9	622.78	303.9	24.2	40.6	39.5	1.8
7	1400.1	382.1	101.9	6.36	233.08	661.16	579.43	472.10	276.5	173.3	811.2	59.5	78.6	30.20	0.030	635.53	101.3	556.98	320.9	151.2	41.3	41.6	1.8
8	569.3	1.2	0.1	3.17	304.73	368.19	262.60	1100.47	15.25	51.4	387.0	61.1	78.5	30.20	0.016	355.31	96.3	253.42	923.6	176.9	42.5	18.0	1.8

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	312.19	319.95	1643.69	1323.6835	44040	1698405	138712.72	123.1	0.19
15.00	2	301.59	146.20	1255.28	1050.0132	34710	1414955	109317.13	156.7	0.16
15.00	3	288.50	120.95	769.71	646.71566	24420	1103004	76639.92	176.3	0.16
10.00	4	275.85	151.45	186.80	141.8949	9960	714145	30598.53	165.5	0.26
10.00	5	103.69	836.13	1104.95	921.27801	34080	1023386	106852.51	22.7	0.16
10.00	6	112.75	456.89	830.04	705.99786	24720	767834	77622.18	8.4	0.15
10.00	7	122.83	121.89	546.02	479.98817	16590	577230	52234.85	39.5	0.12
15.00	8	30.68	17.08	32.71	24.121784	915	60455	2789.18	5.1	0.29

<b>WTD AVG BHP =</b>	<b>136.11</b>	<b>KW =</b>	<b>101.50</b>	W.F.	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10	0.15		
				MODE =	1	2	3	4	5	6	7	8			
				<b>PARTIC. WT, MG =</b>	1.08	0.83	0.62	0.56	1.72	0.76	0.35	0.15			
				Exh.Probe Mass Flow Rate (g/Sec.)=	1.09	0.87	0.68	0.44	0.86	0.67	0.57	0.14			
				Sample Time (Sec.)=	180	180	180	120	120	120	180				
				Dil.Exh.Sample Mass-Part.Filter (kg)=	0.20	0.16	0.12	0.05	0.10	0.08	0.07	0.02			
				Dil.Tunnel Tot Flow Temp. Corr. (scrm)=	65.04	65.29	65.38	65.35	65.20	65.25	65.22	65.32			
				Dil.Tunnel Tot Mass Flow (kg/Hr.)=	5045.83	5064.86	5072.26	5070.10	5058.18	5061.98	5059.60	5067.37			
				Partic.Mass Flow Rate (g/Hr.)=	27.85	26.91	25.62	53.65	84.40	47.78	26.03	30.82			
<b>AVG GM/BHPH =</b>	<b>1.48</b>	<b>1.82</b>	<b>6.04</b>	<b>5.01</b>	<b>177.42</b>	<b>557.28</b>	<b>0.68</b>	<b>GM/BHPH=</b>	0.111	0.137	0.194	2.067	0.402	0.310	0.255
<b>AVG GM/KWH =</b>	1.98	2.44	8.10	6.72	237.92	747.32	0.91	<b>WTD AVG GM/H =</b>	37.866						
<b>WT AVG NO2/NOX RATIO =</b>	0.189363726							<b>WEIGHTED AVG GM/BHPH =</b>	0.278						
								<b>WTD AVG GM/KWH =</b>	0.373						



## **1.0 Scope and Application**

- 1.1 This exhaust gas sampling protocol is specifically designed for sampling dilute exhaust generated from heavy-duty diesel engines being operated over the EPA transient cycle or steady state emission test as described in 40 CFR Part 86.
- 1.2 For transient cycle operation, this official testing protocol involves continual sample integration of all gaseous emissions along with pertinent engine and ambient variables for 1200 seconds (20 minutes). For eight-mode steady state operation, this official testing protocol involves modal sample collection for a total of 20 minutes. Modes 1, 2, 3 and 8 are collected for 3 minutes of the 5 minute mode, while modes 4, 5, 6 and 7 are collected for 2 minutes.
- 1.3 The constant volume sampling system (CVS) continually dilutes the exhaust gas with finely filtered room air to maintain a constant volume of exhaust gas plus dilute air over the test interval. Coincidentally a second bag is being filled continuously with the same air used to dilute the exhaust gas. Therefore at the end of each test there are bag samples of the proportional and integrated dilute exhaust and the filtered air that was used to dilute the exhaust.

## **2.0 Method Summary**

- 2.1 Samples analyzed for Volatile Organic Compounds (VOC's) are collected from the integrated bag samples that have been continuously filled during the test cycle. One bag is filled with dilution air, while the other is filled with dilute exhaust. A Teflon diaphragm pump is T-connected through a valve to the constant volume sampling system to fill a Tedlar bag with either dilution air or dilute exhaust. For every emission sample and tunnel blank sampled, a dilution air sample must be co-sampled for background correction. Tedlar bag samples are analyzed by gas chromatography (GC).

## **3.0 Health and Safety**

- 3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.
- 3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

## **4.0 Sample Preservation, Containers, Handling, and Storage**

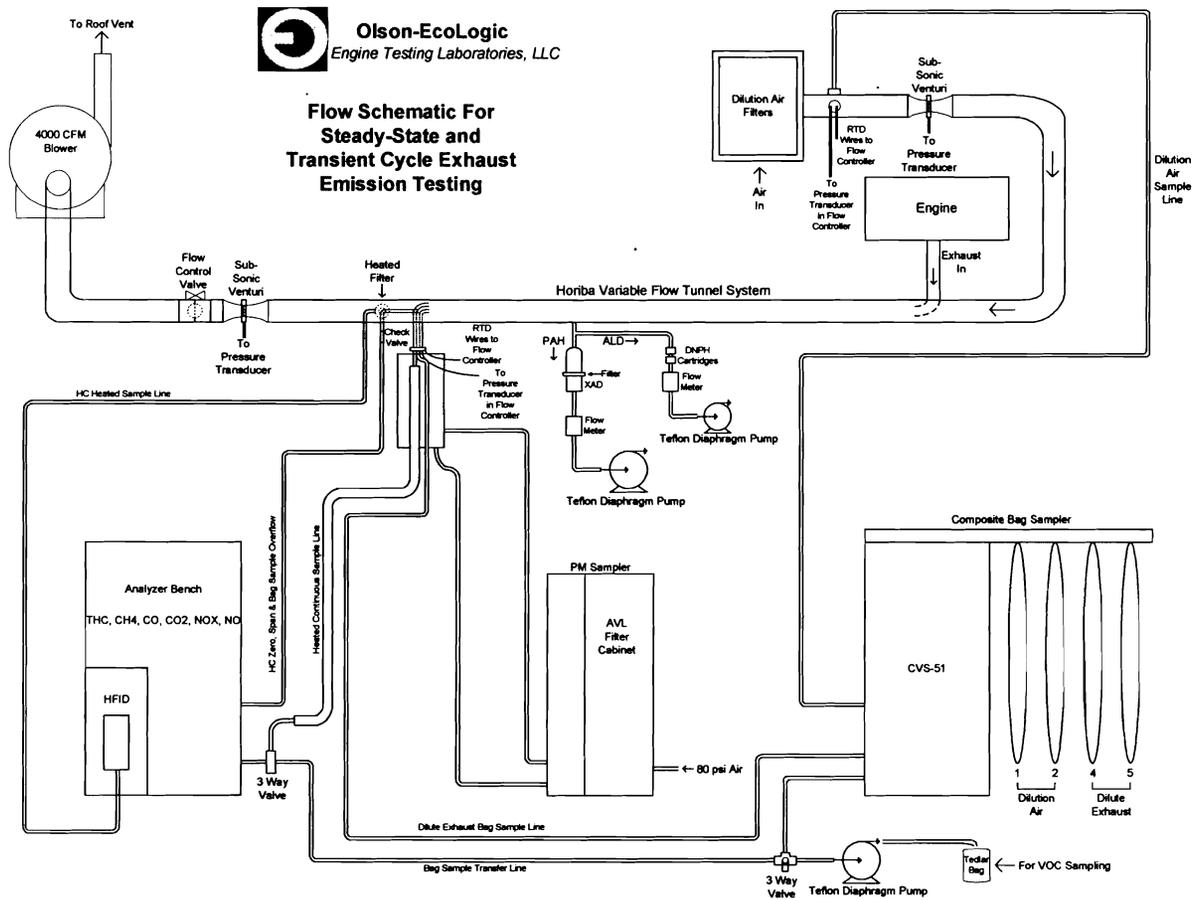
- 4.1 Samples are collected and stored in 1 L Tedlar bags.
- 4.2 Tedlar bags may not be exposed to heat or excessive light. Black Tedlar bags may be used to eliminate photochemically induced reactions.
- 4.3 Samples must be analyzed within 24 hours of collection.
- 4.4 To prevent sample contamination, Tedlar bags are used only once.

**5.0 Interferences and Potential Problems**

- 5.1 To maximize sample integrity, Tedlar bags should not leak or be exposed to excessive light or heat. Tedlar bags must be shielded from direct sunlight to avoid photochemically induced reactions of any reactive hydrocarbons.
  - 5.1.1 The compound 1,3-butadiene, resulting mostly during cold-start testing by the combustion of olefins, is extremely unstable as it easily polymerizes in the presence of oxygen. Therefore all samples analyzed for 1,3-butadiene must be run within 1 hour of collection.

**6.0 Equipment/Apparatus**

- 6.1 Tedlar bags: SKC, Inc., 1 L in capacity, or equivalent
- 6.2 Super Syringe: Fisher Scientific, 1 L
- 6.3 Teflon diaphragm vacuum-pressure pump: Barnant Co., or equivalent
- 6.4 Sampling Train Schematic



## 7.0 Procedure

### 7.1 Sample collection

- 7.1.1 After the analysis of the bag samples collected from the emission test, a T-connected valve is turned enabling the sample to flow through the Teflon diaphragm pump to the Tedlar bag.
- 7.1.2 A dilution air sample is collected first by setting the CVS to “read bag 1 or 2”. Before connecting the Tedlar bag to the pump, the pump should be run for about 30 seconds to flush any contaminants from previous samples. The Tedlar bag is then attached to the pump and powered on to collect the sample.
- 7.1.3 A tunnel blank or dilute exhaust sample is collected next by setting the CVS to “read bag 4 or 5”. Before connecting the Tedlar bag to the pump, the pump should be run for about 30 seconds to flush any contaminants from previous samples. The Tedlar bag is then attached to the pump and powered on to collect the sample.
- 7.1.4 The dilution and work is noted for each dilute exhaust sample taken for final calculations.
- 7.1.5 The Tedlar bag samples are quickly taken into the laboratory and shaded from direct light for analysis by GC.

## 8.0 Quality Control/Quality Assurance

### 8.1 Dilution Air Sample

- 8.1.1 A dilution air sample is collected for every emission sample and tunnel blank to correct for background levels in the ambient dilution air.

### 8.2 Tunnel Blank Sample

- 8.2.1 A tunnel blank sample is collected each analysis day to note any hydrocarbon contamination that may be in the tunnel.

### 8.3 Duplicate Sample

- 8.3.1 A duplicate emission sample is collected each analysis day for quality purposes.

### 8.4 Data Comparisons

- 8.4.1 The VOC sampling protocol provides the same gas sample used in the official calculation of results from the emission test. The hydrocarbon (HC) data can be compared to the GC data.
- 8.4.2 The primary bag gas data are printed out on every test report automatically along with other engine operating data. The dilution ratio and work for the entire test is also printed out on each report from the precise CVS flow data. Accordingly, there is generally no need to dilute the Tedlar bag samples before GC analysis.

## 8.5 Leak Checks

### 8.5.1 Sampling Train

8.5.1.1 To leak check any part of the sampling train, the suspected leak area should be isolated and pressurized or put under vacuum. A gauge may then be used to check if a leak exists.

### 8.5.2 Tedlar Bags

8.5.2.1 Tedlar bags may be leak checked by filling the bag and leaving it under pressure overnight. If the bag deflates, it should not be used for sample collection.

## 8.6 Flow Rates

8.6.1 The flow rate into the CVS bags is controlled by the Horiba CVS with needle valves. The flow rate over the 20 minute transient test cycle is 5 L/min, while the flow rate over the 40 minute steady state test cycle is 2.5 L/min.



## 1.0 Scope and Application

- 1.1 This SOP is based on CARB Method 1002 and describes the use of gas chromatography (GC) coupled with flame ionization detection (FID) for the determination of C<sub>2</sub>-C<sub>5</sub> hydrocarbons (light-end hydrocarbons) in the ppbC range from automotive source samples. The compounds listed below may be determined by this method:

Compound	CAS Registry No.
ethene	00074-85-1
ethyne	00074-86-2
ethane	00074-84-0
propene	00115-07-1
propane	00074-98-6
1,2-propadiene	00463-49-0
1-propyne	00074-99-7
2-methylpropane	00075-28-5
2-methylpropene (isobutene)	00115-11-7
1-butene	00106-98-9
1,3-butadiene	00106-99-0
n-butane	00106-97-8
trans-2-butene	00624-64-6
2,2-dimethylpropane	00463-82-1
1-butyne	00107-00-6
cis-2-butene	00590-18-1
3-methyl-1-butene	00563-45-1
2-methylbutane	00078-78-4
2-butyne	00503-17-3
1-pentene	00109-67-1
2-methyl-1-butene	00563-46-2
n-pentane	00109-66-0
2-methyl-1,3-butadiene	00078-79-5
trans-2-pentene	00646-04-8
3,3-dimethyl-1-butene	00558-37-2
cis-2-pentene	00627-20-3
1-buten-3-yne	00689-97-4
2-methyl-2-butene	00513-35-9
1,3-cyclopentadiene	00542-92-7
2,2-dimethylbutane	00075-83-2
cyclopentene	00142-29-0
4-methyl-1-pentene	00691-37-2
3-methyl-1-pentene	00760-20-3
cyclopentane	00287-92-3
2,3-dimethylbutane	00079-29-8
1-methyl-tert-butyl-ether	01634-04-4
4-methyl-cis-2-pentene	00691-38-3
2-methylpentane	00107-83-5
4-methyl-trans-2-pentene	00674-76-0
3-methylpentane	00096-14-0

2-methyl-1-pentene	00763-29-1
1-hexene	00592-41-6
n-hexane	00110-54-3

- 
- 
- 1.2 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs. Analysts should also be skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 Method Summary

- 2.1 This method provides GC/FID conditions for the detection of the target analytes. Exhaust samples are introduced to the GC from Tedlar bags by means of gas sampling valves. Separation of the sample hydrocarbons takes place in a 50 m 0.32 mm ID PLOT fused silica column. Quantitative analysis is performed by the FID using an external standard approach. The computerized GC data acquisition system identifies the hydrocarbons and concentrations are determined by peak area response factors.
- 2.2 Prior to the use of this method, appropriate sample collection techniques must be used.
- 2.2.1 Samples are collected from the exhaust in Tedlar bags. Dilutions may apply and must be accounted for in final calculations.

## 3.0 Health and Safety

- 3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.
- 3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

## 4.0 Sample Preservation, Containers, Handling, and Storage

- 4.1 Samples are collected and stored in 1 L Tedlar bags.
- 4.2 Tedlar bags may not be exposed to heat or excessive light. Black Tedlar bags may be used to eliminate photochemically induced reactions.
- 4.3 Samples must be analyzed within 24 hours of collection.

## 5.0 Interferences and Potential Problems

- 5.1 To maximize sample integrity, Tedlar bags should not leak or be exposed to excessive light or heat. Tedlar bags must be shielded from direct sunlight to avoid photochemically induced reactions of any reactive hydrocarbons.
- 5.1.1 The compound 1,3-butadiene, resulting mostly during cold-start testing by the combustion of olefins, is extremely unstable as it easily polymerizes in the presence of oxygen. Therefore all samples analyzed for 1,3-butadiene must be run within 1 hour of collection.
- 5.2 Any component present in the sample with a retention time very similar to that of a target hydrocarbon would interfere or coelute. If separation cannot be achieved, confirmation of identification should be done using a different column for separation, or an alternate detector, e.g., mass spectrometer (MS), photoionization detector (PID), etc.

## 6.0 Equipment/Apparatus

- 6.1 GC/FID
- 6.1.1 Gas Chromatograph (GC) – Varian CP-3800 with programmable oven temperatures, 10 mL fixed volume injection loop for automated transfer of gaseous samples from the Tedlar bag to the GC, and analytical column interfaced with a flame ionization detector (FID).
- 6.1.1.1 GC Column – Varian CP 7515 PLOT fused silica, 50 m x 0.32 mm ID, or equivalent.
- 6.1.2 Data System – Dell-PC computer with Varian Star software capable of continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.
- 6.2 Nitrogen, compressed and liquid. Minimum purity of 99.998 %.
- 6.3 Helium, compressed. Minimum purity of 99.995 %.
- 6.4 Hydrogen, compressed. Minimum purity of 99.995 %.
- 6.5 Air, compressed. “Zero” grade (<1 ppmC total hydrocarbon contamination), or better.
- 6.6 Tedlar bags: SKC, Inc., 5 to 10 L in capacity, or equivalent.
- 6.7 Super Syringe: Fisher Scientific, 1 L.

## 7.0 Reagents

- 7.1 NIST-certified SRM or secondary NIST-traceable standards shall be used in all tests. A secondary standard is obtained by a comparison between a SRM and a candidate standard.
- 7.2 Calibration Standard
- 7.2.1 The quantitative calibration standard for all target hydrocarbons is propane. Lehner/Martin, Inc. Propane Std. in zero air – 2955 ppbC, or equivalent

### 7.3 Control Standard

7.3.1 Quality control standard, containing at least ethane, propane, n-butane, 2-methylpropene and 1,3-butadiene at concentrations between 200 and 3000 ppbC based on a propane standard. This standard is used as a daily update of control charts and a daily determination of marker retention time windows.

Scott-Marrin, Inc. 23 Component custom blend in nitrogen, or equivalent

7.4 A high concentration standard (higher than the calibration standard), containing the target hydrocarbons listed in Section 7.3.1, is used for linearity determinations. The high concentration standard must have concentrations verified against a NIST-traceable propane standard.

7.5 A low concentration standard (5 to 10 times the estimated MDL), containing the target hydrocarbons listed in Section 7.3.1, is used for MDL determinations. The low concentration standard must have concentrations verified against a NIST-traceable propane standard.

7.5.1 In lieu of a low concentration standard, a higher concentration standard may be diluted.

## 8.0 Procedure

### 8.1 Sample collection

8.1.1 Specific sample collection procedures can be found in the VOC Sampling Protocol.

8.1.2 In general, samples are collected from the integrated bag samples that have been continuously filled during the 1200 second transient test cycle. One bag is filled with dilution air, while the other is filled with dilute exhaust. A Teflon diaphragm pump is T-connected through a valve to the constant volume sampling system to fill a Tedlar bag with either dilution air or dilute exhaust.

### 8.2 GC chromatographic conditions:

Injection volume: 10 mL fixed loop  
Injector temperature: -180 C (hold 7.10 min) to 250 C (hold 61.25 min) @ 200 C/min  
Helium carrier flow: 3 mL/min  
Nitrogen aux. flow: 27 mL/min  
Hydrogen flow: 30 mL/min  
Air flow: 300 mL/min  
Column temperature: 0 C (hold 10 min) to 200 C (hold 20 min) @ 5 C/min  
Detector temperature: 250 C

8.3 Samples in Tedlar bags are connected to one of sixteen ports on the autosampler and the analytical process then begins.

8.4 The sample is introduced into the carrier gas stream through the injection valve.

8.5 Each separated analyte exits the column into the FID where a response is generated.

8.6 Hydrocarbon concentrations are calculated in parts per billion of carbon (ppbC) by Varian's Star software from the NIST-traceable propane calibration standard.

- 8.7 Analytes with concentrations higher than demonstrated in the instruments range of linearity must be diluted and rerun.
- 8.8 Peak identification and integration are checked and corrected if necessary by the analyst.
- 8.9 Target compounds that coelute are reported as the major component.
- 8.10 The PLOT fused silica analytical column is heated to 200 C to prevent carry over and assure all compounds are eluted before the next run.

## 9.0 Calculations

- 9.1 The target hydrocarbon concentrations, in ppbC, are calculated by the data system using propane as an external standard.

$$\text{Concentration}_{\text{sample}} \text{ (ppbC)} = \text{Peak Area}_{\text{sample}} \times \text{Response Factor}$$

where the response factor (RF) is calculated during daily calibration by:

$$\text{RF} = \frac{\text{Concentration of propane standard (ppbC)}}{\text{area of propane peak}}$$

## 10.0 Quality Control/Quality Assurance

### 10.1 Instrument Blank Run

10.1.1 An instrument blank of pure nitrogen is run each analysis day. All target hydrocarbon concentrations from the blank analysis must be below the method detection limit (MDL) before the analysis may proceed.

10.1.1.1 If the blank shows a peak greater than the MDL in the region of interest, the source of contamination must be investigated and remedied.

### 10.2 Calibration Run

10.2.1 The calibration standard is analyzed each analysis day to generate the response factor used to quantify the sample concentrations.

### 10.3 Control Standard Run

10.3.1 The quality control standard is analyzed at least once each analysis day. Measurements of all compounds specified in Section 7.3.1 must fall within the control limits to ensure the validity of the sample analyses that day. To meet this requirement, it may be necessary to inspect and repair the GC, and rerun the calibration and/or control standards.

#### 10.4 Control Charts

10.4.1 A quality control chart is maintained for each component of the control standard listed in Section 7.3.1, and is performed for new instruments, after making instrument modifications that can affect recovery, and at least once every year. The control charts, used on a daily basis, establish that the method is “in control.” The following describes how to construct a typical control chart:

1. Obtain at least 20 daily control standard results;
2. Calculate the control standard mean concentration and standard deviation for the target hydrocarbon; and
3. Create a control chart for the target hydrocarbon by placing the concentration on the Y-axis and the date on the X-axis. Establish upper and lower warning limits at either two standard deviations (2s) or 5 percent, whichever is greater, above and below the average concentration.
4. A control standard measurement is considered to be out-of-control when the analyzed value exceeds the control limit or two successive control standard measurements of the same analyte exceed the warning limit.
5. If 20 control standard measurements are not yet available to create a control chart (e.g., the control standard was expended and replaced prior to obtaining 20 points with the new standard), measurements must be within 15% relative standard deviation (RSD) of the certified concentration. If the control standard is not a NIST standard, the cylinder should be certified by the laboratory against a NIST standard.

The measured concentrations of all target hydrocarbons contained in the control standard must be within the control limits (in control) for the sample results to be considered acceptable.

#### 10.5 Duplicates

10.5.1 A duplicate analysis of one sample is performed at least once per analysis day. The relative percent difference (RPD) is calculated for each duplicate run:

$$\text{RPD (\%)} = \frac{\text{Difference between duplicate and original measurement}}{\text{Average of duplicate and original measurement}} \times 100$$

For each compound specified in Section 7.3.1, the allowable RPD depends on the average concentration level for the duplicate runs, as shown in the following table:

<u>Average Measurement for the Duplicate Runs</u>		<u>Allowable RPD (%)</u>
1 to 10	times MDL	100
10 to 20	“ “	30
20 to 50	“ “	20
Greater than 50	“ “	15

If the results of the duplicate analyses do not meet these criteria for all compounds specified in Section 7.3.1, the sample may be reanalyzed. If reanalysis is not feasible or if the criteria are still not met on reanalysis, all sample results for that analysis day are invalid.

#### 10.6 Linearity

10.6.1 A multipoint calibration to confirm instrument linearity is performed for the target hydrocarbons in the control standard for new instruments, after making instrument modifications that can affect linearity, and at least once every year. The multipoint calibration consists of at least five concentration or mass loading levels (using smaller or larger volume sample sizes of existing standards is acceptable), each above the MDL, distributed over the range of expected sample concentration. A linear regression analysis is performed using concentration and average area counts to determine the regression correlation coefficient (r). The r must be greater than 0.995 to be considered sufficiently linear for one-point calibrations.

## 10.7 MDL

10.7.1 The MDL for the target hydrocarbons in the control standard must be determined for new instruments, after making instrument modifications that can affect linearity and/or sensitivity, and at least once every year. To make the calculations, it is necessary to run at least seven replicate determinations at a concentration of 5 to 10 times the estimated MDL. The MDL is calculated using the following equation:

$$\text{MDL} = t \times s$$

where s is the standard deviation of the replicates and t is the t-factor for 99 percent confidence for a one-sided normal (Gaussian) distribution. The number of degrees of freedom is equal to the number of replicates, minus one. An abbreviated t-table is:

<u>Degrees of Freedom</u>	<u>t-value</u>
4	3.7
5	3.4
6	3.1
7	3.0

10.7.1.1 The maximum allowable MDL for each compound is 1 ppbC. The calculated laboratory MDL must be equal to or lower than the maximum allowable MDL. All peaks identified as target compounds that are equal to or exceed the maximum allowable MDL must be reported. If the calculated laboratory MDL is less than the maximum allowable MDL, the laboratory may choose to set its reporting limit at the maximum allowable MDL, the calculated laboratory MDL, or any level in between.

10.7.1.2 For the purpose of calculating the total mass of all species, the concentrations of all compounds below the MDL are considered to be zero.

## 10.8 Method 1002/1003 Crossover Check

10.8.1 A crossover check is analyzed at least once each analysis day, and is performed by choosing a compound from a sample to be measured and compared by both Method 1002 and 1003. The crossover compound shall be a compound that can reasonably be expected to be found and measured by both methods in the laboratory performing the analysis. The maximum relative percent difference (RPD) allowed from the results obtained by the two methods is 15%.

## **11.0 References**

- 11.1 California Environmental Protection Agency, Air Resources Board, Method 1002, Revision IV, July 2002



## 1.0 Scope and Application

- 1.1 This SOP is based on CARB Method 1003 and describes the use of gas chromatography (GC) coupled with flame ionization detection (FID) for the determination of C<sub>6</sub>-C<sub>12</sub> hydrocarbons (mid-range hydrocarbons) in the ppbC range from automotive source samples. The compounds listed below may be determined by this method:

Compound	CAS Registry No.
n-hexane	00110-54-3
trans-3-hexene	13269-52-8
cis-3-hexene	07642-09-3
trans-2-hexene	04050-45-7
3-methyl-trans-2-pentene	00616-12-6
2-methyl-2-pentene	00625-27-4
3-methylcyclopentene	01120-62-3
cis-2-hexene	07688-21-3
1-ethyl-tert-butyl-ether	00637-92-3
3-methyl-cis-2-pentene	00922-62-3
2,2-dimethylpentane	00590-35-2
methylcyclopentane	00096-37-7
2,4-dimethylpentane	00108-08-7
2,2,3-trimethylbutane	00464-06-2
3,4-dimethyl-1-pentene	07385-78-6
1-methylcyclopentene	00693-89-0
benzene	00071-43-2
3-methyl-1-hexene	03404-61-3
3,3-dimethylpentane	00562-49-2
cyclohexane	00110-82-7
2-methylhexane	00591-76-4
2,3-dimethylpentane	00565-59-3
cyclohexene	00110-83-8
3-methylhexane	00589-34-4
trans-1,3-dimethylcyclopentane	01759-58-6
cis-1,3-dimethylcyclopentane	02532-58-3
3-ethylpentane	00617-78-7
trans-1,2-dimethylcyclopentane	00822-50-4
1-heptene	00592-76-7
2,2,4-trimethylpentane	00540-84-1
trans-3-heptene	14686-14-7
n-heptane	00142-82-5
2-methyl-2-hexene	02738-19-4
3-methyl-trans-3-hexene	03899-36-3
trans-2-heptene	14686-13-6
3-ethyl-2-pentene	00816-79-5
2,4,4-trimethyl-1-pentene	00107-39-1
2,3-dimethyl-2-pentene	10574-37-5
cis-2-heptene	06443-92-1
methylcyclohexane	00108-87-2

2,2-dimethylhexane	00590-73-8
2,4,4-trimethyl-2-pentene	00107-40-4
ethylcyclopentane	01640-89-7
2,5-dimethylhexane	00592-13-2
2,4-dimethylhexane	00589-43-5
1,2,4-trimethylcyclopentane	02815-58-9
3,3-dimethylhexane	00563-16-6
2,3,4-trimethylpentane	00565-75-3
2,3,3-trimethylpentane	00560-21-4
toluene	00108-88-3
2,3-dimethylhexane	00584-94-1
2-methylheptane	00592-27-8
4-methylheptane	00589-53-7
3-methylheptane	00589-81-1
(1a,2a,3b)-1,2,3-trimethylcyclopentane	15890-40-1
cis-1,3-dimethylcyclohexane	00638-04-0
trans-1,4-dimethylcyclohexane	02207-04-7
2,2,5-trimethylhexane	03522-94-9
trans-1-methyl-3-ethylcyclopentane	02613-65-2
cis-1-methyl-3-ethylcyclopentane	16747-50-5
1-octene	00111-66-0
trans-4-octene	14850-23-8
n-octane	00111-65-9
trans-2-octene	13389-42-9
trans-1,3-dimethylcyclohexane	02207-03-6
cis-2-octene	07642-04-8
2,3,5-trimethylhexane	01069-53-0
2,4-dimethylheptane	02213-23-2
cis-1,2-dimethylcyclohexane	02207-01-4
2,6-dimethylheptane	01072-05-5
ethylcyclohexane	01678-91-7
3,5-dimethylheptane	00926-82-9
ethylbenzene	00100-41-4
2,3-dimethylheptane	03074-71-3
m-&p-xylene	00108-38-3
4-methyloctane	02216-34-4
2-methyloctane	03221-61-2
3-methyloctane	02216-33-3
styrene (ethenylbenzene)	00100-42-5
o-xylene	00095-47-6
1-nonene	00124-11-8
n-nonane	00111-84-2
(1-methylethyl)benzene	00098-82-8
2,2-dimethyloctane	15869-87-1
2,4-dimethyloctane	04032-94-4
2,6-dimethyloctane	02051-30-1
n-propylbenzene	00103-65-1
1-methyl-3-ethylbenzene	00620-14-4
1-methyl-4-ethylbenzene	00622-96-8

1,3,5-trimethylbenzene	00108-67-8
1-methyl-2-ethylbenzene	00611-14-3
1,2,4-trimethylbenzene	00095-63-6
n-decane	00124-18-5
(2-methylpropyl)benzene	00538-93-2
(1-methylpropyl)benzene	00135-98-8
1-methyl-3-(1-methylethyl)benzene	00535-77-3
1,2,3-trimethylbenzene	00526-73-8
1-methyl-4-(1-methylethyl)benzene	00099-87-6
2,3-dihydroindene (indan)	00496-11-7
1-methyl-2-(1-methylethyl)benzene	00527-84-4
1,3-diethylbenzene	00141-93-5
1,4-diethylbenzene	00105-05-5
1-methyl-3-n-propylbenzene	01074-43-7
1-methyl-4-n-propylbenzene	01074-55-1
1,2-diethylbenzene	00135-01-3
1-methyl-2-n-propylbenzene	01074-17-5
1,4-dimethyl-2-ethylbenzene	01758-88-9
1,3-dimethyl-4-ethylbenzene	00874-41-9
1,2-dimethyl-4-ethylbenzene	00934-80-5
1,3-dimethyl-2-ethylbenzene	02870-04-4
n-undecane (hendecane)	01120-21-4
1,2-dimethyl-3-ethylbenzene	00933-98-2
1,2,4,5-tetramethylbenzene	00095-93-2
1-methyl-2-n-butylbenzene	01595-11-5
1,2,3,5-tetramethylbenzene	00527-53-7
1-(1,1-dimethylethyl)-2-methylbenzene	01074-92-6
1,2,3,4-tetramethylbenzene	00488-23-3
n-pentylbenzene	00538-68-1
1-(1,1-dimethylethyl)-3,5-dimethylbenzene	00098-19-1
naphthalene	00091-20-3
n-dodecane	00112-40-3
n-tridecane	00629-50-5

- 1.2 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs. Analysts should also be skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 Method Summary

- 2.1 This method provides GC/FID conditions for the detection of the target analytes. Exhaust samples are introduced to the GC from Tedlar bags by means of gas sampling valves. Separation of the sample hydrocarbons takes place in a 60 m 0.32 mm ID WCOT fused silica column. Quantitative analysis is

performed by the FID using an external standard approach. The computerized GC data acquisition system identifies the hydrocarbons and concentrations are determined by peak area response factors.

2.2 Prior to the use of this method, appropriate sample collection techniques must be used.

2.2.1 Samples are collected from the exhaust in Tedlar bags. Dilutions may apply and must be accounted for in final calculations.

### 3.0 Health and Safety

3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

### 4.0 Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected and stored in 1 L Tedlar bags.

4.2 Tedlar bags may not be exposed to heat or excessive light. Black Tedlar bags may be used to eliminate photochemically induced reactions.

4.3 Samples must be analyzed within 24 hours of collection.

### 5.0 Interferences and Potential Problems

5.1 To maximize sample integrity, Tedlar bags should not leak or be exposed to excessive light or heat. Tedlar bags must be shielded from direct sunlight to avoid photochemically induced reactions of any reactive hydrocarbons.

5.2 Any component present in the sample with a retention time very similar to that of a target hydrocarbon would interfere or coelute. If separation cannot be achieved, confirmation of identification should be done using a different column for separation, or an alternate detector, e.g., mass spectrometer (MS), photoionization detector (PID), etc.

### 6.0 Equipment/Apparatus

6.1 GC/FID

6.1.1 Gas Chromatograph (GC) – Varian CP-3800 with programmable oven temperatures, 30 mL fixed volume injection loop for automated transfer of gaseous samples from the Tedlar bag to the GC, and analytical column interfaced with a flame ionization detector (FID).

6.1.1.1 GC Column – Varian CP 8870 WCOT fused silica, 60 m x 0.32 mm ID, or equivalent.

- 6.1.2 Data System – Dell-PC computer with Varian Star software capable of continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.
- 6.2 Nitrogen, compressed and liquid. Minimum purity of 99.998 %.
- 6.3 Helium, compressed. Minimum purity of 99.995 %.
- 6.4 Hydrogen, compressed. Minimum purity of 99.995 %.
- 6.5 Air, compressed. “Zero” grade (<1 ppmC total hydrocarbon contamination), or better.
- 6.6 Tedlar bags: SKC, Inc., 5 to 10 L in capacity, or equivalent.
- 6.7 Super Syringe: Fisher Scientific, 1 L

## 7.0 Reagents

- 7.1 NIST-certified SRM or secondary NIST-traceable standards shall be used in all tests. A secondary standard is obtained by a comparison between a SRM and a candidate standard.
- 7.2 Calibration Standard
  - 7.2.1 The quantitative calibration standard for all target hydrocarbons is propane.  
Lehner/Martin, Inc. Propane Std. in zero air – 2955 ppbC, or equivalent
- 7.3 Control Standard
  - 7.3.1 Quality control standard, containing at least n-hexane, benzene, toluene, n-octane, ethylbenzene, m&p-xylene, o-xylene and n-decane at concentrations between 200 and 2000 ppbC based on a propane standard. This standard is used as a daily update of control charts and a daily determination of marker retention time windows.  
Scott-Marrin, Inc. 23 Component custom blend in nitrogen, or equivalent
- 7.4 A high concentration standard (higher than the calibration standard), containing the target hydrocarbons listed in Section 7.3.1, is used for linearity determinations. The high concentration standard must have concentrations verified against a NIST-traceable propane standard.
- 7.5 A low concentration standard (5 to 10 times the estimated MDL), containing the target hydrocarbons listed in Section 7.3.1, is used for MDL determinations. The low concentration standard must have concentrations verified against a NIST-traceable propane standard.
  - 7.5.1 In lieu of a low concentration standard, a higher concentration standard may be diluted.

## 8.0 Procedure

## 8.1 Sample collection

8.1.1 Specific sample collection procedures can be found in the VOC Sampling Protocol.

8.1.2 In general, samples are collected from the integrated bag samples that have been continuously filled during the 1200 second transient test cycle. One bag is filled with dilution air, while the other is filled with dilute exhaust. A Teflon diaphragm pump is T-connected through a valve to the constant volume sampling system to fill a Tedlar bag with either dilution air or dilute exhaust.

## 8.2 GC chromatographic conditions:

Injection volume: 30 mL fixed loop  
Injector temperature: -180 C (hold 7.10 min) to 250 C (hold 61.25 min) @ 200 C/min  
Helium carrier flow: 3 mL/min  
Nitrogen aux. flow: 27 mL/min  
Hydrogen flow: 30 mL/min  
Air flow: 300 mL/min  
Column temperature: 10 C (hold 9 min) to 250 C (hold 13 min) @ 5 C/min  
Detector temperature: 300 C

8.3 Samples in Tedlar bags are connected to one of sixteen ports on the autosampler and the analytical process then begins.

8.4 The sample is introduced into the carrier gas stream through the injection valve.

8.5 Each separated analyte exits the column into the FID where a response is generated.

8.6 Hydrocarbon concentrations are calculated in parts per billion of carbon (ppbC) by Varian's Star software from the NIST-traceable propane calibration standard.

8.7 Analytes with concentrations higher than demonstrated in the instruments range of linearity must be diluted and rerun.

8.8 Peak identification and integration are checked and corrected if necessary by the analyst using the following procedure and criteria:

1. The primary peak identification is done by the computer using the relative retention times based on reference calibration runs.
2. Confirm that the relative peak heights of the sample run ("fingerprint") match the typical fingerprint seen in past sample runs.
3. Compare the relative retention times of the sample peaks with those of reference runs.
4. Any peak with a reasonable doubt is labeled 'Unidentified'.

8.9 Target compounds that coelute, with the exception of m&p-xylene, are reported as the major component. Due to the difficulty in separation of m-xylene and p-xylene, they are reported together as m&p-xylene.

8.10 The WCOT fused silica analytical column is heated to 250 C to prevent carry over and assure all compounds are eluted before the next run.

8.10.1 After running a particularly "dirty" sample, the analyst should run a blank before proceeding to the next sample as there may be sample carry over, or flush the sampling system with air.

## 9.0 Calculations

- 9.1 The target hydrocarbon concentrations, in ppbC, are calculated by the data system using propane as an external standard.

$$\text{Concentration}_{\text{sample}} \text{ (ppbC)} = \text{Peak Area}_{\text{sample}} \times \text{Response Factor}$$

where the response factor (RF) is calculated during daily calibration by:

$$\text{RF} = \frac{\text{Concentration of propane standard (ppbC)}}{\text{area of propane peak}}$$

## 10.0 Quality Control/Quality Assurance

### 10.1 Instrument Blank Run

- 10.1.1 An instrument blank of pure nitrogen is run each analysis day. All target hydrocarbon concentrations from the blank analysis must be below the method detection limit (MDL) before the analysis may proceed.

10.1.1.1 If the blank shows a peak greater than the MDL in the region of interest, the source of contamination must be investigated and remedied.

### 10.2 Calibration Run

- 10.2.1 The calibration standard is analyzed each analysis day to generate the response factor used to quantify the sample concentrations.

### 10.3 Control Standard Run

- 10.3.1 The quality control standard is analyzed at least once each analysis day. Measurements of all compounds specified in Section 7.3.1 must fall within the control limits to ensure the validity of the sample analyses that day. To meet this requirement, it may be necessary to inspect and repair the GC, and rerun the calibration and/or control standards.

### 10.4 Control Charts

- 10.4.1 A quality control chart is maintained for each component of the control standard listed in Section 7.3.1, and is performed for new instruments, after making instrument modifications that can affect recovery, and at least once every year. The control charts, used on a daily basis, establish that the method is "in control." The following describes how to construct a typical control chart:

1. Obtain at least 20 daily control standard results;
2. Calculate the control standard mean concentration and standard deviation for the target hydrocarbon; and

3. Create a control chart for the target hydrocarbon by placing the concentration on the Y-axis and the date on the X-axis. Establish upper and lower warning limits at either two standard deviations (2s) or 5 percent, whichever is greater, above and below the average concentration.
4. A control standard measurement is considered to be out-of-control when the analyzed value exceeds the control limit or two successive control standard measurements of the same analyte exceed the warning limit.
5. If 20 control standard measurements are not yet available to create a control chart (e.g., the control standard was expended and replaced prior to obtaining 20 points with the new standard), measurements must be within 15% relative standard deviation (RSD) of the certified concentration. If the control standard is not a NIST standard, the cylinder should be certified by the laboratory against a NIST standard.

The measured concentrations of all target hydrocarbons contained in the control standard must be within the control limits (in control) for the sample results to be considered acceptable.

## 10.5 Duplicates

- 10.5.1 A duplicate analysis of one sample is performed at least once per analysis day. The relative percent difference (RPD) is calculated for each duplicate run:

$$\text{RPD (\%)} = \frac{\text{Difference between duplicate and original measurement}}{\text{Average of duplicate and original measurement}} \times 100$$

For each compound specified in Section 7.3.1, the allowable RPD depends on the average concentration level for the duplicate runs, as shown in the following table:

<u>Average Measurement for the Duplicate Runs</u>		<u>Allowable RPD (%)</u>
1 to 10	times MDL	100
10 to 20	“ “	30
20 to 50	“ “	20
Greater than 50	“ “	15

If the results of the duplicate analyses do not meet these criteria for all compounds specified in Section 7.3.1, the sample may be reanalyzed. If reanalysis is not feasible or if the criteria are still not met on reanalysis, all sample results for that analysis day are invalid.

## 10.6 Linearity

- 10.6.1 A multipoint calibration to confirm instrument linearity is performed for the target hydrocarbons in the control standard for new instruments, after making instrument modifications that can affect linearity, and at least once every year. The multipoint calibration consists of at least five concentration or mass loading levels (using smaller or larger volume sample sizes of existing standards is acceptable), each above the MDL, distributed over the range of expected sample concentration. A linear regression analysis is performed using concentration and average area counts to determine the regression correlation coefficient (r). The r must be greater than 0.995 to be considered sufficiently linear for one-point calibrations.

## 10.7 MDL

- 10.7.1 The MDL for the target hydrocarbons in the control standard must be determined for new instruments, after making instrument modifications that can affect linearity and/or sensitivity, and at least once every year. To make the calculations, it is necessary to run at least seven replicate determinations at a concentration of 5 to 10 times the estimated MDL. The MDL is calculated using the following equation:

$$\text{MDL} = t \times s$$

where *s* is the standard deviation of the replicates and *t* is the *t*-factor for 99 percent confidence for a one-sided normal (Gaussian) distribution. The number of degrees of freedom is equal to the number of replicates, minus one. An abbreviated *t*-table is:

<u>Degrees of Freedom</u>	<u>t-value</u>
4	3.7
5	3.4
6	3.1
7	3.0

- 10.7.1.1 The maximum allowable MDL for each compound is 1 ppbC. The calculated laboratory MDL must be equal to or lower than the maximum allowable MDL. All peaks identified as target compounds that are equal to or exceed the maximum allowable MDL must be reported. If the calculated laboratory MDL is less than the maximum allowable MDL, the laboratory may choose to set its reporting limit at the maximum allowable MDL, the calculated laboratory MDL, or any level in between.
- 10.7.1.2 For the purpose of calculating the total mass of all species, the concentrations of all compounds below the MDL are considered to be zero.

## 10.8 Method 1002/1003 Crossover Check

- 10.8.1 A crossover check is analyzed at least once each analysis day, and is performed by choosing a compound from a sample to be measured and compared by both Method 1002 and 1003. The crossover compound shall be a compound that can reasonably be expected to be found and measured by both methods in the laboratory performing the analysis. The maximum relative percent difference (RPD) allowed from the results obtained by the two methods is 15%.

## 11.0 References

- 11.1 California Environmental Protection Agency, Air Resources Board, Method 1003, Revision IV, July 2002



## 1.0 Scope and Application

- 1.1 This exhaust gas sampling protocol is specifically designed for sampling dilute exhaust generated from heavy-duty diesel engines being operated over the EPA transient cycle or steady state emission test as described in 40 CFR Part 86.
- 1.2 For transient cycle operation, this official testing protocol involves continual sample integration of all gaseous emissions along with pertinent engine and ambient variables for 1200 seconds (20 minutes). For eight-mode steady state operation, this official testing protocol involves modal sample collection for a total of 20 minutes. Modes 1, 2, 3 and 8 are collected for 3 minutes of the 5 minute mode, while modes 4, 5, 6 and 7 are collected for 2 minutes.
- 1.3 Dilute exhaust samples are collected from the dilution tunnel.

## 2.0 Method Summary

- 2.1 Samples analyzed for particulate and gaseous phase polycyclic aromatic hydrocarbons (PAH) are collected continuously by pulling the dilute exhaust from the dilution tunnel, using a Teflon diaphragm pump, through a particulate filter and XAD cartridge.
- 2.2 Once received by the laboratory, samples are taken through a concentration extraction and analyzed via gas chromatography/mass spectrometry (GC/MS).

## 3.0 Health and Safety

- 3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.
- 3.2 The following method analytes have been classified as known or suspected human or mammalian carcinogens: benzo(a)anthracene and dibenzo(a,h)anthracene. A guideline for the safe handling of carcinogens can be found in Section 5209 of Title 8 of the California Administrative Code.
- 3.3 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

## 4.0 Sample Preservation, Containers, Handling, and Storage

- 4.1 Particulate samples are collected and stored on particulate filters, while gaseous samples are collected and stored on XAD cartridges.
- 4.2 Sealed XAD cartridges must be stored away from light and refrigerated, at a temperature less than 4 C, upon receipt from manufacturer, until ready for use.
- 4.3 From the time of collection to extraction, maintain all samples at 4 C or lower and protect from light. All samples must be extracted within 21 days of collection, and all extracts must be analyzed within 40 days of extraction.

**5.0 Interferences and Potential Problems**

5.1 Transformation of PAH and the formation of artifacts can occur in the sampling train. PAH degradation and transformation on the sampling train filters have been demonstrated. Certain reactive PAH such as benzo(a)pyrene, benzo(a)anthracene, and fluoranthene when trapped on filters can readily react with stack gases. These PAH are transformed by reaction with low levels of nitric acid and higher levels of nitrogen oxides, ozone, and sulfur oxides.

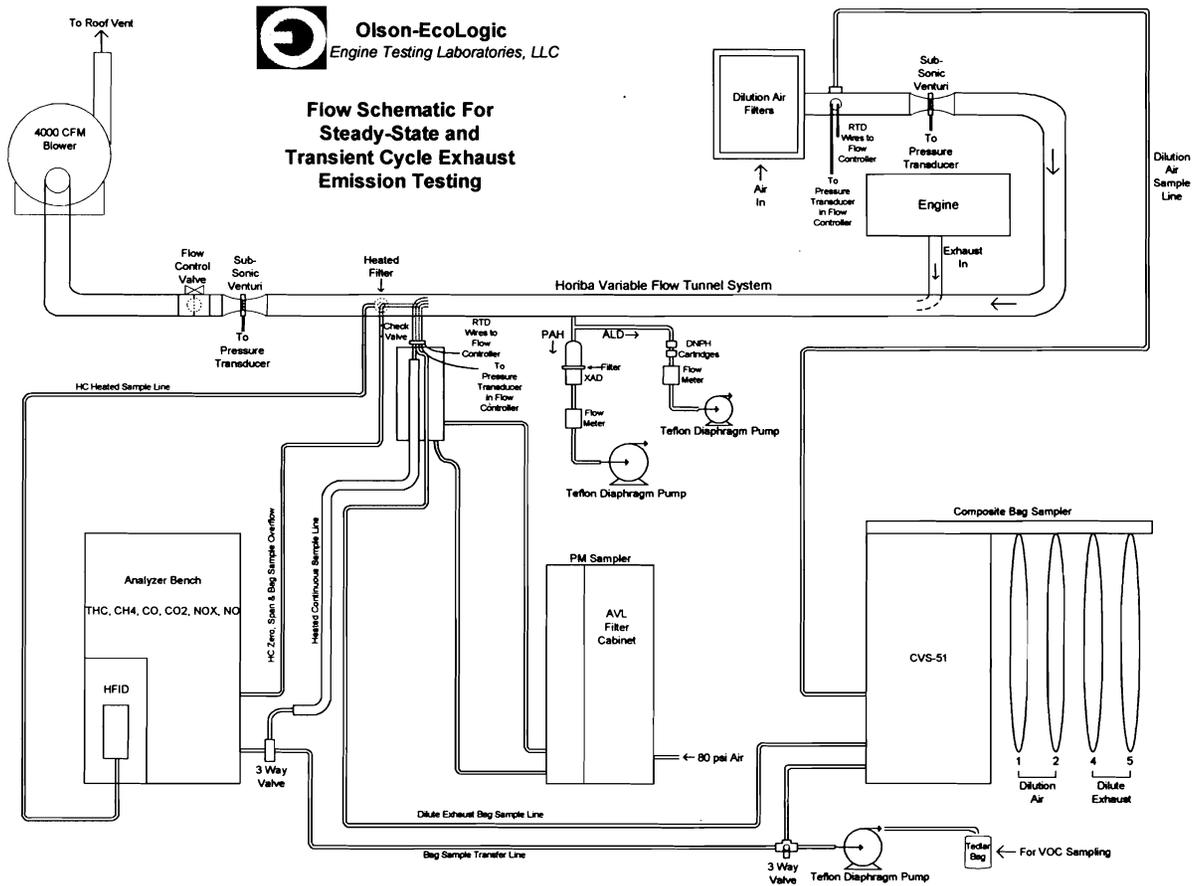
**6.0 Equipment/Apparatus**

6.1 Sampling Train Schematic

6.1.1 8' Gas line from the tunnel to the PAH/ALD sampling system is as follows:

6.1.1.1 Siltek®/Sulfinert® treated ½" 316L grade stainless steel tubing; Restek, or equivalent.

6.1.2 After the "T" connection, all PAH sampling line is ¼" Teflon tubing.



- 6.2 Amberlite XAD cartridges: Acros, or equivalent
- 6.3 PALLFLEX Fiberfilm T60A20 90 mm particulate filter: Pall Life Sciences, or equivalent.
- 6.4 Digital flow meter: TSI, Inc., or equivalent.
- 6.5 Teflon diaphragm vacuum-pressure pump: MFG Corp., or equivalent.

## 7.0 Procedure

### 7.1 Sample collection

- 7.1.1 A particulate filter and XAD cartridge is connected into the sample flow path.
- 7.1.2 Particulate and gaseous phase dilute exhaust samples are collected onto the particulate filter and XAD cartridges by turning on the power to the Teflon diaphragm pump.
  - 7.1.2.1 For transient test cycle sample collection, the Teflon diaphragm pump is turned on for the duration of the 1200 second emission test.
  - 7.1.2.2 For steady state cycle sample collection, the Teflon diaphragm pump is turned on for 3 minutes at the end of modes 1, 2, 3 and 8; and 2 minutes at the end of modes 4, 5, 6 and 7.
- 7.1.3 The flow rate through the particulate filter and XAD cartridge should be 100 L/min.
  - 7.1.3.1 Flow rate should be continuously monitored and adjusted if necessary during sampling.
- 7.1.4 The dilution and work is noted for each dilute exhaust sample taken for final calculations.
- 7.1.5 Particulate filters and XAD cartridges are refrigerated immediately after sample collection and extraction, below a temperature of 4 C, until analyzed by GC/MS.

## 8.0 Calculations

- 8.1 Volumetric flow conversions are as follows:

$$1 \text{ SCFM} = 0.0283 \text{ m}^3/\text{min} = 28.317 \text{ L/min}$$

## 9.0 Quality Control/Quality Assurance

### 9.1 Tunnel Blank Sample

- 9.1.1 A tunnel blank sample must be collected each analysis day. The levels of any unlabelled analyte quantified in the tunnel blank must not exceed 20 % of the level of that analyte in the dilute exhaust sample. If this criterion cannot be met, calculate a reporting limit that is five times the blank value. Do not subtract the blank value from the sample value.

## 9.2 Field Blank Sample

9.2.1 At least one XAD and particulate filter per batch is analyzed as a field blank. The levels of any unlabelled analyte quantified in the tunnel blank must not exceed 20 % of the level of that analyte in the dilute exhaust sample. If this criterion cannot be met, calculate a reporting limit that is five times the blank value.

## 9.3 Leak Checks

### 9.3.1 Sampling Train

9.3.1.1 A leak check must be performed each analysis day to ensure correct sampling system flow rates.

9.3.1.2 To leak check any part of the sampling train, the suspected leak area should be isolated and pressurized or put under vacuum. A gauge may then be used to check if a leak exists.

9.3.1.3 Flow rate must be kept within 2 L/min of the set sampling flow rate of 100 L/min.

## 9.4 Flow Rates

9.4.1 The flow rate into the particulate filter and XAD cartridge is controlled with a digital flow meter. The flow rate over the transient or steady state emission test cycle is 100 L/min.

## 10.0 References

10.1 Desert Research Institute, 4 Channel Sequential FP/SVOC Sampler, 1-750.4, Revision 05, July 2002.



## 1.0 Scope and Application

- 1.1 This exhaust gas sampling protocol is specifically designed for sampling dilute exhaust generated from heavy-duty diesel engines being operated over the EPA transient cycle or steady state emission test as described in 40 CFR Part 86.
- 1.2 For transient cycle operation, this official testing protocol involves continual sample integration of all gaseous emissions along with pertinent engine and ambient variables for 1200 seconds (20 minutes). For eight-mode steady state operation, this official testing protocol involves modal sample collection for a total of 20 minutes. Modes 1, 2, 3 and 8 are collected for 3 minutes of the 5 minute mode, while modes 4, 5, 6 and 7 are collected for 2 minutes.
- 1.3 Dilute exhaust samples are collected from the dilution tunnel.

## 2.0 Method Summary

- 2.1 Samples analyzed for aldehyde and ketone compounds (carbonyls) are collected continuously by pulling the dilute exhaust from the dilution tunnel, using a Teflon diaphragm pump, through a series of two DNPH cartridges.
- 2.2 The absorbing solution (2,4-DNPH) complexes the carbonyl compounds into their diphenylhydrazone derivatives.
- 2.3 Once received by the laboratory, cartridges are eluted with 5 mL acetonitrile and analyzed via high performance liquid chromatography (HPLC).

## 3.0 Health and Safety

- 3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.
- 3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

## 4.0 Sample Preservation, Containers, Handling, and Storage

- 4.1 Samples are collected and stored in DNPH-impregnated cartridges.
- 4.2 DNPH cartridges must be sealed and refrigerated, at a temperature less than 40° F, upon receipt from manufacturer, until ready for use.
- 4.3 If samples are not analyzed the same day as collected, they must be refrigerated at a temperature below 40° F.
- 4.4 Refrigerated samples are stable for up to 30 days.

**5.0 Interferences and Potential Problems**

5.1 To decrease the chance of background contamination variables and oxygenated impurities, DNPH-impregnated cartridges are used rather than impingers.

**6.0 Equipment/Apparatus**

6.1 Sep-Pak® DNPH-impregnated cartridges: Waters Corporation, or equivalent

6.2 Digital flow meter: Dwyer Instruments, Inc., or equivalent.

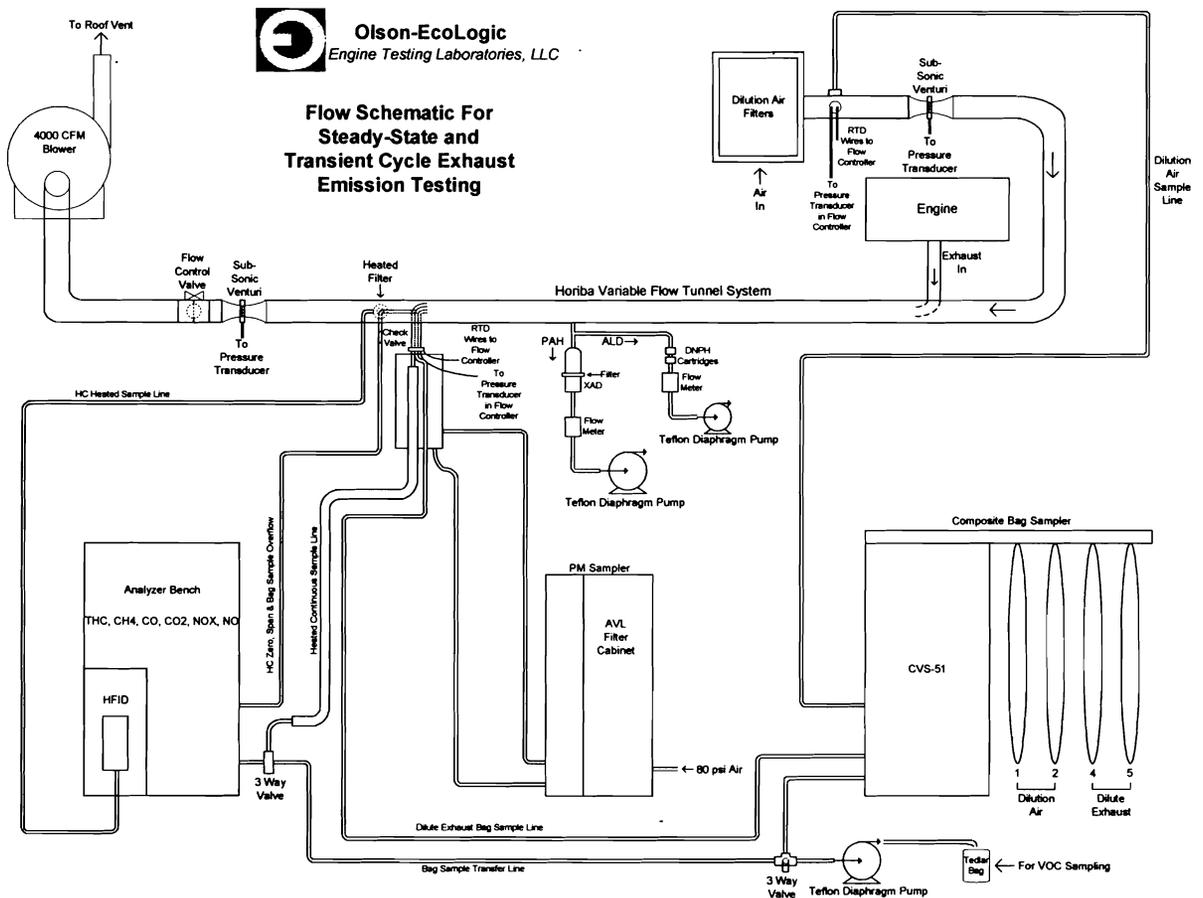
6.3 Teflon diaphragm vacuum-pressure pump: MFG Corp., or equivalent.

**6.4 Sampling Train Schematic**

6.4.1 8' Gas line from the tunnel to the PAH/ALD sampling system is as follows:

6.4.1.1 Siltek®/Sulfinert® treated ½” 316L grade stainless steel tubing: Restek, or equivalent.

6.4.2 After the “T” connection, all ALD sampling line is ¼” Teflon tubing.



## 7.0 Procedure

### 7.1 Sample collection

7.1.1 Two DNPH cartridges connected into the sample flow path.

7.1.2 Dilute exhaust samples are collected onto the DNPH cartridges by turning on the power to the Teflon diaphragm pump.

7.1.2.1 For transient test cycle sample collection, the Teflon diaphragm pump is turned on for the duration of the 1200 second emission test.

7.1.2.2 For steady state cycle sample collection, the Teflon diaphragm pump is turned on for 3 minutes at the end of modes 1, 2, 3 and 8; and 2 minutes at the end of modes 4, 5, 6 and 7.

7.1.3 The flow rate through the DNPH cartridges should be 1 L/min.

7.1.3.1 Flow rate should be continuously monitored and adjusted if necessary during sampling.

7.1.4 The dilution and work is noted for each dilute exhaust sample taken for final calculations.

7.1.5 DNPH cartridges are refrigerated immediately after sample collection, below a temperature of 40° F, until analyzed by HPLC.

## 8.0 Calculations

8.1 Volumetric flow conversions are as follows:

$$1 \text{ SCFM} = 0.0283 \text{ m}^3/\text{min} = 28.317 \text{ L}/\text{min}$$

## 9.0 Quality Control/Quality Assurance

### 9.1 Tunnel Blank Sample

9.1.1 A tunnel blank sample must be collected each analysis day. If the tunnel blank shows a peak greater than the method detection limit (MDL) in the region of interest, the source of the contamination must be investigated and remedied. Do not subtract the blank value from the sample value.

### 9.2 Field Blank Sample

9.2.1 At least one cartridge per batch is analyzed as a field blank. If the cartridge blank shows a peak greater than the method detection limit in the region of interest, the source of the contamination must be investigated and remedied.

### 9.3 Leak Checks

#### 9.3.1 Sampling Train

9.3.1.1 A leak check must be performed each analysis day to ensure correct sampling system flow rates.

9.3.1.2 To leak check any part of the sampling train, the suspected leak area should be isolated and pressurized or put under vacuum. A gauge may then be used to check if a leak exists.

9.3.1.3 Flow rate must be kept within 0.2 L/min of the set sampling flow rate of 1 L/min.

### 9.4 Flow Rates

9.4.1 The flow rate into the DNPH cartridge is controlled with a digital flow meter. The flow rate over the transient or steady state emission test cycle is 1 L/min.

## 10.0 References

10.1 Desert Research Institute, DRI Carbonyl Sampler, 1-710.3, Revision 03, June 1997.

## 1.0 PURPOSE/APPLICABILITY

This analytical method applies to dinitrophenylhydrazine-impregnated cartridges through which air samples have been passed for the collection of carbonyl compounds. Carbonyl compounds react rapidly with 2,4-dinitrophenylhydrazine (DNPH) in acidic media to form yellow to orange-colored hydrazones. The color depends on the type of carbonyl compound, and the amount of the product formed is dependent on the quantity of carbonyl compound provided to the reagent.

Samples are collected by drawing a known volume of air through the DNPH-impregnated cartridges. These exposed cartridges are then returned to the laboratory for isolation, separation, and quantification of the hydrazone products by high-performance liquid chromatography (HPLC). The ambient air concentration of various carbonyl compounds is determined from the quantity of the associated hydrazones found in the exposed cartridges and the volume of air samples. Typically C<sub>1</sub>-C<sub>6</sub> carbonyl compounds, including benzaldehyde, are measured effectively by this technique, with a detection limit of ~ 0.1 ppbv.

This method follows the procedure described in EPA Method TO-11A (January 1997, EPA/625/R-96/010b).

## 2.0 MATERIALS/APPARATUS

### 2.1 DNPH Cartridges

Waters Sep-Pak XPoSure Aldehyde Samplers are purchased from Waters (WAT047205) and sampled directly from the manufacturer without need for laboratory preparation.

## 3.0 SAMPLE ANALYSIS

Aldehydes collected in the cartridge (as the hydrazones) are eluted with acetonitrile and the eluent is analyzed using reverse phase high performance liquid chromatograph (HPLC) (Fung and Grosjean, 1981). Gradient elution is used with a sufficiently polar mobile phase for the separation of acetone, acrolein, and propanal. The mobile phase polarity is then decreased steadily to allow the elution of the higher aldehydes.

### 3.1 Sample Shipping/Storage

The cartridges are returned to the laboratory in secondary aluminum envelopes provided by the manufacturing, labeled with unique Project Media Identification (PMI) numbers, in a cooler at 4°C. In the laboratory, they are stored in a refrigerator until analysis. The time between sampling and extraction should not exceed two weeks. Sample elutes are stable at 4 °C for up to one month.

### 3.2 Sample Preparation

Uncap the cartridge and place it in a small test tube holder. Elute the cartridges with 2 ml of acetonitrile into a volumetric flask and transfer into a septum vial. Cap the vial and write the cartridge number on the side using a permanent marker. If not analyzed the same day, place the extracts in a clean refrigerator.

### 3.3 Standard Solutions

Prepare stock solutions of the hydrazone standards provided by AccuStandard in acetonitrile at carbonyl concentration of 5 µg/mL.

Dilute the stock standards to obtain working solutions in the range of 0.1 to 10 µg/mL concentrations for most applications. Higher concentrations may be needed occasionally if the air carbonyl concentrations exceed ~20 ppb during sampling. At least three concentrations of working standards bracketing the sample concentrations should be prepared for the calibration.

A secondary standard from Restek is used to verify the calibration.

### 3.5 Instrument Conditions

The HPLC instrument is a Waters 2695 Alliance Separation Module with a photo diode array (PDA) detector with Empower software. Data are collected between a wavelength range of 190-450 nm.

## Water Alliance 2695 Gradient Acquisition Method for Carbonyls

Solvent A: 100% HPLC Water  
Solvent B: 100% Optima Acetonitrile  
Solvent C: 100% Optima Methanol  
Column: Polaris C18-A, 4.6x15, 3um  
Detector: PDA, 360 nm

Table 1. Gradient elution solvent conditions.

Time (min)	Flow (mL)	%A	%B
0.01	1.50	70.0	30.0
1.00	1.50	70.0	30.0
20.00	1.50	60.0	40.0
32.00	1.50	55.0	45.0
41.00	1.50	37.0	63.0
45.00	1.50	37.0	63.0
50.00	1.50	30.0	70.0
55.00	1.50	70.0	30.0

The column is conditioned with 50:50 water/methanol for 10 minutes at the end of each sequence.

### 3.6 Calibrations

Following the Millennium<sup>32</sup> 3.20 PDA document, five calibration standards are analyzed using concentrations in Table 2 (see Figures 2 for example of calibration curve). The curve is forced through zero and is acceptable with a  $R^2 \geq 0.95$ . A secondary standard from Restek is used to verify the calibration.

Once the linear response factor has been documented, an intermediate concentration standard near the anticipated level of each component (but at least ten times the detection limit) is used for a daily calibration check standard. A sample chromatogram is shown in Figure 1.

### 3.7 Data Acquisition and Processing

The calibration equation below is used to determine the amount of carbonyls in the samples (this step is performed by the HPLC baseline data system).

Table 2. Calibration Levels

	Level 1	Level 2	Level 3	Level 4	Level 5
Conc. (ug/mL)	1	0.5	0.25	0.1	0.025
Formaldehyde	7.036	3.518	1.759	0.703	0.351
Acetaldehyde	5.13	2.565	1.282	0.513	0.256
Acetone	4.1	2.05	1.025	0.41	0.205
Acrolein	4.366	2.183	1.091	0.436	0.218
Propionaldehyde	4.113	2.056	1.028	0.411	0.2055
Crotonaldehyde	3.57	1.785	0.892	0.357	0.178
2-Butanone (MEK)	3.5	1.75	0.875	0.35	0.175
Methacrolein	3.57	1.785	0.892	0.357	0.178
n-Butyraldehyde	3.51	1.755	0.877	0.351	0.1755
Benzaldehyde	2.7	1.35	0.675	0.27	0.135
Valeraldehyde	3.122	1.561	0.78	0.312	0.156
Glyoxal	0.92	0.46	0.23	0.046	0.023
m-Tolualdehyde	2.506	1.253	0.626	0.25	0.125
Hexaldehyde	3.116	1.558	0.779	0.311	0.155

### 3.8 Calculations

$$C_A = \frac{W_d}{V_m \text{ (or } V_s)} \times 1000$$

where:

$C_A$  = concentration of analyte (ng/L) in the original sample  
 $W_d$  = total quantity of analyte ( $\mu\text{g}$ ) in sample, blank corrected

$$W_d = W \times V_E$$

where:

$W$  = Concentration of analyte in the cartridge ( $\mu\text{g}$ )  
 $V_E$  = final volume (ml) of sample extract  
 $V_m$  = total sample volume (L) under ambient conditions  
 $V_s$  = total sample volume (L) at 25 °C and 760 mm Hg

The analyte concentrations can be converted to ppbv using the following equation:

$$C_A (\text{ppbv}) = C_A (\text{ng/L}) \times \frac{24.4}{MW_A}$$

where:

$C_A(\text{ppbv})$  = concentration of analyte in parts per billion by volume  
 $C_A(\text{ng/L})$  is calculated using  $V_s$

$MW_A$  = molecular weight of analyte.

### 3.9 Quality Control

After calibration, an intermediate concentration calibration check standard is analyzed every 10 samples with a  $\pm 10\%$  recovery.

#### 3.9.1 Blanks

At least one field blank or 10% of the field samples, whichever is larger, should be shipped and analyzed with each group of samples. The field blank is treated identically to the samples except that no air is drawn through the cartridge.

Instrument blanks are analyzed after high concentrations.

#### 3.9.2 Method Precision and Accuracy

10% of the samples are analyzed twice for replicate precision which typically falls within  $\pm 10\%$ .

## 4.0 REFERENCES

Fung, K., and D. Grosjean (1981). "Determination of Nanogram Amounts of Carbonyls as 2,4, dinitrophenylhydrazones by High Performance Liquid Chromatography." *Analy. Chem.*, 53, 168.

U.S. EPA, "Method TO-11A: Determination of Formaldehyde in Ambient Air Using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC) [Active Sampling Methodology]," EPA/625/R-96/010b, in *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*.

Title: Analysis of Carbonyl Compounds  
by High Performance Liquid Chromatography

Number: 2-710.4

Revision: 04

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Winberry, W.T., Jr., N.T. Murphy, and R.M. Riggan (1988). *Method TO11 in Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*. EPA/600/4-89/017, U.S. Environmental Protection Agency, Research Triangle Park, NC.



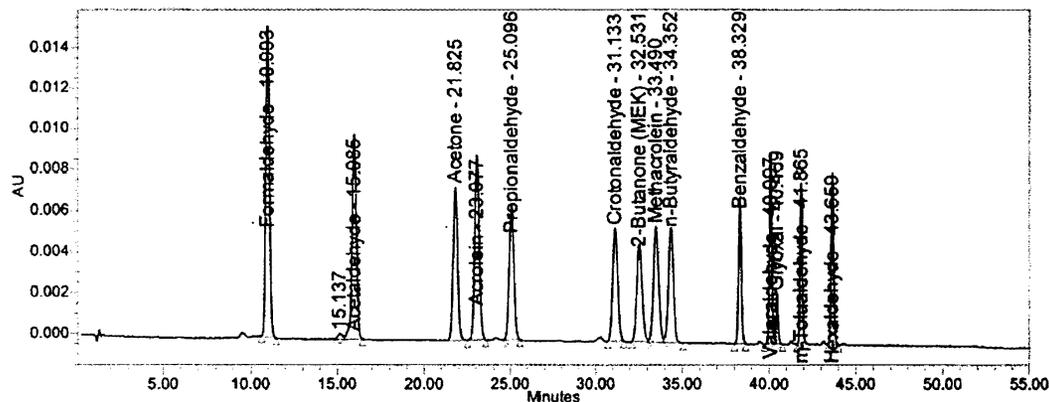
# Sample Report

Reported by User: Katarzy na Rempala Project Name: API\_Winter2004

# of Results: 5

## SAMPLE INFORMATION

Sample Name:	0404level2	Acquired By:	Katy
Sample Type:	Standard	Date Acquired:	4/6/2005 3:56:28 PM
Vial:	3	Acq. Method Set:	Polaris 3um_MS
Injection #:	1	Date Processed:	4/7/2005 11:09:16 AM
Injection Volume:	20.00 ul	Processing Method:	Polaris0405PM
Run Time:	55.0 Minutes	Channel Name:	Extract 360.0
Sample Set Name:	API_040505	Proc. Chnl. Descr.:	PDA 360.0 nm



Peak Name	RT	Area	Amount	Units	PDA Match1 Spect. Name	Match1	PDA Match2 Spect. Name	Match2
1 Formaldehy de	10.993	233572	3.518	ug/ml	Formaldehy de	0.851		
2 Acetaldehy de	15.985	176681	2.565	ug/ml	Acetaldehy de	5.705	n-Buty raldehy de	5.709
3 Acetone	21.825	137633	2.050	ug/ml	Acetone	1.322	2-Butanone (MEK)	1.870
4 Acrolein	23.077	167748	2.183	ug/ml	Acrolein	1.085	Methacrolein	3.072
5 Propionaldehy de	25.096	129540	2.056	ug/ml	n-Buty raldehy de	1.448	Acetaldehy de	1.653
6 Crotonaldehy de	31.133	114907	1.785	ug/ml	Crotonaldehy de	1.065	Methacrolein	6.077
7 2-Butanone (MEK)	32.531	102244	1.750	ug/ml	2-Butanone (MEK)	1.621	Acetone	2.927
8 Methacrolein	33.490	122150	1.785	ug/ml	Methacrolein	3.741	Acrolein	5.489
9 n-Buty raldehy de	34.352	112621	1.755	ug/ml	n-Buty raldehy de	3.275	Valeraldehy de	3.330
10 Benzaldehy de	38.329	82427	1.350	ug/ml	Benzaldehy de	1.003	m-Tolualdehy de	3.987

Figure 1a. Chromatograph of Level 2 standard.



## Sample Report

Reported by User: Katarzyna Rempala Project Name: API\_Winter2004

# of Results: 5

	Peak Name	RT	Area	Amount	Units	PDA Match1 Spect. Name	Match1	PDA Match2 Spect. Name	Match2
11	Valeraldehy de	40.097	97770	1.561	ug/ml	Valeraldehy de	5.956	n-Buty raldehy de	6.388
12	Gly oxal	40.469	21352	0.460	ug/ml				
13	m-Tolualdehy de	41.865	70294	1.253	ug/ml	m-Tolualdehy de	0.975	Benzaldehy de	3.809
14	Hexaldehy de	43.659	84775	1.558	ug/ml	Hexaldehy de	1.304	Valeraldehy de	1.531

Figure 1b. Chromatograph of Level 2 standard.

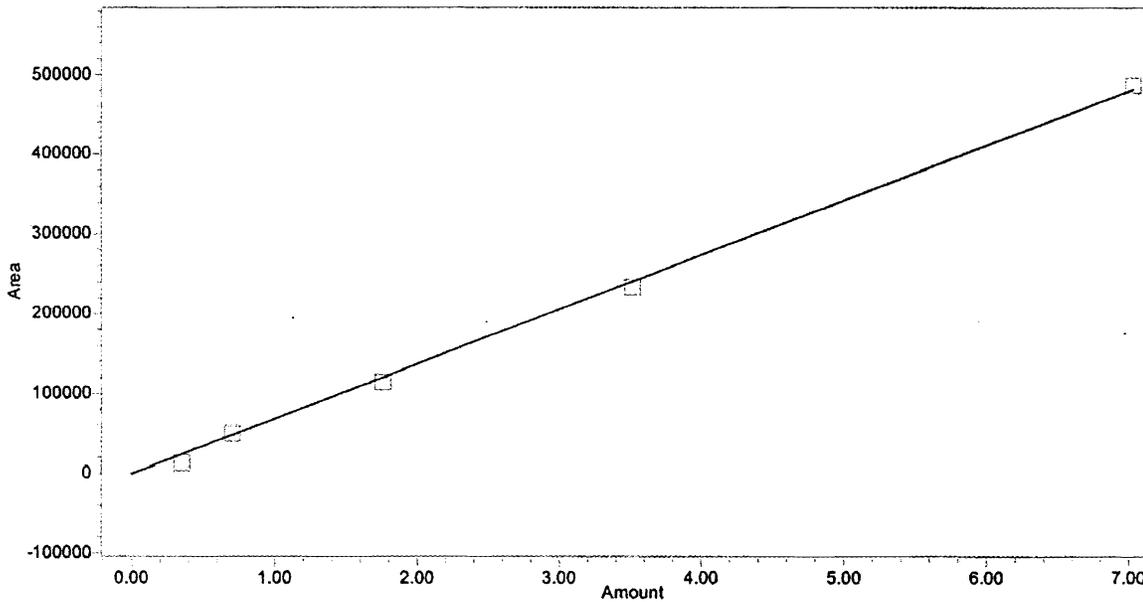


### LC Calibration Report ver1

Reported by User: Katarzyna Rempala (Katy)

Project Name: API\_Winter2004

Processing Method: Polaris0405PM	Project Name: API_Winter2004
Processing Method ID 1175	System: W2695
Calibration ID: 1159	Channel: Extract 360.0
Date Calibrated: 4/7/2005 11:13:37 AM	Proc. Chnl. Descr. PDA 360.0 nm



Name: Formaldehyde; RT: 11.360; Fit Type: Linear thru Zero; Cal Curve Id: 1160; R: 0.999224; R<sup>2</sup>: 0.998448; Weighting: None; Equation: Y = 6.84e+004 X

Figure 2. Calibration curve for formaldehyde.

## 1.0 PURPOSE/APPLICABILITY

This method describes the analysis of semi-volatile organic compounds (SVOC) in air. The SVOCs include non-polar analysis of Polycyclic Aromatic Hydrocarbons (PAH), Aliphatic Hydrocarbon Analysis (Alkanes), Hopanes and Steranes, and Polar analysis. The method uses a sampling train consisting of a Teflon-impregnated glass fiber (TIGF) filter backed up by a PUF/XAD/PUF sandwich solid adsorbent. The separate portions of the sampling train are extracted and combined dependent on analyses. The analysis method is gas chromatography/mass spectrometry (GC/MS). Mass spectrometry provides definitive identification of SVOCs.

This method follows the procedure described in EPA Method TO-13 (June 1988, EPA/600-4-89/017). The exceptions are that 1) the DRI procedure uses a XAD-4 sandwich adsorbent trap where TO-13 recommends either PUF or XAD-2, and 2) the DRI procedure calls for more rigorous cleaning than the EPA method.

## 2.0 MATERIALS/APPARATUS

### 2.1 Sampling Substrates

100 mm TIGF filters (Pall Gellman, ultrapure quality), PUF, and XAD-4 (Fisher Scientific) are obtained. Cleaning is as per Section 4 below. All solvents are Fisher Scientific Optima or HPLC grade.

### 2.2 GC/MS

The chromatographic system consists of a Varian CP-3800 gas chromatograph equipped with an 8200 CX Autosampler and interfaced to a Vairan Saturn 2000 Ion Trap Mass Spectrometer. The alternative system consists of a Varian CP-3800 gas chromatograph with a model CP-8400 Autosampler and interfaced to a Saturn 2000 Ion Trap Mass Spectrometer. Column is a CP-Sil8 30mx0.25 mmX025XX (Chrompack).

## 3.0 PERSONNEL QUALIFICATION

This SOP assumes that personnel performing the procedures are familiar with basic laboratory practice and operation of Dionex Accelerated Solvent Extractor (ASE), rotary evaporators, and the Varian GC/MS system and Saturn Workstation 5.2 computer software. Specific requirements for these instruments are found in the appropriate manuals.

## 4.0 SUBSTRATE CLEANING PROCEDURE

### 4.1. Filters

Teflon-impregnated glass fiber (TIGF) filters (Pall Life Sciences, Type T60A20) are cleaned by sonication for 10 minutes in dichloromethane ( $\text{CH}_2\text{Cl}_2$ ) twice, with the solvent replaced and drained, and sonicated for 10 minutes in methanol twice with the solvent replaced. Filters are then dried in a vacuum oven at  $-15$  to  $-20$  in Hg,  $50^\circ\text{C}$  for minimum of 24 hours, weighed (if necessary), placed in foil packages that have been fired at  $500^\circ\text{C}$  for 4 hours, placed in Uline metallic ZipTop static shielding bags, and stored at room temperature.

If quartz filters (Pall Gellman, ultrapure quality), are used, they are baked at  $900^\circ\text{C}$  for 4 hr before use.

### 4.2 PUF Plugs

PUF plugs are cleaned by first washing with distilled water, followed by Dionex ASE extraction for 15min/cell with  $\sim 170$  mL acetone at 1500 psi and  $80^\circ\text{C}$ , followed by Dionex ASE extraction for 15min/cell with  $\sim 170$  mL of 10% diethyl ether in hexane under the same conditions. The extracted PUF plugs are dried in a vacuum oven at  $-15$  to  $-20$  in Hg,  $50^\circ\text{C}$  for approximately 3 days or until no solvent odor is detected. If storage is necessary, PUF plugs are stored in clean 1L glass jars with Teflon lined lids wrapped in aluminum foil. Powder-free nitrile gloves are worn at all times when handling PUF plugs.

### 4.3 XAD-4

New XAD-4 is washed with Liquinox<sup>TM</sup> soap and hot water, followed by DI water. It is then placed in a Buchner funnel under vacuum, then transferred to the Dionex ASE and extracted for 15min/cell with  $\sim 170$  mL of methanol at 1500 psi and  $80^\circ\text{C}$ , followed by dichloromethane ( $\text{CH}_2\text{Cl}_2$ ), then acetone under the same instrument conditions. The XAD-4 is then dried in a vacuum oven at  $-15$  to  $-20$  in Hg and  $50^\circ\text{C}$ . The cleaned XAD-4 is then transferred to a clean 1L glass jars with an air tight teflon-lined lid. The jar is wrapped with aluminum foil to protect the XAD-4 from light, and stored in a clean room at room temperature.

### 4.4 Certification of Substrate

An aliquot of each batch of cleaned XAD-4 (20g) and TIGF filters are extracted same as samples. Deuterated standards are added to the sample prior to extraction in the Dionex

ASE with ~170 mL dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) for 15 min/cell at 1500 psi and 80°C, followed by ~170 mL acetone extraction under the same conditions. The extract is then concentrated to 1ml and analyzed by GC/MS. Any batch determined to have excessive impurities (more than 10 ng/ul of naphthalene and other compounds in method) will be re-cleaned and checked again for purity.

#### **4.5 Assembly of XAD and PUF/XAD/PUF Cartridge**

The glass cartridges and screen assemblies are washed with Liquinox™ soap and hot water followed by DI water and oven dried. Powder-free nitrile gloves are worn at all times during the cartridge assembly. For XAD-4 cartridges, one assembly of spring, o-ring and screen is placed at the bottom of a clean glass cartridge followed by 20g of XAD-4 and another assembly of screen, o-ring and spring. The XAD cartridge is then placed in Uline ZipTop metallic static shielding bags and stored in a clean room at room temperature.

For PUF/XAD-4/PUF cartridges, one PUF plug is put at the bottom of a clean glass cartridge followed by 10 g of XAD-4 and a second PUF plug. The PUF/XAD/PUF cartridge is then placed in Uline ZipTop metallic static shielding bags and stored at room temperature.

#### **5.0 SAMPLE SHIPPING, RECEIPT, AND STORAGE**

XAD-4 cartridge and filter sets are assigned a unique Project Media Identification (PMI) number and logged (date stamped) into the Laboratory Information Management System (LIMS) when assembled and shipped. Cartridges are packed in a tin can with field data sheets with the same unique PMI number and shipped in coolers on blue ice prior overnight.

In the field, exposed samples are stored at 0-4°C in a refrigerator or freezer and shipped to DRI priority overnight in ice chest (DRI's original shipping containers) with blue ice. Upon receipt by the laboratory, the samples are logged into the LIMS by PMI number, and field data is recorded (sampling location, date, and start and stop time, elapse timer, and flow rate). If the time span between sample login and extraction is greater than 24 hours, the samples must be kept cold at 0-4°C in a freezer or refrigerator. The exposure of the sample media to ultraviolet light emitted by fluorescent lights must be minimized.

#### **6.0 EXTRACTION OF SUBSTRATE**

##### **6.1 Addition of Internal Standards**

### 6.1.1 Polycyclic Aromatic Hydrocarbon (PAH), non-polar

Prior to extraction, the following deuterated internal standards are added to each sample (filter, PUF/XAD/PUF):

naphthalene-d <sub>8</sub>	9.486	ng/μl
biphenyl-d <sub>10</sub>	7.008	ng/μl
acenaphthene-d <sub>10</sub>	5.997	ng/μl
phenanthrene-d <sub>10</sub>	5.991	ng/μl
anthracene-d <sub>10</sub>	5.000	ng/μl
pyrene-d <sub>12</sub>	4.993	ng/μl
benz(a)anthracene-d <sub>12</sub>	2.004	ng/μl
chrysene-d <sub>12</sub>	1.997	ng/μl
benzo[k]fluoranthene-d <sub>12</sub>	1.000	ng/μl
benzo[e]pyrene-d <sub>12</sub>	0.700	ng/μl
benzo[a]pyrene-d <sub>12</sub>	0.703	ng/μl
benzo[g,h,i]perylene-d <sub>12</sub>	0.600	ng/μl
coronene-d <sub>12</sub>	0.500	ng/μl

The amount of internal standards added should correspond to the expected range of concentrations found in real samples and the final volume of extracts during analysis.

### 6.1.2 Hopane and Sterane, non-polar

Prior to extraction, the following deuterated internal standards are added to each sample (filter, PUF/XAD/PUF):

cholestane- d <sub>6</sub>	0.375	ng/μl
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The amount of internal standards added should correspond to the expected range of concentrations found in real samples.

### 6.1.3 Aliphatic Hydrocarbon Analysis (Alkanes), non-polar

Prior to extraction, the following deuterated internal standards are added to each sample (filter, PUF/XAD/PUF):

dodecane-d <sub>26</sub>	10.9	ng/μl
hexadecane-d <sub>34</sub>	2.36	ng/μl
eicosane-d <sub>42</sub>	1.88	ng/μl
octacosane-d <sub>58</sub>	4.9	ng/μl
tetracosane-d <sub>50</sub>	1.89	ng/μl

hexatriacontane-d<sub>74</sub> 10.2 ng/μl

The amount of internal standards added should correspond to the expected range of concentrations found in real samples.

#### 6.1.4 Polar Organic Compounds, polar

Prior to extraction, the following deuterated internal standards are added to each sample (filter-sorbent pair):

cholesterol-2,2,3,4,4,6-d <sub>6</sub>	9.85	ng/μl
levoglucosan-u-13C <sub>6</sub>	31.25	ng/μl
hexanoic-d <sub>11</sub> acid	4.5	ng/μl
benzoic-d <sub>3</sub> acid	4.5	ng/μl
decanoic-d <sub>19</sub> acid-	4.5	ng/μl
palmitic-d <sub>31</sub> acid	4.5	ng/μl
heptadecanoic-d <sub>33</sub> acid	4.4	ng/μl
myristic-d <sub>27</sub> acid	3.3	ng/μl
succinic-d <sub>4</sub> acid	2.55	ng/μl
phthalic 3,4,5,6-d <sub>4</sub> acid	4.6	ng/μl

The amount of internal standards added should correspond to the expected range of concentrations found in real samples and the final volume of extracts during analysis.

## 6.2 Extraction of PUF, XAD-4, and Filter

Depending on analyses, PUF, XAD-4 and Filter will be extracted in the following combinations. Solvents are selected to optimize the polarity range desired for analyses.

### 6.2.1 Non-Polar Analysis Only

Filters and XAD-4 are extracted twice with approximately ~170 mL of dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) using the Dionex ASE for 15 min/cell at 1500 psi and 80°C.

Since PUF media degrades when extracted with dichloromethane, the PUFs are extracted twice with ~170 mL of acetone using the Dionex ASE for 15 min/cell at 1500 psi and 80°C. This method gives good recovery for PAH, aliphatic hydrocarbons (alkanes), and hopanes and steranes.

### 6.2.2 Polar and Non-Polar Analyses

Filters and XAD-4 are extracted with ~170 mL dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) using the Dionex ASE for 15 min/cell at 1500 psi and 80°C followed by ~170 mL acetone extraction under the same conditions.

Since PUF media degrades when extracted with dichloromethane, the PUFs are extracted twice with ~170 mL of acetone using the Dionex ASE for 15 min/cell at 1500 psi and 80°C. This method gives good recovery for PAH, aliphatic hydrocarbons (alkanes), hopanes and steranes, and polar organic compounds.

### 6.3 Treatment of Extracts

#### 6.3.1 Non-Polar Analysis Only

Extracts are concentrated to ~1ml by rotary evaporation at 35 °C under gentle vacuum, and filtered through a 0.2 µm Anotop™ 10 Whatman leuc-lock filter on 4 mL glass syringe), rinsing the flask 3 times with 1 ml dichloromethane and acetone (50/50 by volume) each time. Filtrate is collected in a 4 mL amber glass vial for a total volume of ~4 mL.

Approximately 200 µl of acetonitrile is added at this time and the extract is split into two fractions. Each fraction is then concentrated using a Pierce Reacti-Therm under a gentle stream of ultra-high purity (UHP) nitrogen with a water trap (Chrompack CP-Gas-Clean moisture filter 17971) to 100-200 µL. The final extract volume is adjusted to 100 µL with acetonitrile.

#### 6.3.2 Polar and Non-Polar Analyses

Extracts are concentrated to ~1ml by rotary evaporation at 35 °C under gentle vacuum, and filtered through a 0.2 µm PTFE disposable filter device (Whatman Pura disc™ 25TF), rinsing the flask 3 times with 1 ml dichloromethane and acetone (50/50 by volume) each time. Filtrate is collected in a 4 mL amber glass vial for a total volume of ~4 mL.

Approximately 200 µl of acetonitrile is added at this time and the extract is split into two fractions. Each fraction is then concentrated under a gentle stream of ultra-high purity (UHP) nitrogen with hydrocarbon and water traps to 100-200 µL. The final extract volume is adjusted to 100 µL with acetonitrile.

### 6.4 Cleanup of Samples (non-polar analysis)

For complex samples that contain analytical interference, the following method is used to

clean up the sample using silica gel semi-prep Solid Phase Extraction (SPE 6-mL 0.5-g LC-SI, Supelco Silica).

1. Assuming SVOC in 100  $\mu$ L acetonitrile, concentrate to 25  $\mu$ L and add 25  $\mu$ L dichloromethane and 150  $\mu$ L hexane.
2. Condition SPE-Silica cartridge with 1.5 mL hexane/benzene (1:1), followed by 1.5 mL hexane.
3. Transfer sample into the SPE-Silica cartridge.
4. Elute sample with 1.5 mL hexane, followed by 3 mL hexane/benzene (1:1) in separate 4 mL vials.
5. Concentrate to 100  $\mu$ L (only hexane should remain) and transfer to GC vial insert and concentrate to 20  $\mu$ L.
6. Rinse original vial with 100  $\mu$ L dichloromethane and concentrate to 40  $\mu$ L (hexane/DCM (1:1)) and dilute to total volume of 100  $\mu$ L with acetonitrile.

The hexane fraction contains the non-polar aliphatic hydrocarbons (alkanes), and hopanes and steranes, and the hexane/benzene fraction contains the PAH and N-PAH.

## 6.5 Silylation of Polar Organic Compounds (polar analysis)

If extracts have been split for polar and non-polar analysis, the fraction for the polar analysis is derivatized using a mixture of bis(trimethylsilyl)trifluoroacetamide and pyridine to convert the polar compounds into their trimethylsilyl derivatives for analysis of organic acids, cholesterol, sitosterol, and levoglucosan. Depending upon the expected range of analytes, it is recommended to split the second fraction into two equal fractions, thus providing a second opportunity for a clean silylation reaction.

1. The extract is reduced to a volume of 50  $\mu$ L using a Pierce Reacti-Therm under a gentle stream of ultra-high purity (UHP) nitrogen with a water trap (Chrompack CP-Gas-Clean moisture filter 17971).
2. 50  $\mu$ L of silylation grade pyridine is added to vial.
3. 150  $\mu$ L of bis(trimethylsilyl)trifluoroacetamide is added slowly to each vial and immediately capped.
4. The sample is then placed into thermal plates (custom) containing individual vial wells with the temperature maintained at 70°C for 3 hours.
5. The samples are then analyzed by GC/MS within 18 hours.

## 7.0 ANALYSIS

### 7.1 Instrument Method

The samples are analyzed by the electron impact (EI) GC/MS technique, using a Varian CP-3800 gas chromatograph equipped with a 8200 CX Autosampler and interfaced to a Vairan Saturn 2000 Ion Trap Mass Spectrometer or Varian CP-3400 gas chromatograph with a model CP-8400 Autosampler and interfaced to a Saturn 2000 Ion Trap Mass Spectrometer

Injections are 1  $\mu$ l in size in the splitless mode onto a 30m long 5% phenylmethylsilicone fused silica capillary column (J&W Scientific type DB-5ms): CP-Sil8 Chrompack (30m x 0.25mm x 0.25 mm) for PAH, hopanes and steranes, alkanes and polars; and CP-Sil24 Chrompack (30m x 0.25mm x 0.25 mm) for N-PAH.

Identification and quantification of the analytes are made by Selected Ion Storage (SIS), by monitoring the molecular ions of each analyte and each deuterated analyte.

## 7.2 Preparation Stage

A. The instrument (GC/MS) preparation steps are as follows:

- 1) Check for air and water in the system (Ion Time = 100, a total ion current (TIC) below 700 is preferred).
- 2) Adjust calibration gas pressure for Ion Trap instrument (75% preferred).
- 3) Check calibration gas pressure ~ 75%.
- 4) Perform autotune for electron multiplier setting, mass calibration, and RF ramp.

Identification and quantification of the analytes are made by Selected Ion Storage (SIS), by monitoring the molecular ions of each analyte and each deuterated analyte.

## 7.3 Calibration

Calibration curves are made by the molecular ion peaks of the analytes using the corresponding deuterated species as internal standards. If there is no corresponding deuterated species, the one most closely matching in volatility and retention characteristics is used.

National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1647 (certified PAH), with the addition of the internal standards listed in Section 6.1.1-6.1.4 and the targeted PAH not present in this mixture, is used to make calibration solutions. Six concentration levels for each analyte of interest are employed. Table 1 lists the concentration levels of standard compounds in calibration solutions. The calibration curve for each calibrated compound is constructed; Figures 1 through 6 show examples of acceptable calibration curves. After the calibration is completed, a standard

solution is injected to perform calibration checks. If deviations from the true values exceed  $\pm 20\%$ , the calibration procedure is repeated or new calibration levels must be prepared. One replicate analysis and one calibration check is performed for every 10 injections of samples. If difference between true and measured concentrations exceeds  $\pm 20\%$ , the system is recalibrated. During batch processing, calibration is performed before each batch.

## 8.0 REPORTING

Each sample is reported initially in terms of mass per sample ( $\mu\text{g}/\text{sample}$ ). Ambient concentrations in terms of mass per volume (i.e.,  $\text{ng}/\text{m}^3$  or other units if requested) are reported based upon the sample volume adjusted for ambient temperature and pressure, or reported as "standard" volume.

All information for the sample is recorded and combined into both a printed report and an Excel file for inclusion in the database (see Appendix).

### 8.1 Method Detection Limits (MDLs)

Method detection limits are 0.01-0.03 ng/ $\mu$ l for PAH, hopane and sterane, and alkane compounds, and 0.03-0.04 ng/ $\mu$ l for polar compounds.

### 8.2 Measurement Uncertainty

Measurement uncertainty is reported as one-sigma standard deviation between replicate tests (when 3 tests conducted under same conditions) or the combined root mean square of the analytical measurement uncertainty, which is defined by the following equation:

$$\sqrt{(\text{replicate precision} * \text{analyte concentration})^2 + (\text{analyte detection limit})^2}$$

This equation incorporates the analyte detection limit for each compound so when concentrations approach zero the error is reported as the analyte detection limit. When multiple samples are pooled the difference between samples is typically greater than the precision of any of the analytical techniques employed. Most data has relatively small reported measurement uncertainty's which shows the reproducibility of the samples. When larger errors (>30% of reported concentration) are observed, it is typically because the concentrations of the analyte were close to the detection limit of the measurements.

**Table 1. Calibration Levels for PAH analysis (bold compounds co-elute and are quantified together)**

Compound	Level 1 (ng/uL)	Level 2 (ng/uL)	Level 3 (ng/uL)	Level 4 (ng/uL)	Level 5 (ng/uL)	Level 6 (ng/uL)
1-ethylnaphthalene	0.359	0.718	1.436	2.873	11.491	45.965
1,2-dimethylnaphthalene	0.361	0.722	1.444	2.887	11.548	46.193
1,4-chrysenequinone	0.240	0.479	0.958	1.917	7.667	30.667
<b>1,6 + 1,3 dimethylnaphthalene</b>	<b>0.719</b>	<b>1.438</b>	<b>2.876</b>	<b>5.753</b>	<b>23.012</b>	<b>92.047</b>
1,8-dimethylnaphthalene	0.240	0.481	0.962	1.924	7.695	30.781
1-methylfluorene	0.298	0.596	1.192	2.383	9.533	38.133
1-methylphenanthrene	0.200	0.400	0.799	1.598	6.392	25.568
1-methylpyrene	0.240	0.481	0.961	1.922	7.688	30.752
1-phenylnaphthalene	0.199	0.398	0.796	1.591	6.365	25.461
2-ethylnaphthalene	0.357	0.714	1.428	2.856	11.424	45.696
<b>1,4+1,5+2,3-dimenaphlene</b>	<b>1.078</b>	<b>2.156</b>	<b>4.313</b>	<b>8.625</b>	<b>34.501</b>	<b>138.005</b>
2,6-dimethylnaphthalene	0.352	0.704	1.408	2.817	11.267	45.067
2-methylbiphenyl	0.360	0.720	1.441	2.881	11.525	46.102
2-methylphenanthrene	0.246	0.492	0.983	1.967	7.867	31.467
2-phenylnaphthalene	0.358	0.716	1.433	2.866	11.463	45.853
3,6-dimethylphenanthrene	0.203	0.406	0.813	1.625	6.500	26.000
3-methylbiphenyl	0.361	0.721	1.442	2.884	11.537	46.149
4H-cyclopenta(def)phenanthrene	0.000	0.000	0.000	0.000	0.000	0.000
4-methylbiphenyl	0.369	0.738	1.475	2.950	11.800	47.200
4-methylpyrene	0.240	0.479	0.958	1.917	7.667	30.667
<b>5+6 methylchrysene</b>	<b>0.559</b>	<b>1.119</b>	<b>2.237</b>	<b>4.475</b>	<b>17.899</b>	<b>71.595</b>
7-methylbenz(a)anthracene	0.279	0.558	1.117	2.233	8.933	35.733
7-methylbenzo(a)pyrene	0.290	0.579	1.158	2.317	9.267	37.067
9,10-dihydrobenzo(a)pyren-7(8H)-one	0.281	0.561	1.122	2.244	8.976	35.904
9-anthraldehyde	0.371	0.742	1.483	2.967	11.867	47.467
9-fluorenone	0.280	0.560	1.120	2.240	8.961	35.845
9-methylanthracene	0.239	0.479	0.958	1.916	7.663	30.653
acenaphthene*	0.201	0.402	0.804	1.609	6.435	25.739
acenaphthenequinone	0.202	0.404	0.808	1.617	6.467	25.867
acenaphthylene	0.200	0.400	0.800	1.600	6.400	25.600
anthrone	0.277	0.554	1.108	2.217	8.867	35.467
BaP*	0.160	0.321	0.642	1.283	5.133	20.533
benz(a)anthracene*	0.200	0.400	0.799	1.599	6.395	25.579

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Compound	0.279	0.558	1.117	2.233	8.933	35.733
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
	(ng/uL)	(ng/uL)	(ng/uL)	(ng/uL)	(ng/uL)	(ng/uL)
benz(a)anthracene-7,12-dione	0.360	0.720	1.440	2.880	11.518	46.073
benzanthrone	0.159	0.319	0.638	1.276	5.103	20.411
anthracene*	0.280	0.559	1.119	2.237	8.949	35.795
anthraquinone	<b>0.397</b>	<b>0.794</b>	<b>1.587</b>	<b>3.174</b>	<b>12.697</b>	<b>50.789</b>
<b>benzo(k*+b+j)fluoranthene</b>	0.200	0.400	0.800	1.600	6.401	25.602
benzo(g,h,i)perylene*	0.200	0.400	0.800	1.601	6.403	25.613
benzo(c)phenanthrene	0.240	0.479	0.958	1.917	7.667	30.667
benzonaphthothiophene	0.202	0.403	0.807	1.613	6.453	25.813
BeP*	0.190	0.379	0.758	1.517	6.067	24.267
chrysene*	0.160	0.320	0.640	1.280	5.118	20.474
coronene*	<b>0.323</b>	<b>0.645</b>	<b>1.291</b>	<b>2.582</b>	<b>10.327</b>	<b>41.307</b>
<b>dibenz(ah+ac)anthracene</b>	0.278	0.556	1.111	2.223	8.890	35.560
dibenzofuran	0.241	0.481	0.963	1.925	7.700	30.800
fluorene	0.252	0.503	1.006	2.013	8.050	32.200
fluoranthene	0.161	0.321	0.642	1.284	5.136	20.544
indeno(1,2,3-cd)pyrene	0.279	0.558	1.116	2.232	8.928	35.712
perinaphthenone	0.200	0.400	0.800	1.600	6.400	25.600
perylene	0.201	0.401	0.802	1.604	6.417	25.667
phenanthrene*	0.196	0.392	0.783	1.567	6.267	25.067
pyrene*	0.277	0.555	1.109	2.219	8.875	35.499
retene	0.199	0.399	0.797	1.594	6.378	25.511
2,3,5-trimethylnaphthalene	0.277	0.554	1.108	2.217	8.867	35.467
2,4,5-trimethylnaphthalene	0.239	0.478	0.957	1.914	7.654	30.616
1,4,5-trimethylnaphthalene	0.240	0.481	0.961	1.923	7.691	30.763
xanthone	0.361	0.723	1.446	4.338	17.351	69.403
1-methylnaphthalene	0.300	0.599	1.198	3.594	14.377	57.507
2,7-dimethylnaphthalene	0.360	0.720	1.440	4.319	21.597	107.983
bphenyl*	0.362	0.724	1.448	4.345	21.723	108.617
bibenzyl	0.430	0.860	1.720	5.160	25.800	129.000
2-methylnaphthalene	0.359	0.717	1.435	5.739	34.432	206.592
naphthalene*						

\*deuterated forms of these compounds are added to samples prior to extraction as surrogate for quantitation

**Table 2. Calibration Levels for Hopanes and Steranes Analysis**

Compound	Level 1	Level 2	Level 3	Level 4	
	ng/uL	ng/uL	ng/uL	ng/uL	ng/uL
cholestane-d6*	0.750	0.750	0.750	0.750	0.750
cholestane	0.250	0.500	1.000	2.000	4.000
17 $\alpha$ -21 $\beta$ (H) Hopane (19)	0.250	0.500	1.000	2.000	4.000
17 $\beta$ (H)-30-Norhopane (17a)	0.250	0.500	1.000	2.000	4.000
17 $\beta$ (H)-21 $\beta$ (H) Hopane (23)	0.250	0.500	1.000	2.000	4.000

\*deuterated forms of these compounds are added to samples prior to extraction as surrogate for quantitation

**Table 3. Calibration Levels for Aliphatic Hydrocarbon Analysis (Alkanes), bold compounds co-elute and are quantified together**

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
	ug/uL							
2,6,10-trimethylundecane_(norfarnesane)	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-heptylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
2,6,10-trimethyldodecane_(farnesane)	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-tetradecane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-pentadecane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-octylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-nonylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
<b>n-heptadecane + 2,6,10,14-tetramethylpentadecane_</b>								
<b>pristane</b>	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-hexadecane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
2,6,10-trimethylpentadecane_norpristane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-decylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-undecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-nonadecane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-octadecane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
2,6,10,14-	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000

## tetramethylhexadecane\_phytane

Compound	Level 1 ug/uL	Level 2 ug/uL	Level 3 ug/uL	Level 4 ug/uL	Level 5 ug/uL	Level 6 ug/uL	Level 7 ug/uL	Level 8 ug/uL
n-dodecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-tridecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-tetradecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-heneicosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-eicosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-pentadecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-docosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-tricosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-tetracosane-d50*	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-heptadecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-octadecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
<b>n-tetracosane* + n- hexadecylcyclohexane</b>	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-pentacosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-nonadecylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-heptacosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-eicosylcyclohexane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-hexacosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000
n-octacosane	0.050	0.500	1.000	100.000	200.000	2.500	5.000	10.000

\*deuterated forms of these compounds are added to samples prior to extraction as surrogate for quantitation

**Table 4. Calibration Levels for Polar Organic Compounds Analysis**

Compound	Level 1 ng/uL	Level 2 ng/uL	Level 3 ng/uL	Level 4 ng/uL	Level 5 ng/uL	Level 6 ng/uL
4-pentenoic	0.323	2.155	6.464	10.773	15.083	18.315
hexanoic acid	0.300	2.400	7.199	12.960	18.144	21.384
heptanoic	0.334	2.228	6.685	11.142	15.598	18.941
me-malonic	0.321	2.570	7.710	12.850	17.990	21.203
guaiacol	0.268	2.680	7.370	15.075	20.100	25.125
benzoic acid	0.300	2.400	7.199	12.960	18.144	21.384

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octanoic	0.314	2.091	6.272	10.453	14.635	17.771
glycerol	0.348	2.320	6.960	11.600	16.240	19.720

Compound	Level 1 ng/uL	Level 2 ng/uL	Level 3 ng/uL	Level 4 ng/uL	Level 5 ng/uL	Level 6 ng/uL
maleic	0.328	2.620	7.860	13.100	18.340	21.615
succinic acid	0.300	2.400	7.199	12.960	17.820	21.060
4-methylguaiacol	0.385	3.851	10.591	21.664	28.885	36.106
methylsuccinic acid	0.300	2.400	7.199	12.960	17.820	21.060
o-toluic	0.313	2.500	7.500	12.500	17.500	20.625
picolinic acid	0.300	2.400	7.199	12.960	18.144	21.384
m-tolic	0.327	2.613	7.840	13.067	18.293	21.560
1,2,4-butanetriol	0.300	2.400	7.199	12.960	18.144	21.384
nonanoic	0.318	2.120	6.360	10.600	14.840	18.020
p-toluic	0.169	1.128	3.384	5.640	7.896	9.588
3-methylpicolinic	0.321	2.568	7.704	12.840	17.976	21.186
6-methylpicolinic	0.319	2.550	7.650	12.750	17.850	21.038
2,6-dimethylbenzoic	0.269	2.150	6.450	10.750	15.050	17.738
4-ethylguaiacol	0.260	2.598	7.146	14.616	19.488	24.360
syringol	0.266	2.655	7.301	14.934	19.913	24.891
glutaric acid	0.300	2.400	7.199	12.960	17.820	21.060
2-methylglutaric	0.319	2.550	7.650	12.750	17.850	21.038
2,5-dimethylbenzoic	0.260	2.080	6.240	10.400	14.560	17.160
3-methylglutaric	0.261	2.085	6.256	10.427	14.597	17.204
2,4-dimethylbenzoic	0.263	2.100	6.300	10.500	14.700	17.325
3,5-dimethylbenzoic	0.256	2.050	6.150	10.250	14.350	16.913
2,3-dimethylbenzoic	0.272	2.172	6.516	10.860	15.204	17.919
n-decanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
4-allylguaiacol	0.284	2.843	7.817	15.990	21.320	26.650
4-methylsyringol	0.283	2.832	7.788	15.930	21.240	26.550
3,4-dimethylbenzoic	0.269	2.153	6.460	10.767	15.073	17.765
adipic acid	0.300	2.400	7.199	12.960	17.820	21.060
t-2-decenoic	0.318	2.123	6.368	10.613	14.859	18.043
cis-pinoic acid	0.300	2.400	7.199	12.960	17.820	21.060
3-methyladipic	0.328	2.623	7.868	13.113	18.359	21.637
4-formylguaiacol	0.283	2.832	7.788	15.930	21.240	26.550
undecanoic	0.315	2.523	7.570	12.617	17.663	20.818
isoeugenol	0.300	3.000	8.250	16.875	22.500	28.125
pimelic acid	0.300	2.400	7.199	12.960	17.820	21.060

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acetovanillone	0.266	2.655	7.301	14.934	19.913	24.891
lauric acid	0.300	2.400	7.199	12.960	17.820	21.060

Compound	Level 1 ng/uL	Level 2 ng/uL	Level 3 ng/uL	Level 4 ng/uL	Level 5 ng/uL	Level 6 ng/uL
phthalic acid	0.300	2.400	7.199	12.960	17.820	21.060
levoglucosan	0.300	2.400	7.199	12.960	18.144	21.384
syringaldehyde	0.266	2.655	7.301	14.934	19.913	24.891
tridecanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
suberic acid	0.300	2.400	7.199	12.960	17.820	21.060
isophthalic acid	0.300	2.400	7.199	12.960	17.820	21.060
azelaic acid	0.300	2.400	7.199	12.960	17.820	21.060
myristoleic	0.307	2.046	6.138	10.230	14.322	17.391
myristic acid	0.300	2.400	7.199	12.960	17.820	21.060
sebacic	0.165	1.098	3.294	5.489	7.685	9.332
pentadecanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
undecanedioic	0.165	1.099	3.296	5.493	7.691	9.339
palmitoleic	0.318	2.120	6.360	10.600	14.840	18.020
palmitic acid	0.300	2.400	7.199	12.960	18.144	21.384
isostearic	0.312	2.080	6.240	10.400	14.560	17.680
dodecanedioic acid	0.165	1.099	3.296	5.493	7.691	9.339
heptadecanoic	0.323	2.585	7.756	12.927	18.097	21.329
1,11-undecanedicarboxylic	0.171	1.141	3.424	5.707	7.989	9.701
oleic acid	0.300	2.400	7.199	12.960	18.144	21.384
elaidic acid	0.300	2.400	7.199	12.960	17.820	21.060
stearic acid	0.300	2.400	7.199	12.960	18.144	21.384
1,12-dodecanedioic	0.166	1.105	3.315	5.525	7.735	9.393
nonadecanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
dehydroabietic acid	0.300	2.400	7.199	12.960	17.820	21.060
eicosanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
pentadecanedioic acid	0.166	1.105	3.315	5.525	7.735	9.393
abietic acid	0.300	2.400	7.199	12.960	18.144	21.384
heneicosanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
docosanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
tricosanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
tetracosanoic acid	0.300	2.400	7.199	12.960	17.820	21.060
cholesterol	0.750	5.999	17.998	32.400	44.550	52.650
b-sitosterol	0.750	5.999	17.998	32.400	44.550	52.650

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DRI STANDARD OPERATING PROCEDURE

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APPENDIX

**SVOC Program Information**

I. Before Running

A. Each project must be listed in the database "H:\db\_prg\oalproj.dbf.". Fill in the following columns:

<b>Column</b>	<b>Value</b>
NUM	Use the next number in sequence
PROJ_NAME	A short description you will recognize
PROJ_CODE	The two-digit project code MUST be unique
ROOT_DIR	The directory where the project data are stored
STATUS	"c" for current, or "o" for old
SVOC	enter 1 to run the SVOC programs, 0 otherwise.

B. For each project, there is a list of target compounds for analysis. This list is in the directory "H:\db\_calib\svoc\" and it is called AAcmpd.dbf, where AA is the project code in the oalproj.dbf database. In this same directory is a database called "Template.dbf" which is a template you can copy to make the new ones. The fields you must fill in are:

<b>Column</b>	<b>Description</b>
Field_Name	The mnemonic for the PAH or PAH uncert.
Field_Type	ignore this
Field_Len	ignore this
Field_Dec	ignore this
Compound	The long name for the compounds only, enter nothing for uncert. This MUST exactly match the way it is in the mass spec calibration file.
Type	Enter "c" for a compound, nothing for uncert.

C. If you intend to import GCMS data, you must use Lantastic to attach the GC/MS computer's c: (hard) drive to a drive on the local machine.

II. Running

A. Run the genbatch program and follow inputs.

B. IF this is the first time you have worked on this project, you must first run the option "N" which creates a new set of files. This will make the files you will need.

C. You now can quit the programs and enter samples into the 'lab' database. This is the database the import program uses to determine what to import.

D. If the sample is run diluted, that file name and process status are also noted. When there is no diluted sample, just leave the name blank and set the dil\_f\_proc bit to zero. After the samples are imported, the program automatically enters a 2 for the proc bit.

Column	Description
PID	Standard ID
XMSFLAG	Mass Spec flag
F_NAME	Mass Spec file name for main analysis
F_PROC	Process bit for main (0=do nothing, 1= import normally, 2=import done).
DIL_F_NAME	Mass Spec file name for diluted analysis (if done)
DIL_F_PROC	Process bit for diluted (0=do nothing, 1= import normally, 2=import done).
SAMPLNO	Sample number
LOT	Lot numbers
ANALDATE	Date of analysis
COMMENTS	Notes

E. Once the import is done, AND the field data have been entered, you may continue with the rest of the processing, simply by following the sequence.

F. For the first batch of any project, the menu looks like:

```
** FILE CREATING FOR BATH 1 ONLY **  
N FOR Creating New Project Files  
6 FOR Importing XMS data.  
** Copying files from current Batch \data to \report  
3 FOR Copying Field data.  
4 FOR Copying analysis (xms) data.  
** Continue Processing Field  
5 FOR Processing Field data file.  
** Continue Processing Analysis (xms) file.  
7 FOR Running REP.  
8 FOR Merge FLD and XMS files to CHM file.  
9 FOR Calculate blank values and blank uncertainty.  
10 FOR Convert chm file to con file (ug/m3).
```

Simply follow the sequence through. Note, before going to Step 3 and beyond, you must first make sure the field and xms data are all input.

### III. Continuing a Project: Batch 2 and Following.

#### A. The menu for batch 2 and following looks like:

**\*\* Copying files from previous Batch \report to current \data directories**

- 1 FOR Copying Field data from Batch (prev) to (current).
- 2 FOR Copying analysis (xms) and LAB data from Batch (prev) to (current).
- 6 FOR Importing XMS data.

**\*\* Copying files from current Batch \data to \report**

- 3 FOR Copying Field data.
- 4 FOR Copying analysis (xms) data.

**\*\* Continue Processing Field**

- 5 FOR Processing Field data file.

**\*\* Continue Processing Analysis (xms) file.**

- 7 FOR Running REP.
- 8 FOR Merge FLD and XMS files to CHM file.
- 9 FOR Calculate blank values and blank uncertainty.
- 10 FOR Convert chm file to con file (ug/m<sup>3</sup>).

This is basically the same as before, except you simply want to copy the previous Field, lab and xms files.

## SVOC2 - The Sequel

### Background

We have to analyze for more than just the PAH species, so a second processing program has been written. This program follows the PAH analysis program sequence with a number of exceptions.

### Exceptions

The second SVOC program uses the same lab and field files as the regular program and thus these need to be finished at the same time.

The option exists in this program to define which compounds will be imported from the regular samples and which from diluted ones. This must be the same for all compounds in a project, although some adjustments can be made if necessary. In any case, all compounds must be imported the first pass through and then a sub-group can be imported from a second (called diluted) on file.

Everything is case sensitive, especially the compound names.

### Steps

1. Tell the Data Processing Manager which projects need this so the OALProj database

and the other necessary files can be updated.

2. Update the compound list file. This file is project-specific and it is located in the H:\db\_calib\svoc\ directory in the general form xx2cmpd.dbf, where xx is the project code. The template is nf2cmpd.dbf. This needs to be filled out in the following format:

**Field\_name** This is the mnemonic that will become the field name. Each compound must be followed by its associated uncertainty, just as in the example.

**Field\_type** Leave alone

**Field\_len** Leave alone

**Field\_dec** Leave alone

**Compound** For the compound only (not the uncert.), insert the compound name EXACTLY as it is in the HP GC/MS calibration file. If this is not spelled EXACTLY as it is in the calibration file on the HP GC/MS nothing will work. Do not put in anything for the uncertainties.

**Type** Put in "c" for compounds, nothing for uncertainties. EVERY compound in the list MUST have a "c" in this field.

**Dil** Put in "d" for compounds that will be imported from diluted files, nothing otherwise.

3. Update the Lab database. There are five new fields in the lab database for the second SVOC files. These are:

**F2\_name** Mass spec file name for primary analysis

**F2\_proc** Process status for above (1= ready to import, 2= done)

**Dil\_f2\_nam** MS file name for diluted run

**Dil\_f2\_pro** Process status for above (1= ready to import, 2= done)

**Date2** Analysis date for second compound list.

This should follow the conventions used in the normal data processing for PAH species.

4. Do genbatch and follow the instructions. When you select a project you will be prompted to select either SVOC or Additional SVOC compounds. Selecting the latter (option 7) will take you to the SVOC2 programs. First use the "N" option to build new files and then continue by importing the mass spec data and continuing the processing. This will create XM2 (the raw mass spec data), the CH2 file, and the CN2 (ng/m3) file.

Calibration Plot (Int Stds)    Filename: PAH    Correlation Coeff: 0.999  
1,7-dimethylphenanthrene    Compound: 45 of 83    Standard Deviation: 0.028  
(Area of Sample/Area of Standard) vs (Amount(sample)/Amount(standard)) (Lin/Lin)  
[  $y = 0.800000 \text{ E}8 \text{ x}^2 + 1.269278 \text{ E}8 \text{ x} + -4.631979 \text{ E}-3$  ]

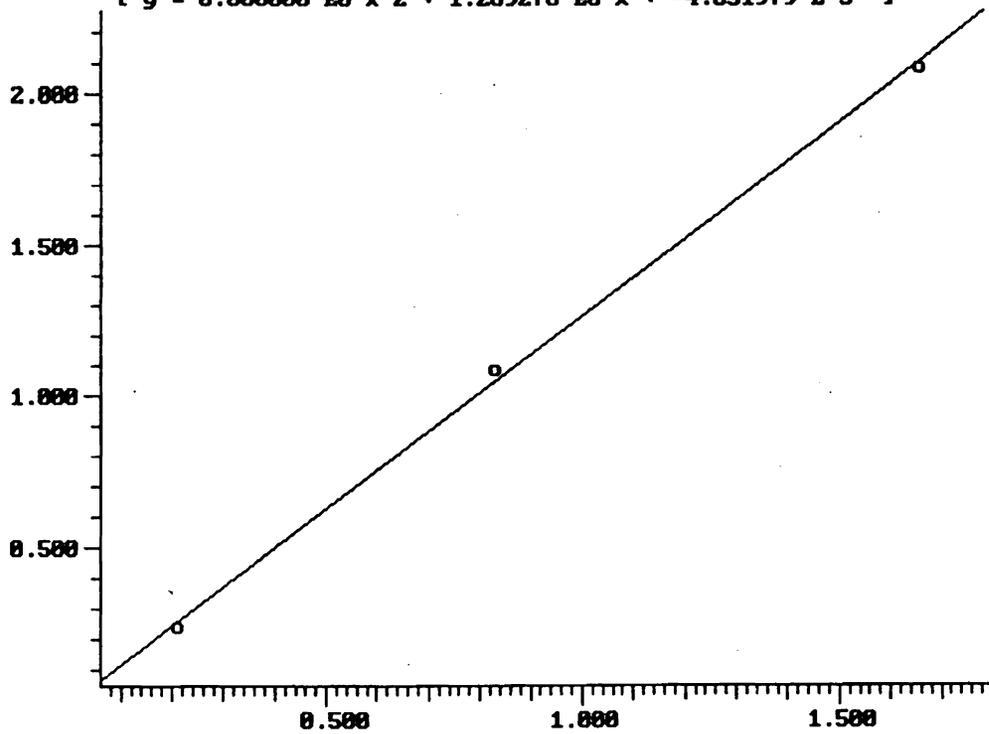


Figure 1

Calibration Plot (Int Stds)    Filename: PAH            Correlation Coeff: 0.999  
C-dimethylphenanthrene    Compound: 44 of 83    Standard Deviation: 0.028  
(Area of Sample/Area of Standard) vs (Amount(sample)/Amount(standard)) [Lin/Lin]  
[  $y = 0.000000 \text{ E}0 \text{ x}^2 + 1.269930 \text{ E}0 \text{ x} + -4.550553 \text{ E}-3$  ]

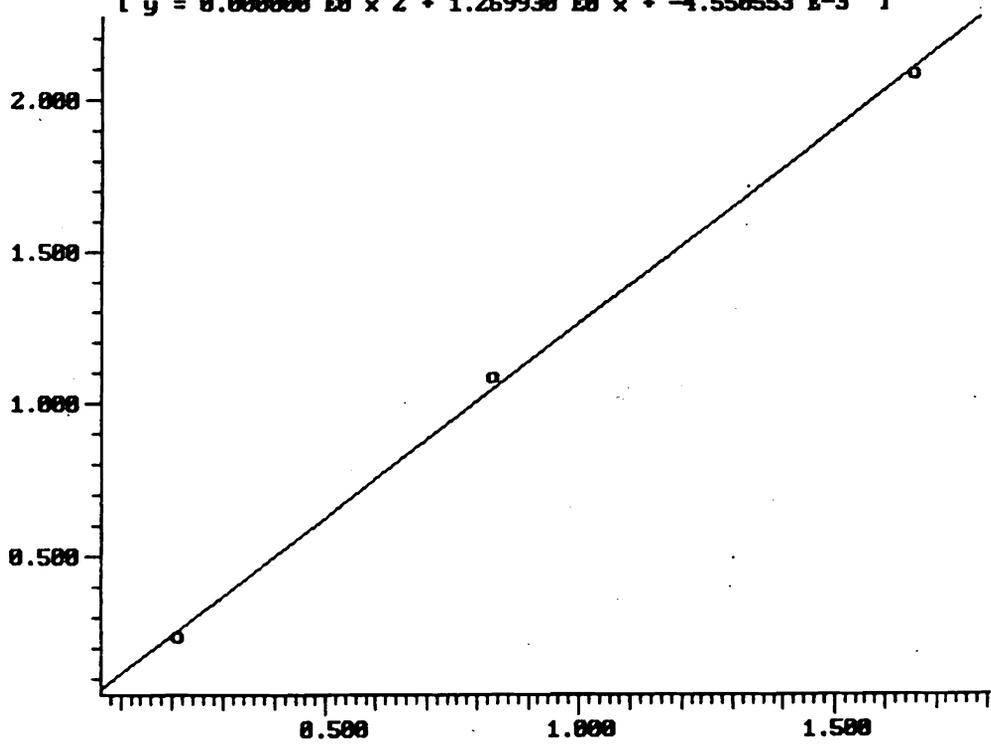


Figure 2

Calibration Plot (Int Stds)    Filename: PAH    Correlation Coeff: 0.999  
B-dimethylphenanthrene    Compound: 43 of 83    Standard Deviation: 0.026  
(Area of Sample/Area of Standard) vs (Amount(sample)/Amount(standard)) [Lin/Lin]  
[  $y = 0.000000 \text{ E}0 \text{ x}^2 + 1.272932 \text{ E}0 \text{ x} + -6.577547 \text{ E}-3$  ]

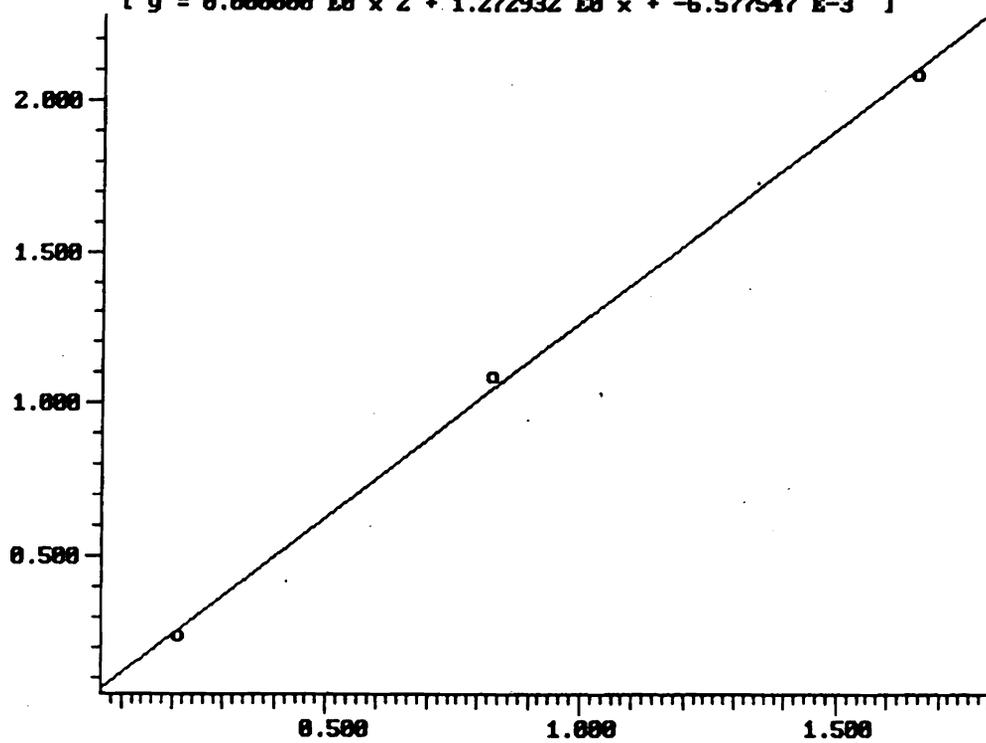


Figure 3

Calibration Plot (Int Stds)    Filename: PAH    Correlation Coeff: 0.999  
A-dimethylphenantherene    Compound: 42 of 83    Standard Deviation: 0.026  
(Area of Sample/Area of Standard) vs (Amount(sample)/Amount(standard)) (Lin/Lin)  
[  $y = 0.000000 \text{ E}0 \text{ x}^2 + 1.272866 \text{ E}0 \text{ x} + -6.185425 \text{ E}-3$  ]

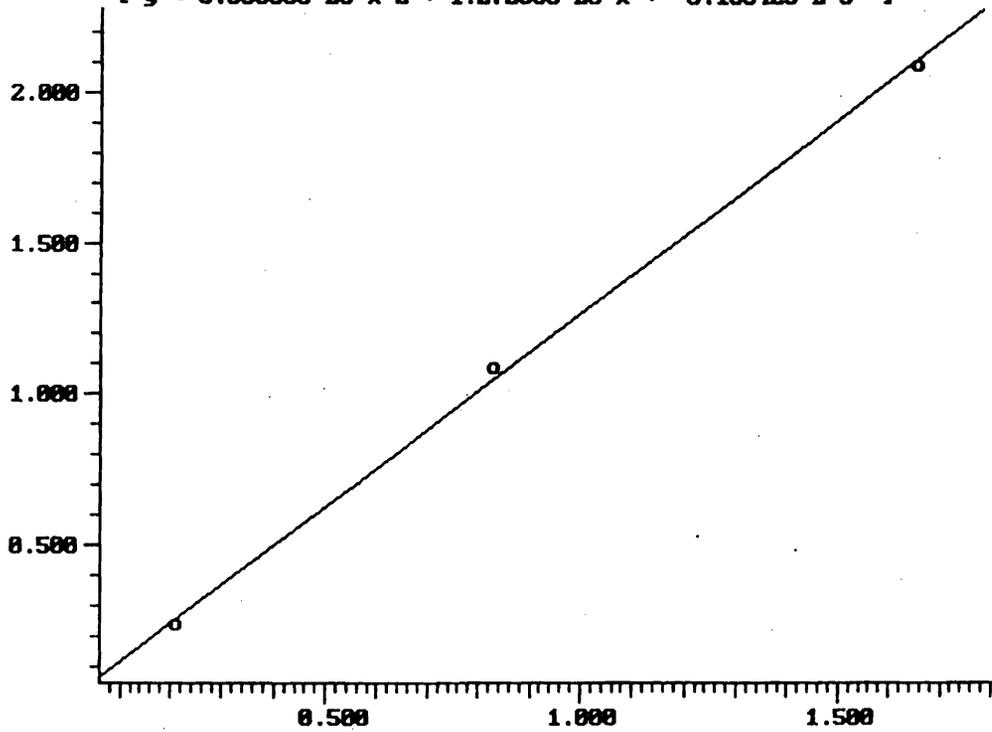


Figure 4

Calibration Plot (Int Stds)    Filename: PAH    Correlation Coeff: 0.997  
2,6+2,7-dimethylnaphthalene    Compound: 7 of 83    Standard Deviation: 0.053  
(Area of Sample/Area of Standard) vs (Amount(sample)/Amount(standard)) (LinLin)  
[  $y = 0.000000 \text{ E}0 \text{ x}^2 + 5.571070 \text{ E-1 x} + 0.442393 \text{ E-2}$  ]

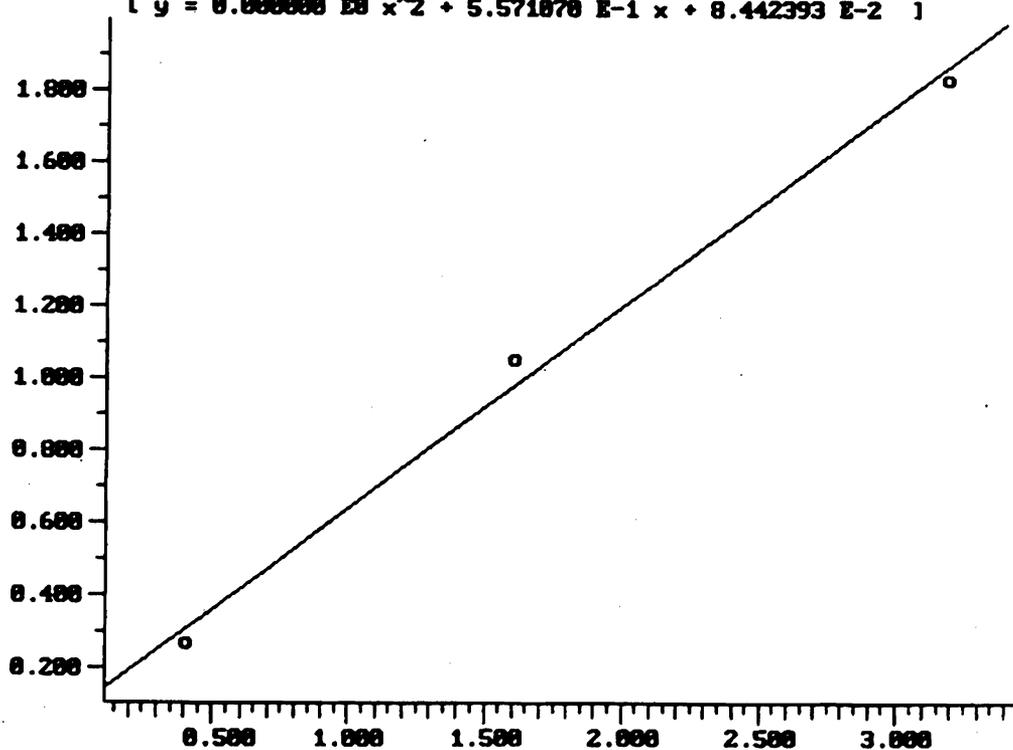


Figure 5

Calibration Plot (Int Stds)    Filename: PAH    Correlation Coeff: 0.996  
Biphenyl    Compound: 6 of 83    Standard Deviation: 0.022  
(Area of Sample/Area of Standard) vs (Amount(sample)/Amount(standard)) [Lin|Lin]  
[  $y = 0.000000 \text{ E}0 \text{ x}^2 + 0.520552 \text{ E-1 x} + 3.587838 \text{ E-2}$  ]

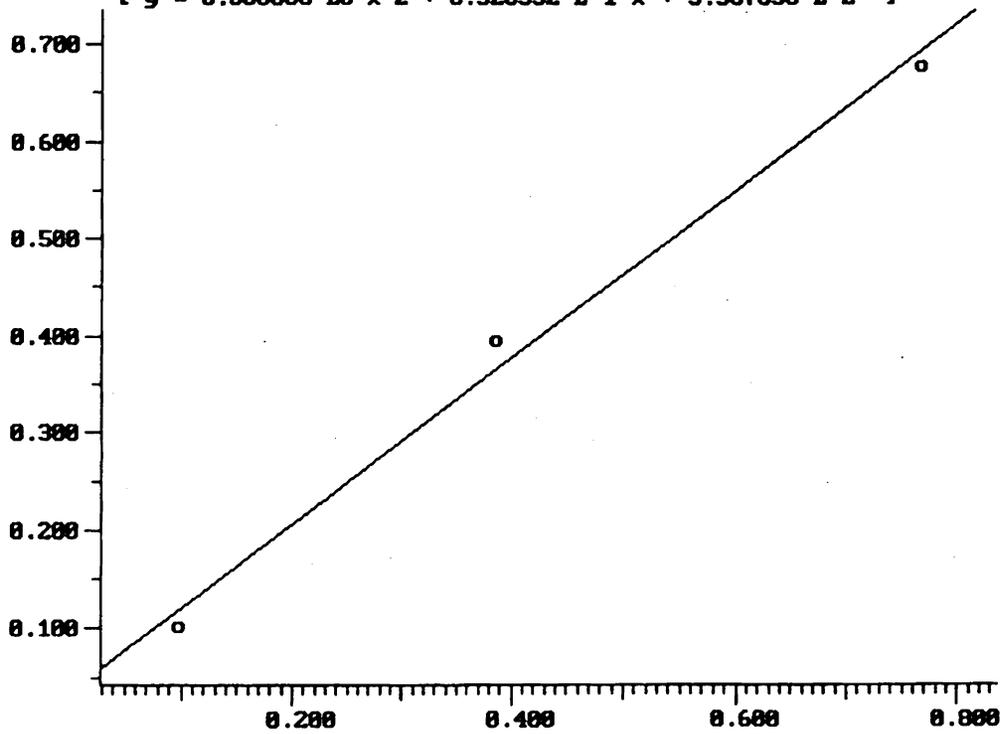


Figure 6



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## **FINAL REPORT**

### **Engine and Fuel Testing to Measure Exhaust Emissions from a Model 3306 Caterpillar Diesel Engine**

When Operating Over the ISO 8178 8-Mode Off-Road  
Emission Test Cycle and When Operating With  
Viscon Treated Ultra Low Sulfur Diesel Fuel  
Meeting California ULSD Specifications

**Prepared for  
Mr. Mike Porter  
Viscon California  
Formerly GTAT California  
Bakersfield, CA**

**January 4, 2008**

**Prepared by  
Olson-Ecologic Engine Testing Laboratories  
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## **Introduction and Background**

This emission testing project is a follow-on project for Viscon California, formerly GTAT (Las Palmas Oil and Dehydration Company) using the same diesel engine that has already completed standard controlled substance emission testing in accordance with the CARB protocol entitled *Verification Procedure, Warranty and In-Use Strategies to Control Emissions from Diesel Engines as Described in the Final Regulation Order, Title 13, California Code of Regulations, Sections 2700 through 2710.*

Original emission testing and 1000 hours of durability operation with the same test engine was completed in 2004 with Viscon fuel treatment resulting in significant reductions of NOx and Particulates compared to original baseline emission measurements before fuel treatment with Viscon.

Subsequent to conclusion of the above testing project, CARB has specified that the Viscon treated diesel fuel must additionally be tested as an alternative fuel with detailed exhaust emission measurements of uncontrolled substances, including toxic hydrocarbons and Carbonyls. The emission report of 2006 provides all of the CARB required data after operating the test engine and measuring emissions in exact accordance with CARB pre-approved testing and analysis protocols as described in the detailed report, but still CARB required another 1000 hours of durability testing (to make 3000 hours total of durability operation and emission testing) with Viscon treated fuel.

Accordingly, the same engine was operated and emission tested on commercially available ultra low sulfur diesel fuel that meets California ULSD specifications. Emissions were again measured after 1000 additional hours of operation with ULSD test fuel treated with Viscon. Emissions were again measured with the Viscon treated fuel for comparison to the baseline data.

## **Test Engine and Reference to Earlier Reports**

The test engine for this project is a Model 3306 six cylinder Caterpillar diesel engine rated at 265 HP. This is the same engine used in the prior emission testing to measure changes in exhaust emissions with the Viscon polymer additive admixed to the baseline no. 2 diesel fuel. The previous initial engine testing is described in an ETS (Olson Engineering, Inc.) summary report dated August 19, 2003 providing the data comparisons before durability testing and a second summary report (Olson-EcoLogic) dated November 26, 2004 covering the emission/fuel economy measurements after 1000 hours of engine durability operation with the Viscon treated fuel. The test work completed in 2006 is available in a report originally dated December 1, 2006 and revised August 8, 2007. All reports are available upon request.



## Test Fuel

Previous testing with the specified test engine was done in 2003 and 2004 using commercially available no. 2 CARB diesel fuel with sulfur limits of 500 ppm or less for the baseline and Viscon treated test fuel. This fuel met California fuel specifications that existed in the earlier time frame, but such fuel is no longer available in the California commercial market. Accordingly, all of the test work done in 2006 and again as described in this report was done with one batch of commercially available no. 2 ultra low sulfur diesel fuel. This test fuel has a sulfur content of less than 15 ppm and other composition differences compared to the test fuel used in earlier years with this engine. Fuel specifications are provided in the Appendix.

## Test Protocol

First, an additional 1000 hours of engine durability operation using a representative and repetitive load cycle was completed again by Olson-Ecologic, operating on ULSD test fuel treated with Viscon at 1 oz. per 20 gallons. The engine was then removed from the outside durability test cell and re-installed in the same test cell used previously for all collection of all official emission data. Then, as in all previous emission testing, for proper comparison of new data to data collected previously with the specified test engine the official ISO 8178 8-mode steady-state test protocol was used as required for certification of heavy-duty diesel engines in off-road applications. This operating test protocol involves the collection and analysis of emissions from a hot-start while operating the engine over 8-modes of fully warmed up operation at specific loads and speeds under stabilized conditions. The detailed testing cycle is as follows:

Mode No.	Weighting Factor %	MPH	Temp, °F
1	15	Rated	100
2	15	Rated	75
3	15	Rated	50
4	10	Rated	10
5	10	Max torque, rpm	100
6	10	Max torque	75
7	10	Max torque	50
8	15	Idle	0

The engine was operated and emissions were recorded every second of each mode for five minutes to stabilize emissions. The last two minutes of each mode were recorded second-by-second and averaged to provide the stabilized steady-state emission/fuel economy results.

All engine exhaust for all modes passed through the Horiba Olson-Ecologic dilution tunnel. The dilution tunnel was set to operate at a total constant flow of 70 cubic meters per minute, providing variable exhaust dilution for each mode as a function of actual



exhaust flow rates. Primary air was measured through the initial critical flow venturi (dilution air) and the total temperature corrected volume ( $V_{mix}$ ) of dilutant air plus exhaust was measured continuously at the second critical flow venturi. The calculated dilution ratio mode-by-mode is simply the temperature corrected composite total ( $V_{mix}$ ) divided by the exhaust flow ( $V_{mix}$  – dilution air). The measured dilution ratio is expected to be essentially a constant for the same mode for all tests, but the actual dilution ratios were used as measured for each individual test to allow for minor test-to-test variance.

Gaseous emissions for each test mode were measured every tenth of a second and then converted to recorded data for each second for each five minutes of mode operation. Only the last two minutes of each mode were averaged to provide the stabilized diluted mode data. Dilute data from each mode were then multiplied by the specific mode dilution ratio to obtain the raw modal data and then multiplied by the appropriate mode weighting factor before adding all eight modes together. Finally, the total sum of the weighted modal data was divided by the weighted horsepower and by the test time to provide the weighted grams per bhp-hr for direct data comparisons to the official emission standards for controlled gaseous emissions.

Particulate sampling involved initial calculation of exhaust mass flow per mode from an actual full 8-mode test. Secondary dilution ratios in the AVL Smart Sampler calculation were programmed for each mode and time of sampling was adjusted to properly compensate for specified modal weighting factors. Since the AVL PM Sampler draws its sample from the exhaust dilution tunnel for each mode the dilution ratio existing for each mode was included in the sample calculation to ultimately determine the total raw exhaust volume for proper calculation of the generated particulates in grams/bhp-hr. All particulate sampling resulted in the capture of measured and weighted particulates on a single filter media for each mode of each 8-mode test (eight complete particulate samples per test).

Exhaust sampling and subsequent gas chromatography analysis for uncontrolled toxic hydrocarbons (C2-C12) followed the detailed Standard Operating Procedure (S.O.P.) described in the Appendix. Modal sample collection was done for a total of 20 minutes. Modes 1, 2, 3 and 8 were collected for three minutes of the five minute mode, while modes 4, 5, 6 and 7 were collected for two minutes. Samples were drawn from each bag and analyzed by gas chromatography. The overall dilution ratio for the 8-modes (20 minutes of testing time) was used to correct and calculate the resulting G.C. data in grams/bhp-hr.  $((CONC^{ppb}/10^{-9})(V_{mix},ft^3)(Density,g/ft^3)/Work,bhp-hr)$ . Simultaneously samples were drawn and captured for Carbonyls for analysis at an off-site location (Desert Research Institute) as described in the Appended S.O.P. for this procedure.

### **Task Descriptions**

The specific tasks and order of operation were as follows:

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1. The engine was connected to the outside durability dynamometer and all required sensor systems were calibrated and verified. Normal maintenance intervals were followed with fuel and oil filters and oil changed every 250 hours.
2. The engine was operated with the Viscon treated fuel over a representative load cycle for 1,000 hours while monitoring RPM and load and all pertinent temperatures.
3. After accumulation of 1,000 durability hours the engine was re-installed in test cell no.1 for subsequent emission testing.
4. Instrumented and MAPPED engine, verified test cycle and all calibrations – initiated engine testing.
5. Conducted standard ISO 8178 8-mode emission tests with the Viscon treated fuel for PM (each mode) and gaseous emissions simultaneously capturing dilute exhaust gas samples from three of the tests for in house measurement of toxic hydrocarbons by gas chromatography and for off-site (Desert Research Institute) analysis of Carbonyls.
6. Conducted quality audit functions for all data, calculated and reported changes in all measured emissions caused by the Viscon treated fuel compared to the baseline fuel at a 95% confidence level. Prepared final report.

### **Test Facility, Equipment and Capability**

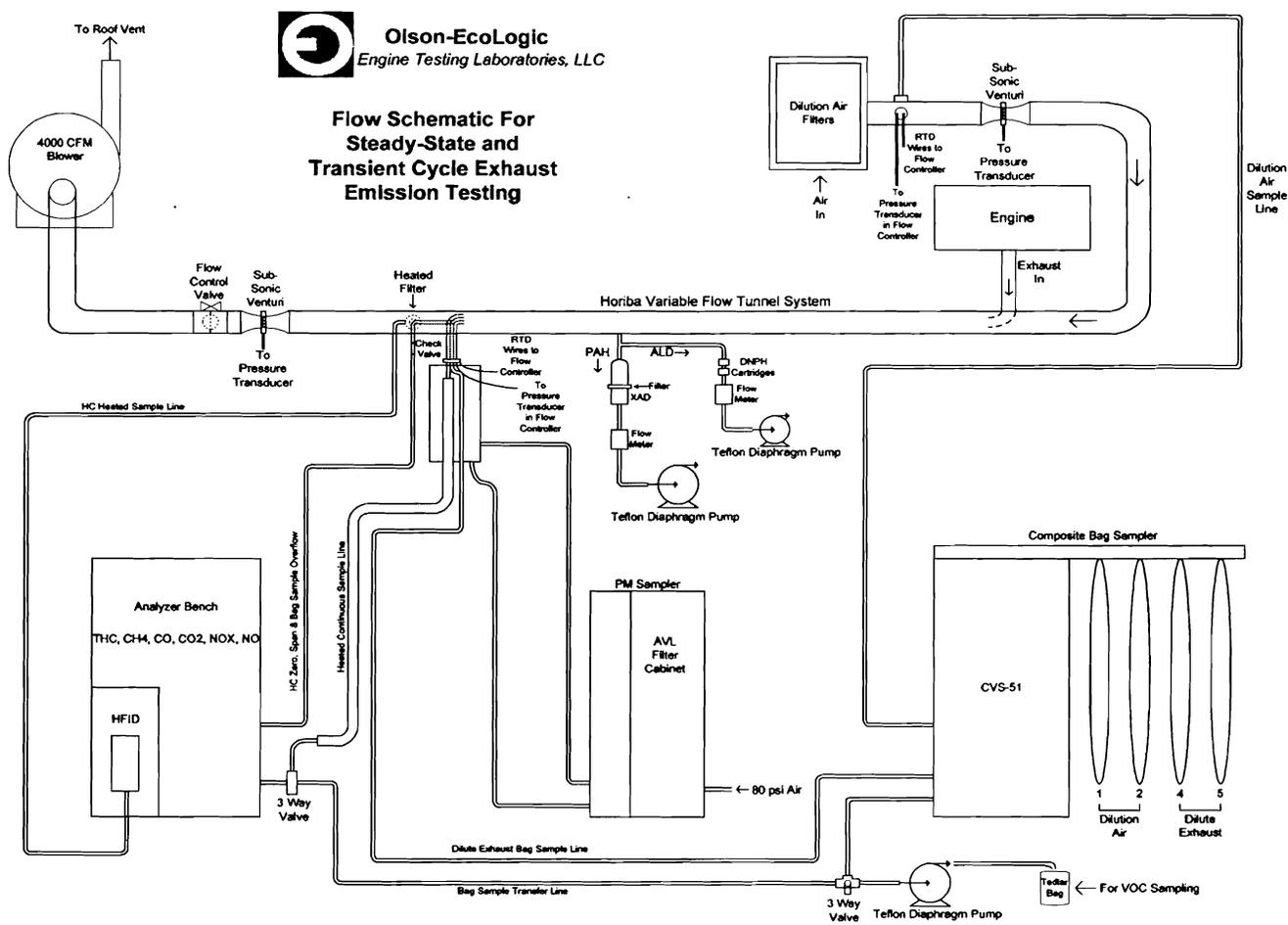
All testing discussed in this report was conducted at the Olson-EcoLogic heavy-duty engine test facility located in Fullerton, California. Engine durability accumulation was conducted over a representative load cycle in the outside test cell using the O-E 1000 HP SuperFlow dyno for engine loading. All initial engine operation and emission testing was conducted in the Olson-EcoLogic test cell 1. The test cell is equipped with a 450 horsepower full electric dynamometer, air conditioning and conventional sensors for continuous measurement of pressures, temperatures and air mass. State-of-the-art Horiba analyzers were used for dilute gas measurement of THC, CO, CO<sub>2</sub>, NO<sub>x</sub> and NO. An AVL Smart Particulate Sampler was used to capture particulates and fuel consumption was measured gravimetrically. The test cell operation is controlled by a fully integrated Labview based custom software program.

All corrected PM calculations are provided in detail for each test. The calculations to sum the results of multiple modes has been done in exact accordance with the ISO-8178 protocol for dilute sampling as provided in Section 15 of ISO 8178-1:2006 (E) and specifically sections 15.4 and 15.5. This test protocol originates in EPA CFR paragraph 89.112 (C) which further references paragraph 89.6. Finally, the PM and test cycle procedures for California refers back to use of the ISO 8178-1, Section 15 listed above and this is what was used to correct all of the PM data as described in report.



Effectively, this involves calculated removal of primary and secondary dilution and ultimate summation of the weighted mass PM flow rate for all eight modes divided by the 8-mode sum of weighted average power.

Heated sample lines were used to transport the exhaust gases to HC, NO<sub>x</sub> and NO analyzers. Calibrations with certified gases were routed to the analysers through the same sample lines used to sample the exhaust gases. In all cases the requirements of 40 CFR were followed. The particulate filters were conditioned for temperature and humidity before and after accumulation of particulates. Air mass to the engine was measured by a temperature corrected Sierra Air mass sensor as a secondary backup calibrated by the dilution tunnel venturi measurements of diluent air and total V<sub>mix</sub>. Engine intake air was controlled between 68 and 86 degrees F and fuel temperature was controlled to 100 degrees F +/- 10 degrees F as specified in CFR 40. A schematic of the complete exhaust gas sampling system is shown in the following figure.



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The quality management system of Olson-EcoLogic is ISO 9001:2000 registered. The Company is officially recognized by EPA and CARB as a capable emission test facility for the protocols used in this project. All test activity was under the direction and responsibility of Donel R. Olson. Mr. Olson is a registered professional mechanical engineer with 50 years of experience in the measurement and analysis of engine emissions. Over twenty engine emission test laboratories in the United States and Germany have been under his ownership and direction during the past 40 years. Details of the Olson-EcoLogic emission test facility capability can be viewed and obtained on the Olson-EcoLogic website ([www.ecologiclabs.com](http://www.ecologiclabs.com)).

### **Other**

Olson-EcoLogic Engine Testing Laboratories, LLC was acting as an independent contractor and not as agents or employees of the client in the conduct of work related to this project.

The Company does not guarantee the results of any test protocol, however, if repeat tests are not within acceptable variances Olson-EcoLogic on their own decision repeats the tests at no additional cost to the client.

### **Test Results**

All of the exhaust emission tests reported here are listed chronologically in Table 1 and Table 2. No new baseline data were acquired because this emission testing was done immediately after 1000 hours of additional engine durability testing while operating on the Viscon treated diesel fuel. Baseline data for the engine were accumulated in 2003 before any operation of the engine with Viscon treated fuel. The engine has actually undergone over 3,000 hours of CARB required durability and emission testing with Viscon treated fuel. The baseline data are available in a separate 2003 report.

Viscon additive at the normal concentration of 1oz. per 20 gallons was admixed in the diesel fuel used to operate the engine after measuring baseline emissions in 2003. Beginning with Viscon treated fuel test no. 8MLPV48 ten new official tests were completed to compare the average to the reference fuel baseline data. One of the tests (50) was not included in the ten test average because there were no PM values for mode 8. Table 2 includes the modal PM data and includes the averages, standard deviation and 95% confidence level for each mode of the PM sampling.

Concurrently with the above tests, as shown in Table 3, data were collected similarly for toxic compounds analysis including VOC's and Carbonyls. Triplicate sets of data were obtained with the Viscon treated reference fuel (test nos. 8MLPV52, 53 and 54). VOC data were collected a second time (test nos. 8MLPV58 through 61) because of significant data variance with these measurements. Table 3 provides the results of all



VOC and Carbonyls data for the triplicate baseline sets compared to the multiple sets of data obtained with the Viscon treated reference fuel.

### **Discussion of Results**

The measured effect of the Viscon additive on all controlled emissions must be compared to the baseline data without Viscon to see its effectiveness. The Viscon product as demonstrated for the Caterpillar Model 3306 engine under steady-state operating conditions will reduce exhaust particulates to satisfy level 1 of CARB criteria (25-50%). This result is consistent with earlier data accumulated on the same engine.

Additional data collection required by CARB involved two major expansions of the project.

- 1) Exhaust particulates were sampled for each mode of the 8-mode test protocol instead of a composite sample collection of all 8 modes on one filter media and
- 2) Integrated samples over the 8-modes of testing were collected and analyzed for toxic hydrocarbons (VOC's) and Carbonyls. These specific testing protocols are described in the appendix.

### **Particulate Sampling and Analysis**

The PM sampling for each mode resulted in measurement of PM for eight specific conditions for each 8-mode test. To be consistent with the ISO 8-mode test protocol, the specific sampling for each mode was weighted by dilution ratio selection and time of sampling capture to match the ISO test protocol specific weighting factors. Accordingly, modes 1, 2, 3 and 8 were weighted 15% each and modes 4, 5, 6 and 7 were weighted 10% each to total 100% for all eight modes.

The analysis of the PM data was accomplished by weighing the captured PM for each mode and dividing that weight by sampling time and the horsepower generated during the mode to provide the weighted PM result in grams/hp-hr. For the idle mode, (mode 8) this calculation was not possible because hp at idle is zero. Therefore, the idle PM data are reported in grams/hour only. However, the idle PM weight does go into the calculation of total weighted grams/bhp-hr. The PM values from all 7 of the other operating modes have been summed and tabulated in Tables 1 and 2 to show the total PM generated in grams/bhp-hr.

The VOC and Carbonyls comparisons shown in Table 3 for the same tests are somewhat of a mixed bag. For example, 1,3-butadiene initially is very low in concentration beyond the measurable data in the exhaust with the Viscon additive. As expected by the technical theory, the Carbonyls also show significant reductions with



the Viscon fuel treatment. Such data cannot be compared to the true engine baselines because CARB did not require the measurement of these compounds in 2003 when the true engine baseline was measured.

An anomaly involving the PM calculation from the weight of PM on individual filters from each mode compared to the composite sampling on a single mode being somewhat more with the multiple filters is commonly known. This difference is on the order of 20-30% higher weight for the multiple filters than for the single composite filter. This effect tends to be verified with duplicate data obtained at the end of the project capturing PM on a single composite filter and shown in Table 4.

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## Summary of Emissions

Comparison of the Averages of the Baseline Tests to the Viscon Tests  
Conducted at Olson-Ecologic Labs  
For Viscon California

### Baseline Tests Conducted October 2006

	grams/bhp-hr					
	<u>HC</u>	<u>CO</u>	<u>NOx</u>	<u>CO2</u>	<u>FUEL</u>	<u>PM</u>
9-Test Average	1.38	1.69	5.79	561.26	178.51	0.265

### Viscon Tests Conducted December 2007

	grams/bhp-hr					
	<u>HC</u>	<u>CO</u>	<u>NOx</u>	<u>CO2</u>	<u>FUEL</u>	<u>PM</u>
10-Test Average	1.43	1.62	5.89	552.58	175.80	0.197

### % Change

Baseline Tests vs. Viscon Tests	3.6%	-4.1%	1.7%	-1.5%	-1.5%	-25.7%
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**Table 1**  
**Data Summary**  
**Caterpillar 3306 Diesel Engine Tested with ULSD fuel with the Viscon Additive Treatment**  
**Testing Conducted by Olson-Ecologic Engine Testing Laboratories**  
**For Viscon California**  
**December 2007**

DATE	FUEL	TEST NO.	WHT (G/G)	WHT (PPM)						
12/10/2007	Viscon	8MLPV50	130.52	1.43	1.66	5.94	5.41	174.21	547.48	0.199

Test data not used in calculations (no PM data for mode 8)

12/10/2007	Viscon	8MLPV50	130.52	1.43	1.66	5.94	5.41	174.21	547.48	0.199
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**Table 2a**  
**PM Data Summary Table**  
**Caterpillar 3306 Diesel Engine Tested with ULSD Fuel**  
**Testing Conducted by Olson-Ecologic Engine Testing Laboratories**  
**For Viscon California**  
**Testing Conducted October 2006**

FUEL	TEST NO.	GRAMS PER BRP HR							WTL AVG. Total PM	GRAMS/HR Idle (PM Mode 8)	GRAMS/HR Mode 8 Nox
		PM Mode 1	PM Mode 2	PM Mode 3	PM Mode 4	PM Mode 5	PM Mode 6	PM Mode 7			
ULSD	8MLP14	0.129	0.183	0.232	3.094	0.342	0.237	0.234	0.289	20.54	27.31
ULSD	8MLP15	0.122	0.145	0.251	2.106	0.373	0.261	0.248	0.250	2.06	28.49
ULSD	8MLP16	0.108	0.127	0.251	2.209	0.355	0.261	0.241	0.240	2.06	17.11
ULSD	8MLP17	0.122	0.143	0.263	2.221	0.427	0.318	0.292	0.284	14.39	18.36
ULSD	8MLP18	0.121	0.145	0.254	2.430	0.392	0.281	0.314	0.297	30.85	25.37
ULSD	8MLP19	0.127	0.135	0.207	2.211	0.410	0.265	0.226	0.276	26.72	23.95
ULSD	8MLP20	0.118	0.153	0.241	2.322	0.352	0.236	0.241	0.273	26.70	17.85
ULSD	8MLP21	0.112	0.141	0.228	2.108	0.348	0.241	0.243	0.248	12.34	27.76
ULSD	8MLP22	0.102	0.161	0.210	1.917	0.312	0.208	0.124	0.231	18.51	26.47
<b>AVERAGES (BASELINE)</b>		<b>0.118</b>	<b>0.148</b>	<b>0.237</b>	<b>2.291</b>	<b>0.368</b>	<b>0.256</b>	<b>0.240</b>	<b>0.265</b>	<b>17.128</b>	<b>23.630</b>

**Table 2b**  
**PM Data Summary Table**  
**Caterpillar 3306 Diesel Engine Tested with ULSD fuel with the Viscon Additive Treatment**  
**Testing Conducted by Olson-Ecologic Engine Testing Laboratories**  
**For Viscon California**  
**December 2007**

DATE	FUEL	TEST NO.	WGT/ACC	PM10	PM10/ACC	PM2.5	PM2.5/ACC	PM2.5/PM10	PM2.5/ACC	PM2.5/ACC	WGT/ACC	PM10/ACC
12/10/2007	Viscon	<b>8MLPV48</b>	131.80	0.104	0.106	0.158	1.595	0.337	0.210	0.058	0.200	14.38
12/10/2007	Viscon	<b>8MLPV49</b>	130.69	0.109	0.103	0.169	1.697	0.361	0.216	0.058	0.197	4.11
12/10/2007	Viscon	<b>8MLPV51</b>	130.09	0.111	0.116	0.164	1.665	0.355	0.223	0.058	0.211	14.38
12/10/2007	Viscon	<b>8MLPV52</b>	131.89	0.112	0.119	0.181	1.372	0.391	0.191	0.030	0.196	4.65
12/10/2007	Viscon	<b>8MLPV53</b>	131.48	0.094	0.104	0.156	1.132	0.383	0.226	0.062	0.194	12.32
12/11/2007	Viscon	<b>8MLPV54</b>	130.57	0.104	0.101	0.153	1.449	0.348	0.171	0.079	0.196	15.95
12/11/2007	Viscon	<b>8MLPV55</b>	130.80	0.108	0.114	0.165	1.492	0.345	0.201	0.055	0.193	6.47
12/12/2007	Viscon	<b>8MLPV56</b>	130.51	0.109	0.104	0.138	1.286	0.333	0.191	0.034	0.192	18.47
12/12/2007	Viscon	<b>8MLPV57</b>	131.15	0.105	0.105	0.141	1.339	0.385	0.184	0.051	0.190	10.26
12/20/2007	Viscon	<b>8MLPV58</b>	131.54	0.091	0.088	0.136	1.241	0.323	0.196	0.058	0.201	15.14
		<b>10-Test Average</b>	<b>131.05</b>	<b>0.105</b>	<b>0.106</b>	<b>0.156</b>	<b>1.427</b>	<b>0.356</b>	<b>0.201</b>	<b>0.054</b>	<b>0.197</b>	<b>11.613</b>
		<b>Standard Deviation</b>	<b>0.609</b>	<b>0.007</b>	<b>0.009</b>	<b>0.015</b>	<b>0.187</b>	<b>0.024</b>	<b>0.018</b>	<b>0.014</b>	<b>0.006</b>	<b>5.023</b>
		<b>95% Confidence Level</b>	<b>0.378</b>	<b>0.004</b>	<b>0.005</b>	<b>0.009</b>	<b>0.116</b>	<b>0.015</b>	<b>0.011</b>	<b>0.009</b>	<b>0.004</b>	<b>3.114</b>
<b>Test data not used in calculations (no PM data for mode 8)</b>												
12/10/2007	Viscon	<b>8MLPV50</b>	130.52	0.102	0.097	0.137	1.385	0.357	0.211	0.095	0.199	0.00

**Table 3a**  
**G.C. Comparison Summary**  
**Caterpillar 3306 Diesel Engine**  
**Testing Conducted by Olson-Ecologic Engine Testing Laboratories**  
**For Viscon California**  
**December 2007**  
*(Units in g/bhp-hr)*

		1,3-butadiene	benzene	toluene	ethylbenzene	m&p-xylene	o-xylene
<b>10/10/2006</b>	<b>Baseline Avg.</b>	<b>0.000252</b>	<b>0.009026</b>	<b>0.004228</b>	<b>0.001811</b>	<b>0.003898</b>	<b>0.002311</b>
12/21/2007	8MLPV58	0.004201	0.009855	0.005123	0.002436	0.003837	0.002282
12/21/2007	8MLPV59	0.005098	0.009093	0.005008	0.002532	0.004109	0.002894
12/21/2007	8MLPV60	0.003878	0.008764	0.004917	0.002426	0.004064	0.002525
12/21/2007	8MLPV61	0.004816	0.008920	0.004927	0.001301	0.003972	0.002523
<b>4-Test Viscon Average</b>		<b>0.004498</b>	<b>0.009158</b>	<b>0.004994</b>	<b>0.002174</b>	<b>0.003996</b>	<b>0.002556</b>
<b>Standard Deviation</b>		<b>0.000558</b>	<b>0.000484</b>	<b>0.000095</b>	<b>0.000584</b>	<b>0.000120</b>	<b>0.000253</b>
<b>95% Confidence Level</b>		<b>0.000547</b>	<b>0.000474</b>	<b>0.000093</b>	<b>0.000572</b>	<b>0.000118</b>	<b>0.000248</b>

Difference: 168.2%      8.2%      8.2%      20.3%      22%

**Table 3b**

**Carbonyls**

G.C. Comparison Summary

Percent change comparing ULSF to ULSF w/ Viscon - (Units in g/bhp-hr)

Caterpillar 3306 Diesel Engine

Testing Conducted by Olson-Ecologic Engine Testing Laboratories

For Viscon California

December 2007

Analyzed via High Performance Liquid Chromatography by Desert Research Institute

	<u>10/10/2006</u>	<u>12/11/2007</u>	<u>12/11/2007</u>	<u>12/11/2007</u>		
	<b>Baseline Avg.</b>	<b>8MLPV52</b>	<b>8MLPV53</b>	<b>8MLPV54</b>	<b>Avg.</b>	<b>Change</b>
<i>formaldehyde</i>	<b>0.027257</b>	0.032759	0.026156	0.031159	<b>0.030025</b>	<b>10%</b>
<i>acetaldehyde</i>	<b>0.007385</b>	0.007214	0.006248	0.007383	<b>0.006948</b>	<b>-6%</b>
<i>acetone</i>	<b>0.001867</b>	0.000364	0.001660	0.000632	<b>0.000886</b>	<b>-53%</b>
<i>acrolein</i>	<b>0.001025</b>	0.000178	0.001332	0.000507	<b>0.000672</b>	<b>-34%</b>
<i>propionaldehyde</i>	<b>0.001332</b>	0.001030	0.001104	0.001136	<b>0.001090</b>	<b>-18%</b>
<i>crotonaldehyde</i>	<b>0.000997</b>	0.000000	0.000894	0.000101	<b>0.000332</b>	<b>-67%</b>
<i>methyl ethyl ketone</i>	<b>0.000462</b>	0.000028	0.000241	0.000047	<b>0.000105</b>	<b>-77%</b>
<i>methacrolein</i>	<b>0.000447</b>	0.000045	0.000000	0.000064	<b>0.000037</b>	<b>-92%</b>
<i>butyraldehyde</i>	<b>0.004759</b>	0.000519	0.000511	0.000645	<b>0.000558</b>	<b>-88%</b>
<i>benzaldehyde</i>	<b>0.000633</b>	0.000350	0.000751	0.000366	<b>0.000489</b>	<b>-23%</b>
<i>glyoxal</i>	<b>0.000322</b>	0.000000	0.000000	0.000000	<b>0.000000</b>	<b>-100%</b>
<i>valeraldehyde</i>	<b>0.000617</b>	0.000000	0.000000	0.000000	<b>0.000000</b>	<b>-100%</b>
<i>m-tolualdehyde</i>	<b>0.000016</b>	0.000170	0.000340	0.000158	<b>0.000223</b>	<b>1260%</b>
<i>hexanaldehyde</i>	<b>0.000316</b>	0.000420	0.000316	0.000448	<b>0.000395</b>	<b>25%</b>

**Table 3c****PAH's**

G.C. Comparison Summary

Percent change comparing ULSF to ULSF w/ Viscon - (Units in g/bhp-hr)

Caterpillar 3306 Diesel Engine

Testing Conducted by Olson-Ecologic Engine Testing Laboratories

For Viscon California

October 2006

Analyzed via High Performance Liquid Chromatography by Desert Research Institute

	<i>8MLP17</i>	<i>ULSF</i> <i>8MLP18</i>	<i>8MLP19</i>	<i>Average</i>	<i>8MLP39</i>	<i>ULSF w/ Viscon</i> <i>8MLP40</i>	<i>8MLP41</i>	<i>Average</i>	<i>Change</i>
<i>PAH's</i> (Analyzed via Gas Chromatography/Mass Spectrometry by Desert Research Institute)									
naphthalene	0.0000125192	0.0000071081	0.0000119718	0.0000105330	0.0000051731	0.0000046456	0.0000039993	0.0000046060	<b>-56%</b>
2-methylnaphthalene	0.0000112961	0.0000084828	0.0000095142	0.0000097644	0.0000024317	0.0000027416	0.0000025569	0.0000025767	<b>-74%</b>
acenaphthylene	0.0000002048	0.0000001814	0.0000001930	0.0000001930	0.0000001683	0.0000001606	0.0000001573	0.0000001621	<b>-16%</b>
acenaphthene	0.0000005852	0.0000006578	0.0000007354	0.0000006595	0.0000000615	0.0000000706	0.0000000610	0.0000000644	<b>-90%</b>
fluorine	0.0000005526	0.0000005425	0.0000005102	0.0000005351	0.0000001376	0.0000001311	0.0000001238	0.0000001308	<b>-76%</b>
phenanthrene	0.0000013504	0.0000013821	0.0000013007	0.0000013444	0.0000004623	0.0000005479	0.0000004993	0.0000005032	<b>-63%</b>
anthracene	0.0000001898	0.0000001834	0.0000001699	0.0000001810	0.0000000647	0.0000000816	0.0000000780	0.0000000748	<b>-59%</b>
fluoranthene	0.0000000719	0.0000000720	0.0000000579	0.0000000673	0.0000000559	0.0000000599	0.0000000350	0.0000000503	<b>-25%</b>
pyrene	0.0000002207	0.0000002166	0.0000002438	0.0000002270	0.0000001584	0.0000001207	0.0000000982	0.0000001258	<b>-45%</b>
benz(a)anthracene	0.0000000074	0.0000000084	0.0000000070	0.0000000076	0.0000000025	0.0000000015	0.0000000006	0.0000000015	<b>-80%</b>
chrysene-triphenylene	0.0000000108	0.0000000105	0.0000000141	0.0000000118	0.0000000073	0.0000000045	0.0000000042	0.0000000054	<b>-55%</b>
benzo(b+j)fluoranthene	0.0000000016	0.0000000006	0.0000000006	0.0000000010	0.0000000000	0.0000000000	0.0000000010	0.0000000003	<b>-67%</b>
benzo(k)fluoranthene	0.0000000005	0.0000000002	0.0000000003	0.0000000003	0.0000000002	0.0000000000	0.0000000003	0.0000000002	<b>-50%</b>
BeP	0.0000000013	0.0000000006	0.0000000006	0.0000000009	0.0000000005	0.0000000005	0.0000000006	0.0000000005	<b>-37%</b>
BaP	0.0000000002	0.0000000000	0.0000000010	0.0000000004	0.0000000000	0.0000000000	0.0000000000	0.0000000000	<b>-100%</b>
perylene	0.0000000006	0.0000000000	0.0000000002	0.0000000003	0.0000000000	0.0000000000	0.0000000000	0.0000000000	<b>-100%</b>
indeno[123-cd]pyrene	0.0000000004	0.0000000000	0.0000000000	0.0000000001	0.0000000002	0.0000000000	0.0000000000	0.0000000001	<b>-50%</b>
dibenzo(ah+ac)anthracene	0.0000000002	0.0000000000	0.0000000000	0.0000000001	0.0000000000	0.0000000000	0.0000000007	0.0000000002	<b>299%</b>
benzo(ghi)perylene	0.0000000000	0.0000000000	0.0000000000	0.0000000000	0.0000000000	0.0000000000	0.0000000004	0.0000000001	



**Table 4**  
**Emission Data Obtained at the End of the Viscon Project**  
**Using a Single Composite PM Filter**

Test Using Capture of PM on a Single Composite Filter With Viscon Additive	Average HP	HC	CO	NOx	CO <sub>2</sub>	PM	Prel
8MLPV62	131.7	1.42	1.53	6.02	5.41	0.159	174.70
8MLPV63	131.1	1.44	1.58	5.70	5.32	0.164	175.26

QA/QC

Please complete in chronological order.

PER TEST				
1	8MLPV48	12/11/07	9:17	HC,NO,Nox, CO,CO2
2	8MLPV49	12/11/07	11:54	HC,NO,Nox, CO,CO2
3	8MLPV52	12/11/07	10:18	HC,NO,Nox, CO,CO2
4	8MLPV53	12/11/07	11:37	HC,NO,Nox, CO,CO2
5	8MLPV54	12/11/07	13:56	HC,NO,Nox, CO,CO2
6	8MLPV55	12/11/07	10:10	HC,NO,Nox, CO,CO2
7	8MLPV56	12/11/07	12:11	HC,NO,Nox, CO,CO2
8	8MLPV57	12/11/07	13:43	HC,NO,Nox, CO,CO2
9	8MLPV58	12/20/07	14:23	HC,NO,Nox, CO,CO2
10				
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### Proposed Test Matrix

1	1	Viscon Additive		
1	2	Viscon Additive		
2	3	Viscon Additive	Carbonyls	Sample**
2	4	Viscon Additive	Carbonyls	Sample
2	5	Viscon Additive	Carbonyls	Sample
3	6	Viscon Additive		
3	7	Viscon Additive		
3	8	Viscon Additive		
4	9	Viscon Additive	VOC's	Sample**
4	10	Viscon Additive	VOC's	Sample
4	11	Viscon Additive	VOC's	Sample
4	12	Viscon Additive	VOC's	Sample

\*\* Tunnel blank will be run before first test is conducted

**TOXICS**

**Format for Reporting Emissions Test Data for Each Emissions Test Run for the Post-Durability Test**

Viscon	<b>9 (8MLPV58)</b>					
	VOC's					
	Mode 1	12/20/2007	2:25 PM	2:28 PM	0:03	N/A
	Mode 2	12/20/2007	2:31 PM	2:34 PM	0:03	N/A
	Mode 3	12/20/2007	2:36 PM	2:39 PM	0:03	N/A
	Mode 4	12/20/2007	2:43 PM	2:45 PM	0:02	N/A
	Mode 5	12/20/2007	2:49 PM	2:51 PM	0:02	N/A
	Mode 6	12/20/2007	2:54 PM	2:56 PM	0:02	N/A
	Mode 7	12/20/2007	3:00 PM	3:02 PM	0:02	N/A
Mode 8	12/20/2007	3:04 PM	3:07 PM	0:03	N/A	
Viscon	<b>10 (8MLPV59)</b>					
	VOC's					
	Mode 1	12/20/2007	3:54 PM	3:57 PM	0:03	N/A
	Mode 2	12/20/2007	4:00 PM	4:03 PM	0:03	N/A
	Mode 3	12/20/2007	4:05 PM	4:08 PM	0:03	N/A
	Mode 4	12/20/2007	4:12 PM	4:14 PM	0:02	N/A
	Mode 5	12/20/2007	4:17 PM	4:19 PM	0:02	N/A
	Mode 6	12/20/2007	4:22 PM	4:24 PM	0:02	N/A
	Mode 7	12/20/2007	4:28 PM	4:30 PM	0:02	N/A
Mode 8	12/20/2007	4:32 PM	4:35 PM	0:03	N/A	
Viscon	<b>11 (8MLPV60)</b>					
	VOC's					
	Mode 1	12/20/2007	6:33 PM	6:36 PM	0:03	N/A
	Mode 2	12/20/2007	6:38 PM	6:41 PM	0:03	N/A
	Mode 3	12/20/2007	6:44 PM	6:47 PM	0:03	N/A
	Mode 4	12/20/2007	6:50 PM	6:52 PM	0:02	N/A
	Mode 5	12/20/2007	6:56 PM	6:58 PM	0:02	N/A
	Mode 6	12/20/2007	7:01 PM	7:03 PM	0:02	N/A
	Mode 7	12/20/2007	7:07 PM	7:09 PM	0:02	N/A
Mode 8	12/20/2007	7:11 PM	7:14 PM	0:03	N/A	
Viscon	<b>12 (8MLPV61)</b>					
	VOC's					
	Mode 1	12/20/2007	8:38 PM	8:41 PM	0:03	N/A
	Mode 2	12/20/2007	8:44 PM	8:47 PM	0:03	N/A
	Mode 3	12/20/2007	8:49 PM	8:52 PM	0:03	N/A
	Mode 4	12/20/2007	8:56 PM	8:58 PM	0:02	N/A
	Mode 5	12/20/2007	9:01 PM	9:03 PM	0:02	N/A
	Mode 6	12/20/2007	9:07 PM	9:09 PM	0:02	N/A
	Mode 7	12/20/2007	9:12 PM	9:14 PM	0:02	N/A
Mode 8	12/20/2007	9:16 PM	9:19 PM	0:03	N/A	

**TOXICS**

**Format for Reporting Emissions Test Data for Each Emissions Test Run for the Post-Durability Test**

Viscon	<b>3 (8MLPV52)</b>					
	Carbonyls					
	Mode 1	12/11/2007	10:20 AM	10:23 AM	0:03	1 L/min
	Mode 2	12/11/2007	10:25 AM	10:28 AM	0:03	1 L/min
	Mode 3	12/11/2007	10:31 AM	10:34 AM	0:03	1 L/min
	Mode 4	12/11/2007	10:37 AM	10:39 AM	0:02	1 L/min
	Mode 5	12/11/2007	10:43 AM	10:45 AM	0:02	1 L/min
	Mode 6	12/11/2007	10:48 AM	10:50 AM	0:02	1 L/min
	Mode 7	12/11/2007	10:54 AM	10:56 AM	0:02	1 L/min
Mode 8	12/11/2007	10:59 AM	11:02 AM	0:03	1 L/min	
Viscon	<b>4 (8MLPV53)</b>					
	Carbonyls					
	Mode 1	12/11/2007	11:39 AM	11:42 AM	0:03	1 L/min
	Mode 2	12/11/2007	11:45 AM	11:48 AM	0:03	1 L/min
	Mode 3	12/11/2007	11:51 AM	11:54 AM	0:03	1 L/min
	Mode 4	12/11/2007	11:57 AM	11:59 AM	0:02	1 L/min
	Mode 5	12/11/2007	12:02 PM	12:04 PM	0:02	1 L/min
	Mode 6	12/11/2007	12:07 PM	12:09 PM	0:02	1 L/min
	Mode 7	12/11/2007	12:12 PM	12:14 PM	0:02	1 L/min
Mode 8	12/11/2007	12:17 PM	12:20 PM	0:03	1 L/min	
Viscon	<b>5 (8MLPV54)</b>					
	Carbonyls					
	Mode 1	12/11/2007	1:58 PM	2:01 PM	0:03	1 L/min
	Mode 2	12/11/2007	2:03 PM	2:06 PM	0:03	1 L/min
	Mode 3	12/11/2007	2:09 PM	2:12 PM	0:03	1 L/min
	Mode 4	12/11/2007	2:16 PM	2:18 PM	0:02	1 L/min
	Mode 5	12/11/2007	2:22 PM	2:24 PM	0:02	1 L/min
	Mode 6	12/11/2007	2:28 PM	2:30 PM	0:02	1 L/min
	Mode 7	12/11/2007	2:33 PM	2:35 PM	0:02	1 L/min
Mode 8	12/11/2007	2:37 PM	2:40 PM	0:03	1 L/min	

**PM**  
**Format for Reporting Emissions Test Data for the Post-Durability Test**

<b>8MLPV48</b>	<b>12/10/07</b>					
Mode 1		9:17	9:22	0:05	0:03	1.088
Mode 2		9:23	9:28	0:05	0:03	0.868
Mode 3		9:28	9:33	0:05	0:03	0.682
Mode 4		9:33	9:38	0:05	0:02	0.441
Mode 5		9:39	9:44	0:05	0:02	0.859
Mode 6		9:44	9:49	0:05	0:02	0.671
Mode 7		9:50	9:55	0:05	0:02	0.567
Mode 8		9:55	10:00	0:05	0:03	0.137
<b>8MLPV49</b>	<b>12/10/07</b>					
Mode 1		11:54	11:59	0:05	0:03	1.088
Mode 2		12:00	12:05	0:05	0:03	0.868
Mode 3		12:05	12:10	0:05	0:03	0.682
Mode 4		12:11	12:16	0:05	0:02	0.441
Mode 5		12:16	12:21	0:05	0:02	0.859
Mode 6		12:22	12:27	0:05	0:02	0.671
Mode 7		12:27	12:32	0:05	0:02	0.567
Mode 8		12:32	12:37	0:05	0:03	0.137
<b>8MLPV52</b>	<b>12/11/07</b>					
Mode 1		10:18	10:23	0:05	0:03	1.088
Mode 2		10:23	10:28	0:05	0:03	0.868
Mode 3		10:29	10:34	0:05	0:03	0.682
Mode 4		10:34	10:39	0:05	0:02	0.441
Mode 5		10:40	10:45	0:05	0:02	0.859
Mode 6		10:45	10:50	0:05	0:02	0.671
Mode 7		10:51	10:56	0:05	0:02	0.567
Mode 8		10:57	11:02	0:05	0:03	0.137
<b>8MLPV53</b>	<b>12/11/07</b>					
Mode 1		11:37	11:42	0:05	0:03	1.088
Mode 2		11:43	11:48	0:05	0:03	0.868
Mode 3		11:49	11:54	0:05	0:03	0.682
Mode 4		11:54	11:59	0:05	0:02	0.441
Mode 5		11:59	12:04	0:05	0:02	0.859
Mode 6		12:04	12:09	0:05	0:02	0.671
Mode 7		12:09	12:14	0:05	0:02	0.567
Mode 8		12:15	12:20	0:05	0:03	0.137
<b>8MLPV54</b>	<b>12/11/07</b>					
Mode 1		13:56	14:01	0:05	0:03	1.088
Mode 2		14:56	15:01	0:05	0:03	0.868
Mode 3		15:56	16:01	0:05	0:03	0.682
Mode 4		16:56	17:01	0:05	0:02	0.441
Mode 5		17:56	18:01	0:05	0:02	0.859
Mode 6		18:56	19:01	0:05	0:02	0.671
Mode 7		19:56	20:01	0:05	0:02	0.567
Mode 8		20:56	21:01	0:05	0:03	0.137

8MPLV35	11/12/07					
Mode 1		10:10	10:15	0:05	0:03	1.088
Mode 2		10:16	10:21	0:05	0:03	0.868
Mode 3		10:21	10:26	0:05	0:03	0.682
Mode 4		10:26	10:31	0:05	0:02	0.441
Mode 5		10:32	10:37	0:05	0:02	0.859
Mode 6		10:38	10:43	0:05	0:02	0.671
Mode 7		10:43	10:48	0:05	0:02	0.567
Mode 8		10:49	10:54	0:05	0:03	0.137
8MPLV36	12/12/07					
Mode 1		12:11	12:16	0:05	0:03	1.088
Mode 2		12:17	12:22	0:05	0:03	0.868
Mode 3		12:22	12:27	0:05	0:03	0.682
Mode 4		12:27	12:32	0:05	0:02	0.441
Mode 5		12:33	12:38	0:05	0:02	0.859
Mode 6		12:39	12:44	0:05	0:02	0.671
Mode 7		12:44	12:49	0:05	0:02	0.567
Mode 8		12:50	12:55	0:05	0:03	0.137
8MPLV57	12/12/07					
Mode 1		13:43	13:48	0:05	0:03	1.088
Mode 2		14:43	14:48	0:05	0:03	0.868
Mode 3		15:43	15:48	0:05	0:03	0.682
Mode 4		16:43	16:48	0:05	0:02	0.441
Mode 5		17:43	17:48	0:05	0:02	0.859
Mode 6		18:43	18:48	0:05	0:02	0.671
Mode 7		19:43	19:48	0:05	0:02	0.567
Mode 8		20:43	20:48	0:05	0:03	0.137
8MPLV58	12/20/07					
Mode 1		14:23	14:28	0:05	0:03	1.088
Mode 2		15:23	15:28	0:05	0:03	0.868
Mode 3		16:23	16:28	0:05	0:03	0.682
Mode 4		17:23	17:28	0:05	0:02	0.441
Mode 5		18:23	18:28	0:05	0:02	0.859
Mode 6		19:23	19:28	0:05	0:02	0.671
Mode 7		20:23	20:28	0:05	0:02	0.567
Mode 8		21:23	21:28	0:05	0:03	0.137



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**CAT. 3306**  
**12-10-07 TEST# 8MLPV48**

**8-MODE STEADY-STATE**  
**W/ARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	f/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.0	567.6	237.8	6.22	178.93	739.30	679.06	425.48	679	489.9	895.0	24.4	76.9	30.20	0.0291599	653.37	84.9	600.1	1.82	423.9	59.5	18.1
2	2200.0	469.2	196.5	5.96	141.44	690.35	635.62	435.49	563.5	434.9	642.8	24.2	77.0	30.20	0.0279666	609.75	87.7	561.4	1.85	433.6	59.9	17.9
3	2200.0	315.1	132.0	5.33	158.09	579.13	522.53	581.07	405	359.1	742.8	23.9	76.4	30.20	0.0252237	511.21	89.9	461.3	2.62	578.5	61.4	18.0
4	2200.0	62.0	26.0	3.31	253.41	225.70	188.41	851.76	167	270.0	500.7	23.8	75.3	30.20	0.0160992	199.20	91.4	166.3	4.58	847.2	63.7	18.6
5	1400.0	699.4	186.4	8.02	847.79	923.51	860.02	311.80	491	256.3	964.9	24.1	74.6	30.20	0.0374844	815.59	96.4	759.5	3.41	308.4	51.7	19.3
6	1400.0	577.8	154.0	7.78	576.69	938.90	879.17	313.13	407	224.7	924.8	23.9	75.4	30.20	0.036288	828.78	98.1	776.1	3.96	309.2	52.6	18.6
7	1400.0	382.5	101.9	6.98	197.28	889.02	841.00	497.48	273.5	180.9	772.1	24.0	76.0	30.20	0.0326355	784.99	97.4	742.6	5.15	492.3	53.8	18.4
8	742.6	1.1	0.1	2.98	222.24	248.01	231.59	789.88	20.25	78.6	414.4	23.4	75.8	30.20	0.0145246	218.70	95.2	204.2	18.96	770.9	28.9	18.0

WT. FAC %	Mode No	GRAMS/HOUR							NO2/NOX RATIO	
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2		NMHC
15.00	1	278.36	235.29	1411.54	1291.308	40740	1442737	128447.04	277.3	0.08
15.00	2	246.81	161.13	1141.18	1048.483	33810	1246927	106601.52	245.8	0.08
15.00	3	263.18	143.92	784.81	692.1438	24300	990968	76301.75	262.0	0.10
10.00	4	251.55	150.43	194.27	165.4887	10020	634530	30867.85	250.2	0.17
10.00	5	113.87	622.35	983.81	913.9778	29460	818094	92448.47	112.6	0.07
10.00	6	98.01	362.82	856.63	801.175	24420	699668	76861.49	96.8	0.06
10.00	7	116.75	93.07	608.37	577.3467	16410	520963	51726.30	115.6	0.05
15.00	8	31.40	17.76	28.71	27.49248	1215	85146	3740.90	30.7	0.07

WTD AVG BHP = **131.80**      KW = **98.28**

Mode	HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	W.F.	W.F.	MODE =												
											1	2	3	4	5	6	7	8					
WTD AVG GM/H =	180.98	206.58	766.19	705	23041	832192	72454	179.88	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
AVG GM/BHPH =	1.37	1.57	5.81	5.35	174.82		549.74	1.36	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
AVG GM/KWH =	1.84	2.10	7.80	7.17	234.44		737.22	1.83	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10

WT AVG NO2/NOX RATIO = 0.083863578

GM/BHPH= 0.104    0.106    0.158    1.595    0.337    0.210    0.058

WTD AVG GM/H = 26.352

WEIGHTED AVG GM/BHPH = 0.200

WTD AVG GM/KWH = 0.268

8-Mode Test Results

**CAT. 3306**  
**12-10-07 TEST# 8MLPV49**  
**8-MODE STEADY-STATE**  
**W/ARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh degF	ABSHUM GR/LB	Air In degF	Baro P InHg	f/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.0	551.9	231.2	8.05	170.01	708.13	852.33	427.34	660	483.1	884.9	20.6	77.8	30.20	0.0283996	620.44	101.4	571.6	1.6	425.8	59.9	14.9
2	2200.0	469.5	196.7	5.87	141.67	685.42	634.02	430.05	568	439.3	842.5	20.4	78.7	30.20	0.0275966	600.17	101.7	555.2	1.9	428.1	60.0	14.2
3	2200.0	315.3	132.1	5.28	161.32	574.44	519.57	585.06	406.5	380.0	742.7	20.0	78.9	30.20	0.0249818	502.63	101.4	454.6	2.6	582.5	61.3	13.9
4	2200.0	62.0	26.0	3.24	265.93	233.41	192.97	851.13	167	272.0	499.9	19.8	78.3	30.20	0.0157903	204.11	100.6	168.7	4.8	846.4	63.6	14.0
5	1400.0	693.8	184.9	7.97	845.92	911.53	855.64	316.39	489	254.7	964.8	19.8	78.0	30.20	0.0372863	797.05	103.3	748.2	3.7	312.7	51.8	14.2
6	1400.0	578.0	154.1	7.78	600.92	930.07	880.63	326.86	407.5	222.2	928.7	19.5	78.7	30.20	0.0363133	812.84	104.3	769.6	4.3	322.6	52.5	13.6
7	1400.0	382.1	101.9	6.96	206.00	889.41	840.13	496.33	273.5	178.7	775.5	19.2	78.9	30.20	0.0325688	776.76	103.6	733.7	5.5	490.8	54.1	13.3
8	731.1	0.9	0.1	3.03	268.13	275.61	236.48	856.11	20.25	76.7	351.7	19.0	78.7	30.20	0.0148152	240.56	100.5	208.1	21.7	834.4	28.4	13.3

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	279.23	223.29	1338.74	1228.579	39600	1438095	124833.95	278.21	0.08
15.00	2	249.06	164.91	1147.79	1059.236	34080	1272606	107448.03	247.96	0.08
15.00	3	268.61	148.87	782.05	691.5652	24390	1003496	78563.34	267.43	0.10
10.00	4	256.37	161.00	203.02	171.3416	10020	646381	30836.05	254.97	0.17
10.00	5	115.71	621.86	962.61	901.8584	29340	818445	92061.41	114.37	0.06
10.00	6	102.36	378.27	840.62	795.115	24450	699638	76918.97	101.03	0.05
10.00	7	116.73	97.38	603.27	571.6519	16410	521650	51719.58	115.44	0.06
15.00	8	33.36	21.00	30.95	27.51381	1215	83447	3729.61	32.54	0.13

WTD AVG BHP = **130.69**      KW = **97.46**

WTD AVG GM/H =	AVG GM/BPH =	AVG GM/KWH =	W.F.																				
			HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC													
183.66	1.41	1.88	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	
			MODE =	1	2	3	4	5	6	7	8												
			PARTIC. WT, MG =	0.98	0.63	0.54	0.46	1.36	0.53	0.08	0.02												
			GP (g/Sec.) =	1.087	0.868	0.882	0.441	0.859	0.671	0.587	0.137												
			Sample Time (Sec.) =	180.00	180.00	180.00	120.00	120.00	120.00	120.00	180.00												
			mSEP (kg) =	0.196	0.156	0.123	0.053	0.103	0.081	0.068	0.025												
			Dilution Tunnel Total Flow (scmm) =	65.11	65.21	65.32	65.37	65.18	65.21	65.24	65.31												
			qmedf (kg/Hr.) =	5051.6	5058.9	5067.4	5071.2	5056.3	5058.8	5061.1	5067.0												
			qmPT (g/Hr.) =	25.2503	20.2368	22.2904	44.081	66.7114	33.2984	5.95069	4.10953												

WT AVG NO2/NOX RATIO = **0.091919937**

GM/BPH = **0.109**    **0.103**    **0.169**    **1.697**    **0.361**    **0.216**    **0.058**

WTD AVG GM/H = **25.787**

WEIGHTED AVG GM/BPH = **0.197**

WTD AVG GM/KWH = **0.265**

8-Mode Test Results

**CAT. 3306**  
**12-10-07 TEST# 8MLPV50**  
**8-MODE STEADY-STATE**  
**W/ARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	faCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.0	550.0	230.4	6.00	161.98	717.18	660.45	405.61	657	484.8	884.7	19.3	75.9	30.20	0.0281469	626.45	98.0	576.9	1.48	404.15	59.5	14.8
2	2200.0	469.6	196.7	5.82	144.43	688.20	633.21	429.90	568	426.9	849.1	20.5	79.6	30.20	0.027338	602.80	98.9	554.6	1.76	428.14	59.8	13.9
3	2200.0	314.8	131.9	5.21	171.29	584.36	522.07	595.17	407	355.1	754.1	20.2	82.0	30.20	0.0246565	511.49	98.6	457.0	2.64	592.54	61.1	12.7
4	2200.0	62.1	26.0	3.13	265.12	233.78	188.36	898.53	168	267.3	511.9	20.0	81.6	30.20	0.0152883	204.55	97.9	164.8	4.67	893.86	63.5	12.7
5	1400.0	691.9	184.4	7.82	866.24	955.40	875.85	316.99	489	248.3	977.5	20.4	81.8	30.20	0.036633	836.66	96.4	766.8	3.86	313.33	51.4	12.9
6	1399.9	578.3	154.1	7.61	631.81	975.20	897.35	318.50	408	220.9	944.7	20.0	82.0	30.20	0.0355615	853.14	96.3	785.0	4.34	314.15	52.3	12.5
7	1400.0	382.7	102.0	6.78	208.97	933.02	857.71	518.83	247	178.8	790.1	19.8	81.8	30.20	0.0317783	815.86	95.8	750.0	5.33	513.50	53.7	12.5
8	735.5	1.0	0.1	2.49	278.34	280.88	221.39	792.43	19.75	76.6	366.5	19.5	81.2	30.20	0.0122925	245.27	95.5	193.5	19.83	772.80	28.6	12.5

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	266.28	213.73	1357.94	1245.351	39420	1443793	124316.92	265.30	0.08
15.00	2	251.39	169.76	1164.03	1068.51	34080	1284352	107433.06	250.37	0.08
15.00	3	277.30	160.41	786.94	705.5693	24420	1017685	76613.30	276.08	0.11
10.00	4	281.36	168.87	211.51	174.3485	10080	671296	30938.91	279.94	0.19
10.00	5	118.08	648.58	1029.14	941.1925	29340	832593	92011.96	116.72	0.08
10.00	6	102.05	406.93	902.73	829.7013	24480	714829	76970.43	100.67	0.08
10.00	7	113.03	91.51	586.95	541.7205	14820	482494	46678.73	111.88	0.08
15.00	8	36.38	25.68	37.18	30.2257	1185	97853	3617.20	35.50	0.21

WTD AVG BHP = 130.52 KW = 97.33

Mode	HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	W.F.													
									MODE =	GP (g/Sec.)=	Sample Time (Sec.)=	mSEP (kg)=	Dilution Tunnel Total Flow (scmm)=	qmedf (kg/Hr.)=	qmPT (g/Hr.)=	GM/BHPH=	0.15	0.15	0.15	0.10	0.10	0.10
1	266.28	213.73	1357.94	1245.351	39420	1443793	124316.92	265.30	0.15	1.01	180	0.196	65.06	5047.6	26.06	0.102	0.15	0.15	0.15	0.10	0.10	0.15
2	251.39	169.76	1164.03	1068.51	34080	1284352	107433.06	250.37	0.15	0.88	180	0.156	65.21	5059.4	22.02	0.097	0.15	0.15	0.15	0.10	0.10	0.15
3	277.30	160.41	786.94	705.5693	24420	1017685	76613.30	276.08	0.15	1.087	180	0.156	65.31	5066.7	20.22	0.137	0.15	0.15	0.15	0.10	0.10	0.15
4	281.36	168.87	211.51	174.3485	10080	671296	30938.91	279.94	0.10	1.087	180	0.156	65.31	5066.7	20.22	0.137	0.15	0.15	0.15	0.10	0.10	0.15
5	118.08	648.58	1029.14	941.1925	29340	832593	92011.96	116.72	0.10	1.087	180	0.156	65.31	5066.7	20.22	0.137	0.15	0.15	0.15	0.10	0.10	0.15
6	102.05	406.93	902.73	829.7013	24480	714829	76970.43	100.67	0.10	1.087	180	0.156	65.31	5066.7	20.22	0.137	0.15	0.15	0.15	0.10	0.10	0.15
7	113.03	91.51	586.95	541.7205	14820	482494	46678.73	111.88	0.10	1.087	180	0.156	65.31	5066.7	20.22	0.137	0.15	0.15	0.15	0.10	0.10	0.15
8	36.38	25.68	37.18	30.2257	1185	97853	3617.20	35.50	0.15	1.087	180	0.156	65.31	5066.7	20.22	0.137	0.15	0.15	0.15	0.10	0.10	0.15

WT AVG NO2/NOX RATIO = 0.115355213

WTD AVG GM/H = 26.1352

WEIGHTED AVG GM/BHPH = 0.199

WTD AVG GM/KWH = 0.267

**CAT. 3306**  
**12-10-07 TEST# 8MLPV51**  
**8-MODE STEADY-STATE**  
**W/ARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	l/aCAL FACTOR	KNOx PPM	Fuelln degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidity %
1	2200.0	541.7	226.9	6.06	188.99	735.42	668.96	425.75	654	477.2	904.5	32.0	102.9	30.20	0.0284459	661.41	102.9	601.6	1.56	424.18	59.1	10.4
2	2200.0	469.7	196.7	5.84	160.96	696.00	643.03	442.75	569	437.4	861.3	35.4	103.7	30.20	0.0274577	631.07	103.7	583.0	1.78	440.97	59.2	11.3
3	2200.0	315.0	131.9	5.22	173.60	580.32	526.50	595.65	407	361.3	757.3	37.6	103.4	30.20	0.0247302	528.88	103.4	479.8	2.71	592.93	61.0	12.1
4	2200.0	61.8	25.9	3.15	265.85	224.79	186.93	868.49	169	276.0	511.6	38.4	101.9	30.20	0.0153957	205.24	101.9	170.7	4.73	863.76	63.2	12.9
5	1400.0	888.8	183.7	7.87	847.85	931.31	860.98	302.15	526	256.4	977.4	43.2	103.6	30.20	0.0368308	860.13	103.6	795.2	3.54	298.61	51.0	13.7
6	1400.0	577.8	154.0	7.64	622.65	945.33	873.77	302.62	408	225.9	945.6	45.7	104.8	30.20	0.0356844	878.35	104.8	811.9	4.12	298.50	51.9	14.0
7	1400.0	382.3	101.9	6.78	222.36	895.63	829.80	503.62	276	183.9	790.1	44.4	103.3	30.20	0.0317537	829.54	103.3	768.6	5.29	498.33	53.5	14.2
8	725.0	10.2	1.4	2.88	283.52	327.26	276.98	778.55	22	77.1	364.2	40.6	101.2	30.20	0.0140805	300.40	101.2	254.3	20.51	758.04	26.9	13.9

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	275.21	245.55	1411.84	1279.497	39240	1425001	123665.63	274.2	0.09
15.00	2	258.21	188.89	1215.33	1120.446	34140	1283800	107572.80	257.2	0.08
15.00	3	276.67	162.08	811.23	738.6269	24420	1017181	78612.65	275.4	0.09
10.00	4	271.82	167.13	211.96	180.1131	10140	672373	31160.27	270.2	0.17
10.00	5	120.39	679.02	1131.69	1043.298	31560	893735	99024.22	119.0	0.08
10.00	6	96.62	399.59	926.06	854.7894	24480	714970	76999.14	95.3	0.08
10.00	7	122.70	108.89	667.39	620.4005	16560	541379	52160.19	121.4	0.07
15.00	8	34.70	25.40	44.21	38.39127	1320	95610	4052.73	33.8	0.15

WTD AVG BHP = **130.09** KW = **97.01**

WTD AVG GM/H =	HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	W.F. 0.15	W.F. 0.15	0.15	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.15	
																							MODE =
187.85	228.72	816.10	746	23142	855485	72720	186.68	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
1.44	1.76	6.27	5.74	177.89	558.99	1.43	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
1.94	2.36	8.41	7.69	238.56	749.62	1.92	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10

WT AVG NO2/NOX RATIO = 0.10124061

WTD AVG GM/H = **27.4694**

WEIGHTED AVG GM/BHPH = **0.211**

WTD AVG GM/KWH = **0.283**

8-Mode Test Results

**CAT. 3306**  
**12-11-07 TEST# 8MLPV52**  
**8-MODE STEADY-STATE**  
**WARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh degF	ABSHUM GR/LB	Air In degF	Baro P InHga	f/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.0	569.8	238.7	6.06	183.84	717.26	680.71	440.93	679.5	485.2	911.2	21.2	83.5	30.20	0.0284794	629.23	89.4	597.16	1.47	439.47	59.5	12.7
2	2200.0	469.9	196.8	5.83	142.02	684.17	651.63	439.69	568	434.3	860.6	21.0	84.3	30.20	0.0273869	600.00	92.0	571.47	1.81	437.88	59.6	12.3
3	2200.0	314.4	131.7	5.24	152.34	579.83	534.83	576.82	406	357.3	759.0	21.3	83.7	30.20	0.0247701	508.80	92.5	469.31	2.53	574.29	61.1	12.7
4	2200.0	62.4	26.1	3.19	237.67	235.54	190.90	823.42	166.75	272.9	511.7	20.9	83.0	30.20	0.0155064	206.48	92.5	167.34	4.44	818.98	63.5	12.7
5	1400.0	696.3	185.6	7.90	855.17	967.76	881.95	308.16	497	256.1	984.5	20.8	82.3	30.20	0.0389823	848.25	92.8	773.04	3.45	304.71	51.5	12.9
6	1400.0	578.1	154.1	7.70	598.51	987.78	907.06	319.03	407.75	226.1	948.1	20.6	82.9	30.20	0.0359733	865.32	93.7	794.61	4.00	315.03	52.1	12.5
7	1400.0	382.8	102.1	6.82	184.19	938.21	859.92	506.19	274.5	185.7	789.5	20.4	82.9	30.20	0.0319152	821.67	93.9	753.10	4.99	501.20	54.0	12.5
8	725.0	1.2	0.2	2.90	215.75	273.98	224.16	798.87	19	75.4	363.9	20.0	83.0	30.20	0.014131	239.73	93.8	196.13	18.77	780.11	27.6	12.2

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	295.78	247.81	1393.85	1317.889	40770	1478665	128468.16	294.8	0.05
15.00	2	256.65	166.63	1156.51	1099.113	34080	1282213	107421.38	255.6	0.05
15.00	3	266.81	141.64	777.18	719.1762	24380	1010797	76484.88	265.7	0.08
10.00	4	252.23	146.34	208.86	172.7211	10005	657143	30824.39	250.9	0.19
10.00	5	115.52	644.38	1050.07	954.7152	29820	838550	93554.71	114.2	0.09
10.00	6	100.95	380.68	904.19	829.3556	24465	706552	76967.42	99.7	0.08
10.00	7	122.01	89.24	654.01	601.4594	16470	534033	51906.73	120.8	0.08
15.00	8	30.64	16.63	30.36	25.50149	1140	82045	3506.30	29.9	0.18

WTD AVG BHP = **131.89**      KW = **98.35**

WTD AVG GM/H =	AVG GM/BHPH =	AVG GM/KWH =	W.F.							
			HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC
186.55	1.41	1.90	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.15
1.41	1.61	2.15	MODE = 1	MODE = 2	MODE = 3	MODE = 4	MODE = 5	MODE = 6	MODE = 7	MODE = 8
1.90	1.61	2.15	PARTIC. WT, MG = 1.04	0.72	0.58	0.37	1.48	0.47	0.04	0.02
			GP (g/Sec.) = 1.087	0.868	0.682	0.441	0.859	0.671	0.567	0.137
			Sample Time (Sec.) = 180	180	180	120	120	120	120	180
			mSEP (kg) = 0.196	0.156	0.123	0.053	0.103	0.081	0.068	0.025
			Dilution Tunnel Total Flow (scmm) = 65.10	65.24	65.28	65.37	65.20	65.25	65.25	65.33
			qmedf (kg/Hr.) = 5050.6	5061.6	5064.7	5071.2	5058.6	5061.9	5062.0	5068.5
			qmPT (g/Hr.) = 26.78	23.39	23.80	35.86	72.64	29.48	3.03	4.65

WT AVG NO2/NOX RATIO = 0.09803331      GM/BHPH = 0.112    0.119    0.181    1.372    0.391    0.191    0.030

WTD AVG GM/H = 25.8944

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WEIGHTED AVG GM/BHPH = 0.196

WTD AVG GM/KWH = 0.263

8-Mode Test Results

**CAT. 3306**  
**12-11-07 TEST# 8MLPV53**  
**8-MODE STEADY-STATE**  
**WARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	f/aCAL FACTOR	KNOx PPM	Fuelln degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.1	565.3	238.8	6.04	176.33	720.88	666.03	405.05	678.5	486.4	903.0	17.4	85.3	30.20	0.0283456	627.03	101.3	579.3	1.06	404.00	59.9	9.8
2	2200.0	469.5	196.7	5.87	147.18	895.97	640.05	441.88	571	436.4	857.1	17.1	85.4	30.20	0.0275808	604.97	100.4	556.4	0.11	441.77	59.8	9.7
3	2200.0	314.9	131.9	5.32	159.55	581.31	525.30	587.62	409.75	353.7	753.5	17.2	83.2	30.20	0.0251564	505.33	99.5	456.8	0.25	587.38	61.3	10.4
4	2200.0	62.2	26.1	3.23	245.48	230.67	188.09	849.94	169.75	274.6	509.3	15.9	80.9	30.20	0.0157431	199.96	98.5	183.1	1.13	848.81	63.4	10.4
5	1400.1	692.1	184.5	7.99	841.94	933.40	865.50	304.50	490	251.1	973.8	16.8	80.1	30.20	0.0373823	810.64	101.0	751.7	0.76	303.74	51.6	11.2
6	1400.0	577.9	154.0	7.80	603.41	941.69	883.56	301.04	406.75	224.1	939.9	17.0	80.3	30.20	0.036386	818.26	102.3	767.8	0.84	300.21	52.3	11.3
7	1400.0	382.3	101.9	6.98	182.01	895.43	837.52	487.15	275	179.2	782.4	17.0	80.1	30.20	0.0326417	778.10	101.5	727.8	1.01	486.14	53.6	11.3
8	725.1	1.5	0.2	3.33	189.57	285.70	231.43	768.10	20	74.9	356.0	16.9	80.0	30.20	0.0161197	248.22	99.4	201.1	4.48	763.61	27.5	11.3

WT. FAC %	Mode No	GRAMS/HOUR							NO2/NOX RATIO	
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2		
15.00	1	272.61	238.54	1393.58	1281.791	40710	1480491	128364.58	271.9	0.08
15.00	2	257.42	172.34	1163.79	1067.973	34260	1279471	107982.99	257.4	0.08
15.00	3	270.02	147.37	766.79	695.1916	24585	1004268	77182.07	269.9	0.10
10.00	4	261.01	151.53	202.78	168.7815	10185	658606	31361.55	260.7	0.18
10.00	5	111.29	618.53	978.39	905.144	29400	817751	92271.61	111.0	0.07
10.00	6	93.91	378.35	842.89	789.798	24405	696758	76802.31	93.6	-0.06
10.00	7	114.93	66.31	606.20	566.7054	16500	523216	52029.18	114.7	0.06
15.00	8	27.13	13.46	28.95	23.97403	1200	75823	3713.38	27.0	0.19

WTD AVG BHP = **131.48**      KW = **98.05**

WTD AVG GM/H	AVG GM/BPH	AVG GM/KWH	W.F.	MODE	PARTIC. WT, MG	GP (g/Sec.)	Sample Time (Sec.)	mSEP (kg)	Dilution Tunnel Total Flow (scmm)	qmedf (kg/Hr.)	qmPT (g/Hr.)	W.F.							
												0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
182.19	1.39	1.86	0.15	1	0.86	1.087	180	0.196	65.09	5050.0	22.30	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
			0.15	2	0.83	0.868	180	0.156	65.19	5057.1	20.39	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
			0.15	3	0.50	0.682	120	0.123	65.27	5064.0	20.58	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
			0.15	4	0.31	0.441	120	0.103	65.34	5068.8	29.50	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
			0.15	5	1.44	0.859	120	0.081	65.20	5059.9	34.81	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
			0.15	6	0.55	0.671	120	0.068	65.22	5062.0	6.32	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
			0.15	7	0.09	0.567	120	0.068	65.25	5062.1		0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10
			0.15	8	0.06	0.567	120	0.068	65.25	5062.1		0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.10

WT AVG NO2/NOX RATIO = 0.104786037

WTD AVG GM/H = **25.4685**

WEIGHTED AVG GM/BPH = **0.194**

WTD AVG GM/KWH = **0.260**

**CAT. 3306**  
**12-11-07 TEST# 8MLPV54**  
**8-MODE STEADY-STATE**  
**WARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh degF	ABSHUM GR/LB	Air In degF	Baro P In-Hga	f/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.0	551.3	231.0	8.09	173.83	738.75	873.81	423.20	862.75	473.7	899.8	17.5	82.5	30.20	0.0286018	640.93	97.4	586.0	0.21	422.98	59.8	10.8
2	2200.0	470.1	196.9	5.90	149.19	708.80	648.78	444.44	569.75	438.4	855.8	17.0	83.2	30.20	0.0277702	615.89	97.8	562.0	0.04	444.40	59.9	10.3
3	2200.0	315.0	132.0	5.25	163.54	590.12	531.42	606.77	408.75	380.3	753.9	17.0	83.2	30.20	0.0248537	512.80	97.1	461.8	0.27	606.50	61.3	10.3
4	2200.0	81.9	25.9	3.14	246.35	234.90	191.72	836.91	167.25	277.2	510.8	16.8	82.5	30.20	0.0152831	204.02	96.6	166.5	1.01	835.90	63.5	10.4
5	1399.9	689.8	183.9	7.81	867.88	931.56	862.06	316.16	487.25	253.7	975.7	17.2	82.1	30.20	0.0365908	809.80	98.6	749.4	0.63	315.53	51.7	10.7
6	1399.9	578.0	154.1	7.61	615.82	948.22	883.17	313.67	409.25	221.8	943.7	16.7	82.2	30.20	0.0355835	823.36	100.3	766.9	0.61	313.06	52.6	10.4
7	1400.0	382.3	101.9	6.78	191.81	909.84	848.79	529.01	274.75	182.5	785.5	16.8	82.4	30.20	0.0317343	790.20	100.0	737.2	0.69	528.31	53.9	10.4
8	725.0	0.8	0.1	2.70	209.74	297.55	244.27	928.39	19.75	76.4	360.5	16.5	81.9	30.20	0.0132694	258.27	98.5	212.0	4.66	923.73	27.8	10.4

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	275.86	227.33	1378.64	1255.804	39765	1433546	125364.14	275.5234018	0.09
15.00	2	257.18	173.53	1176.90	1071.661	34185	1271205	107743.11	257.1600457	0.09
15.00	3	281.61	152.56	785.92	710.3812	24525	1013694	78946.27	281.4869418	0.10
10.00	4	260.96	154.40	210.07	175.066	10035	668218	30879.69	260.6480667	0.18
10.00	5	117.49	648.11	993.73	917.8115	29235	830165	91880.30	117.2542028	0.07
10.00	6	100.75	397.59	873.31	812.3941	24555	716267	77227.99	100.5532874	0.07
10.00	7	128.38	93.47	633.26	592.9746	16485	537199	51927.71	128.2127571	0.07
15.00	8	39.45	17.92	36.24	30.74658	1185	90698	3619.71	39.25928648	0.18

WTD AVG BHP = 130.57 KW = 97.37

WTD AVG GM/H =	AVG GM/BHPH =	AVG GM/KWH =	W.F.																				
			HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC													
188.84	1.45	1.94	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	
			MODE =	1	2	3	4	5	6	7	8												
			PARTIC. WT, MG =	0.93	0.61	0.49	0.39	1.30	0.42	0.11	0.08												
			GP (g/Sec.) =	1.087	0.868	0.682	0.441	0.859	0.671	0.587	0.137												
			Sample Time (Sec.) =	180	180	180	120	120	120	120	180												
			mSEP (kg) =	0.196	0.156	0.123	0.053	0.103	0.081	0.068	0.025												
			Dilution Tunnel Total Flow (scmm) =	65.10	65.24	65.28	65.37	65.20	65.25	65.33													
			qmedf (kg/Hr.) =	5050.6	5061.6	5064.7	5071.2	5058.6	5081.9	5062.0	5068.5												
			qmPT (g/Hr.) =	24.04	19.80	20.25	37.55	64.02	26.28	8.08	15.95												

WT AVG NO2/NOX RATIO = 0.107174765

WTD AVG GM/H = 25.5993

WEIGHTED AVG GM/BHPH = 0.196

WTD AVG GM/KWH = 0.263



8-Mode Test Results

**CAT. 3306**  
**12-12-07 TEST# 8MLPV56**  
**8-MODE STEADY-STATE**  
**WARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	#aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.1	551.0	230.8	5.98	157.61	708.02	654.04	434.71	664.75	477.9	895.9	17.0	84.1	30.40	0.0280926	615.18	95.2	568.29	0.00	434.71	59.8	10.0
2	2199.9	469.6	196.7	5.79	130.06	688.69	638.07	442.46	569.75	426.5	851.5	16.9	84.8	30.40	0.0272035	598.31	95.5	554.33	0.00	442.46	60.0	9.8
3	2199.9	313.7	131.4	5.12	154.52	572.18	519.71	604.64	408.25	363.8	749.9	16.7	84.4	30.40	0.0242641	496.89	95.4	451.33	0.01	604.64	61.4	9.8
4	2200.0	61.4	25.7	3.03	238.88	222.95	184.36	841.34	168.75	276.7	501.4	16.5	81.0	30.40	0.0148028	193.53	95.0	160.03	0.63	840.71	63.7	10.8
5	1400.2	891.6	184.4	7.74	777.53	924.13	862.82	310.46	488.25	259.0	980.8	16.7	79.8	30.40	0.0362248	802.47	98.0	749.24	0.31	310.14	51.6	11.3
6	1400.2	578.7	154.3	7.57	560.54	939.66	883.33	323.66	407.25	222.3	937.7	17.0	80.9	30.40	0.0353911	816.57	100.5	767.63	0.31	323.35	52.4	11.1
7	1400.1	380.7	101.5	6.58	181.76	880.58	830.46	521.07	273.25	184.9	775.9	16.7	81.6	30.40	0.0308679	784.74	100.4	721.21	0.32	520.75	54.0	10.7
8	724.9	3.7	0.5	2.28	209.28	245.43	208.34	783.27	19.75	77.9	338.3	16.3	81.5	30.40	0.0112568	212.92	98.7	180.74	2.83	780.44	28.1	10.5

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	289.31	210.84	1351.98	1244.171	39885	1483094	125728.99	289.3	0.08
15.00	2	280.85	154.12	1164.80	1076.832	34185	1293856	107782.03	260.8	0.07
15.00	3	287.25	147.56	779.54	710.7381	24495	1036423	76840.81	287.2	0.09
10.00	4	273.40	156.03	207.67	175.4817	10125	695732	31124.32	273.2	0.17
10.00	5	116.80	588.00	996.99	928.7539	29295	839923	91967.92	116.7	0.07
10.00	6	104.03	362.14	866.99	813.855	24435	716542	76891.32	103.9	0.06
10.00	7	129.40	90.73	627.13	593.6506	16395	548800	51642.28	129.3	0.06
15.00	8	38.30	21.11	35.28	30.91351	1185	106700	3618.33	38.2	0.15

WTD AVG BHP = 130.51 KW = 97.32

	HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	W.F.	W.F.	0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.15
									0.15	MODE =	1	2	3	4	5	6	7	8
WTD AVG GM/H =	193.72	199.73	769.59	711	22988	865111	72255	193.65	0.15	PARTIC. WT, MG =	0.98	0.63	0.44	0.35	1.25	0.47	0.07	0.09
AVG GM/BHPH =	1.48	1.53	5.90	5.44	176.13		553.63	1.48	0.10	GP (g/Sec.)=	1.087	0.868	0.682	0.441	0.859	0.671	0.567	0.137
AVG GM/KWH =	1.99	2.05	7.91	7.30	236.20		742.43	1.99	0.10	Sample Time (Sec.)=	180	180	180	120	120	120	180	180
									0.10	mSEP (kg)=	0.196	0.158	0.123	0.053	0.103	0.081	0.102	0.025
									0.10	Dilution Tunnel Total Flow (scmm)=	65.07	65.21	65.36	65.39	65.22	65.22	65.29	65.23
									0.15	qmedf (kg/Hr.)=	5047.9	5058.9	5070.5	5073.1	5060.2	5060.0	5065.1	5080.2
										qmPT (g/Hr.)=	25.2318	20.5284	18.1738	33.0731	61.3619	29.4729	3.4244	18.4677

WT AVG NO2/NOX RATIO = 0.09451517

GM/BHPH= 0.109 0.104 0.138 1.286 0.333 0.191 0.034

WTD AVG GM/H = 25.0935

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WEIGHTED AVG GM/BHPH = 0.192

WTD AVG GM/KWH = 0.258

**CAT. 3306**  
**12-12-07 TEST# 8MLPV57**  
**8-MODE STEADY-STATE**  
**W/ARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P In-Hga	#aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm	Oil P psig	Humidy %
1	2200.0	559.5	234.4	5.98	186.12	716.10	860.83	425.00	672.5	480.8	904.8	17.5	84.8	30.30	0.0281078	622.96	95.1	574.9	0.20	424.80	59.7	10.1
2	2200.0	469.8	196.8	5.76	151.81	676.79	628.07	445.11	569.5	444.7	858.5	16.8	84.2	30.30	0.0271109	587.83	96.7	545.5	0.03	445.08	60.0	9.8
3	2200.0	314.8	131.9	5.14	158.08	564.48	515.09	599.37	406	364.8	753.8	16.7	81.5	30.30	0.0243652	490.20	96.5	447.3	0.23	599.13	61.4	10.7
4	2200.0	61.5	25.8	3.05	236.92	219.31	180.43	833.89	168.5	276.8	512.0	17.1	80.5	30.30	0.0148564	190.63	96.4	156.8	1.05	832.84	63.5	11.3
5	1400.0	689.5	183.8	7.67	806.05	919.29	854.44	329.51	488.75	256.7	975.9	17.5	81.1	30.30	0.0359309	799.68	99.1	743.3	0.69	328.82	51.8	11.4
6	1400.0	578.0	154.1	7.44	560.14	929.22	870.91	328.30	406.75	227.2	943.9	17.4	81.7	30.30	0.0348096	808.19	102.4	757.5	0.80	327.50	52.5	11.1
7	1400.0	381.8	101.8	6.57	197.43	877.79	822.75	536.54	274	182.4	788.3	17.4	81.8	30.30	0.0308394	763.38	103.6	715.5	0.97	535.57	53.8	11.0
8	725.0	7.5	1.0	2.54	235.31	303.29	251.95	927.28	21.3	77.7	369.0	17.4	81.8	30.30	0.0125432	263.77	103.2	219.1	5.61	921.67	27.1	11.0

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	285.98	251.74	1384.27	1272.224	40350	1479481	127155.54	285.85	0.08
15.00	2	263.22	180.45	1147.93	1063.02	34170	1297574	107665.40	263.21	0.07
15.00	3	281.97	149.49	761.54	697.4976	24360	1026533	78424.69	281.86	0.09
10.00	4	269.58	153.96	203.51	171.0422	10110	692290	31091.89	269.26	0.18
10.00	5	124.65	612.87	998.92	926.5753	29205	844045	91617.54	124.38	0.07
10.00	6	107.21	367.68	871.55	816.0051	24405	727247	76777.05	106.95	0.06
10.00	7	133.74	98.92	628.36	591.295	16440	550846	51758.96	133.50	0.06
15.00	8	44.99	22.95	42.26	36.34594	1278	103418	3890.38	44.73	0.17

WTD AVG BHP = 131.15 KW = 97.80

WTD AVG GM/H =	AVG GM/BPH =	AVG GM/KWH =	W.F.													
			HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	MODE =	GP (g/Sec.) =	Sample Time (Sec.) =	mSEP (kg) =	Dilution Tunnel Total Flow (scmm) =	qmedf (kg/Hr.) =
			0.15	0.15	0.15	0.10	0.10	0.10	0.10	0.15	1	180.00	0.196	65.07	5047.9	24.51
			0.15	0.64	0.45	0.36	1.44	0.45	0.07	0.05	2	180.00	0.868	65.39	5070.5	20.72
			0.15	0.95	0.682	0.441	0.859	0.671	0.567	0.137	3	180.00	0.682	65.22	5073.1	18.59
			0.10	0.45	0.45	0.07	0.05	0.025	0.025	0.025	4	120.00	0.053	65.22	5080.2	34.51
			0.10	0.081	0.081	0.081	0.081	0.081	0.081	0.081	5	120.00	0.081	65.22	5080.2	70.69
			0.10	0.07	0.07	0.07	0.07	0.07	0.07	0.07	6	120.00	0.07	65.22	5080.2	28.28
			0.15	0.05	0.05	0.05	0.05	0.05	0.05	0.05	7	180.00	0.05	65.22	5080.2	5.21
			0.15	0.025	0.025	0.025	0.025	0.025	0.025	0.025	8	180.00	0.025	65.22	5080.2	10.26

WT AVG NO2/NOX RATIO = 0.098221833

WTD AVG GM/H = 24.9808

WEIGHTED AVG GM/BPH = 0.190

WTD AVG GM/KWH = 0.255



**CAT. 3306**  
**12-27-07 TEST# 8MLPV62**  
**8-MODE STEADY-STATE**  
**W/ARCO-VISCON BLEND DIESEL FUEL.**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	O2 %	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	Air In InH2O	ABSHUM GR/LB	Air In degF	Baro P InHga	l/aCAL FACTOR	KNOx PPM	FuelIn degF	KNO PPM	Methane ppm	NMHC ppm
1	2200.0	564.9	236.6	5.99	175.85	744.03	673.69	426.70	0.00	671	482.2	899.5	0.0	9.3	81.1	30.10	0.0281288	635.52	93.2	575.43872	0	
2	2200.0	470.2	197.0	5.77	128.67	716.49	647.36	434.68	0.00	566	429.7	850.5	0.0	9.1	82.0	30.10	0.0271213	611.64	94.6	552.62398	0	
3	2200.0	315.1	132.0	5.16	148.88	597.61	532.89	585.29	0.00	408	352.6	748.9	0.0	8.9	82.0	30.10	0.0244199	509.93	87.7	454.7117	0	
4	2200.0	61.3	25.7	3.13	218.69	241.32	192.46	826.89	0.00	167.25	262.8	502.0	0.0	8.6	80.3	30.10	0.015257	205.78	87.7	164.11201	0	
5	1400.0	699.0	186.3	7.78	813.79	965.07	874.24	306.67	0.00	495	245.6	975.1	0.0	8.8	81.0	30.10	0.0364269	823.30	85.9	745.80902	0.00	
6	1400.0	577.8	154.0	7.60	555.44	996.15	904.35	306.81	0.00	405	219.2	936.6	0.0	8.6	81.7	30.10	0.035498	849.40	87.0	771.12628	0	
7	1400.0	382.8	102.0	6.61	166.10	938.01	851.48	494.71	0.00	273	177.9	776.2	0.0	8.3	81.6	30.10	0.0309485	799.30	87.4	725.5736	0	
8	727.8	0.3	0.0	2.23	160.95	258.60	202.59	709.04	0.00	17	76.8	343.8	0.0	8.0	81.3	30.10	0.0109662	220.22	87.1	172.51972	0.00	

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC	
15.00	1	266.27	237.14	1407.93	1269.71	40260	1473441	126891.07	0	0.09
15.00	2	255.36	151.94	1186.58	1069.677	33960	1287733	107066.49	0	0.10
15.00	3	276.06	141.15	794.26	710.6543	24480	1028213	78838.47	0	0.11
10.00	4	258.27	137.30	212.24	172.7864	10035	688573	30915.04	0	0.20
10.00	5	116.31	620.36	1031.08	931.8075	29700	848052	93207.96	0	0.09
10.00	6	97.76	355.74	893.74	810.3405	24300	709683	76491.40	0	0.09
10.00	7	122.40	82.61	653.09	594.8193	16380	546270	51629.38	0	0.09
15.00	8	31.44	14.34	32.24	26.03783	1020	94139	3125.34	0	0.22

**Single Filter**  
 PARTIC. WT, MG = 0.312  
 PARTIC. SAMPLE VOL., STD LITERS = 9.89

WTD AVG BHP = 131.65      KW = 98.17

	HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC		MG/SL	0.03155
WTD AVG GM/H =	186.84	201.29	792.17	712	23000	859587	72313	0.00	WTD AVG GM/H =	20.944	
AVG GM/BHPH =	1.42	1.53	6.02	5.41	174.70	549.26	0.00	AVG GM/BHPH =	0.159		
AVG GM/KWH =	1.90	2.05	8.07	7.26	234.27	736.58	0.00	AVG GM/KWH =	0.213		

WT AVG NO2/NOX RATIO = 0.125484728

**CAT 3306**  
**12/28/2007 Test# 8MLPV63**  
**8-MODE STEADY-STATE**  
**W/ARCO-VISCON BLENDED FUEL**

Mode	EngSpd RPM	DynTrq lb-ft	EngPwr Hp	CO2 %	CO ppm	NOx ppm	NO ppm	HC FID ppm	FUEL RT GM/MIN	AirMas scfm	EngExh deg/F	ABSHUM GR/LB	Air In degF	Baro P InHga	f/aCAL FACTOR	KNOx PPM	Fuelln degF	KNO PPM	Methane ppm	NMHC ppm	Humidy %	Oil P psig
1	2200.0	556.1	232.9	6.11	173.86	710.81	665.07	430.46	864.5	478.1	897.1	17.4	83.2	30.10	0.0286625	618.17	92.7	578.39	0.001039	430.46	10.5	59.8
2	2200.0	470.7	197.2	5.85	142.71	677.23	632.98	460.60	569.5	432.9	854.0	18.7	81.9	30.10	0.0274861	590.70	95.5	552.11	0.029711	460.57	11.7	60.1
3	2200.0	315.0	131.9	5.22	170.63	558.48	509.39	605.49	405.5	358.9	752.0	19.8	80.9	30.10	0.0247223	488.34	96.7	445.41	0.0005544	805.49	12.8	61.6
4	2200.0	62.0	26.0	3.19	257.48	222.00	183.48	857.81	167	274.6	506.2	20.5	79.8	30.10	0.0155283	194.47	96.8	160.73	0.0030598	857.81	13.8	63.8
5	1400.0	895.9	185.5	7.81	822.00	895.09	846.07	310.71	490.5	254.9	974.9	21.2	78.9	30.10	0.0365792	785.19	99.8	742.19	0.00	310.72	14.8	52.0
6	1400.0	578.0	154.1	7.59	548.32	899.83	858.23	304.13	407.5	225.5	940.9	21.7	79.3	30.10	0.0354561	790.28	99.5	753.74	0.0022308	304.13	14.8	52.8
7	1399.9	382.5	102.0	6.67	173.13	841.25	804.63	477.84	274.5	182.3	784.0	21.9	79.8	30.10	0.0312153	739.25	98.0	707.07	0.0030837	477.83	14.7	54.2
8	730.3	0.5	0.1	2.88	166.48	238.95	213.39	696.46	20	76.1	349.5	22.0	80.2	30.10	0.0130553	210.01	96.4	187.54	0.01	696.45	14.8	28.3

WT. FAC %	Mode No	GRAMS/HOUR								NO2/NOX RATIO	Single Filter PARTIC. WT, MG = PARTIC. SAMPLE VOL., STD LITERS =
		HC	CO	KNOX	KNO	FUEL	EXHAUST	CO2	NMHC		
15.00	1	280.52	227.74	1330.30	1239.984	39870	1434334	125682.43	280.5	0.06	0.32
15.00	2	268.57	167.26	1137.39	1061.132	34170	1280658	107669.25	268.6	0.07	9.78
15.00	3	280.30	158.78	748.53	683.4943	24330	1011240	76319.86	280.3	0.09	
10.00	4	262.80	158.56	196.75	166.0846	10020	657188	30819.56	262.8	0.17	
10.00	5	116.27	618.25	970.22	915.0139	29430	836419	92351.84	116.3	0.05	
10.00	6	97.62	353.78	837.67	797.8645	24450	716170	76972.44	97.6	0.05	
10.00	7	117.83	85.81	601.98	577.5164	16470	545748	51925.30	117.8	0.04	
15.00	8	30.46	14.63	30.33	27.76143	1200	93406	3701.02	30.5	0.11	
WTD AVG BHP =		131.07		KW =	97.74						
WTD AVG GM/H =		188.43	206.90	747.34	698	22973	848498	72213	188.43		MG/SL 0.03272
AVG GM/BHPH =		1.44	1.58	5.70	5.32	175.26		550.94	1.44		AVG GM/BHPH = 0.164
AVG GM/KWH =		1.93	2.12	7.65	7.14	235.03		738.82	1.93		AVG GM/KWH = 0.219
WT AVG NO2/NOX RATIO =		0.080490911									



## 1.0 Scope and Application

- 1.1 This exhaust gas sampling protocol is specifically designed for sampling dilute exhaust generated from heavy-duty diesel engines being operated over the EPA transient cycle or steady state emission test as described in 40 CFR Part 86.
- 1.2 For transient cycle operation, this official testing protocol involves continual sample integration of all gaseous emissions along with pertinent engine and ambient variables for 1200 seconds (20 minutes). For eight-mode steady state operation, this official testing protocol involves modal sample collection for a total of 20 minutes. Modes 1, 2, 3 and 8 are collected for 3 minutes of the 5 minute mode, while modes 4, 5, 6 and 7 are collected for 2 minutes.
- 1.3 The constant volume sampling system (CVS) continually dilutes the exhaust gas with finely filtered room air to maintain a constant volume of exhaust gas plus dilute air over the test interval. Coincidentally a second bag is being filled continuously with the same air used to dilute the exhaust gas. Therefore at the end of each test there are bag samples of the proportional and integrated dilute exhaust and the filtered air that was used to dilute the exhaust.

## 2.0 Method Summary

- 2.1 Samples analyzed for Volatile Organic Compounds (VOC's) are collected from the integrated bag samples that have been continuously filled during the test cycle. One bag is filled with dilution air, while the other is filled with dilute exhaust. A Teflon diaphragm pump is T-connected through a valve to the constant volume sampling system to fill a Tedlar bag with either dilution air or dilute exhaust. For every emission sample and tunnel blank sampled, a dilution air sample must be co-sampled for background correction. Tedlar bag samples are analyzed by gas chromatography (GC).

## 3.0 Health and Safety

- 3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.
- 3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

## 4.0 Sample Preservation, Containers, Handling, and Storage

- 4.1 Samples are collected and stored in 1 L Tedlar bags.
- 4.2 Tedlar bags may not be exposed to heat or excessive light. Black Tedlar bags may be used to eliminate photochemically induced reactions.
- 4.3 Samples must be analyzed within 24 hours of collection.
- 4.4 To prevent sample contamination, Tedlar bags are used only once.

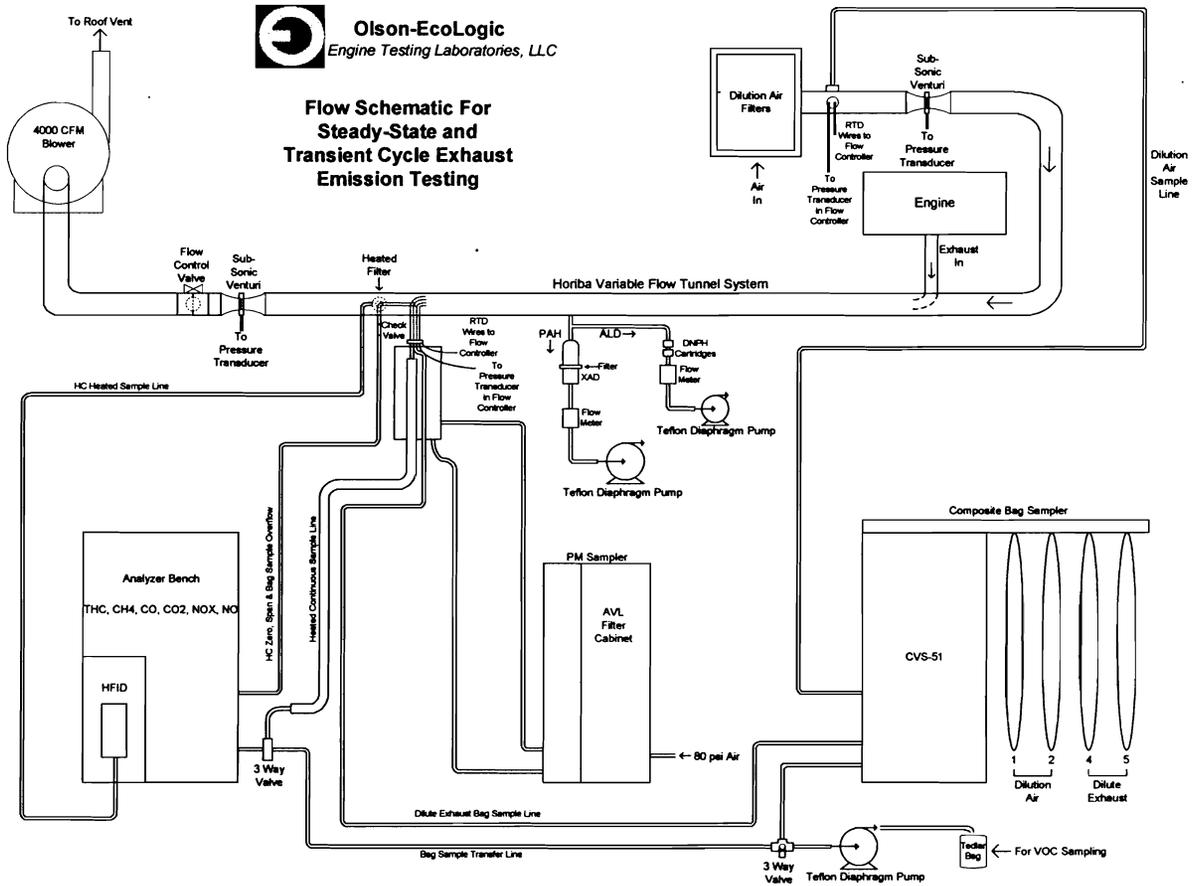
**5.0 Interferences and Potential Problems**

5.1 To maximize sample integrity, Tedlar bags should not leak or be exposed to excessive light or heat. Tedlar bags must be shielded from direct sunlight to avoid photochemically induced reactions of any reactive hydrocarbons.

5.1.1 The compound 1,3-butadiene, resulting mostly during cold-start testing by the combustion of olefins, is extremely unstable as it easily polymerizes in the presence of oxygen. Therefore all samples analyzed for 1,3-butadiene must be run within 1 hour of collection.

**6.0 Equipment/Apparatus**

- 6.1 Tedlar bags: SKC, Inc., 1 L in capacity, or equivalent
- 6.2 Super Syringe: Fisher Scientific, 1 L
- 6.3 Teflon diaphragm vacuum-pressure pump: Barnant Co., or equivalent
- 6.4 Sampling Train Schematic



## 7.0 Procedure

### 7.1 Sample collection

- 7.1.1 After the analysis of the bag samples collected from the emission test, a T-connected valve is turned enabling the sample to flow through the Teflon diaphragm pump to the Tedlar bag.
- 7.1.2 A dilution air sample is collected first by setting the CVS to “read bag 1 or 2”. Before connecting the Tedlar bag to the pump, the pump should be run for about 30 seconds to flush any contaminants from previous samples. The Tedlar bag is then attached to the pump and powered on to collect the sample.
- 7.1.3 A tunnel blank or dilute exhaust sample is collected next by setting the CVS to “read bag 4 or 5”. Before connecting the Tedlar bag to the pump, the pump should be run for about 30 seconds to flush any contaminants from previous samples. The Tedlar bag is then attached to the pump and powered on to collect the sample.
- 7.1.4 The dilution and work is noted for each dilute exhaust sample taken for final calculations.
- 7.1.5 The Tedlar bag samples are quickly taken into the laboratory and shaded from direct light for analysis by GC.

## 8.0 Quality Control/Quality Assurance

### 8.1 Dilution Air Sample

- 8.1.1 A dilution air sample is collected for every emission sample and tunnel blank to correct for background levels in the ambient dilution air.

### 8.2 Tunnel Blank Sample

- 8.2.1 A tunnel blank sample is collected each analysis day to note any hydrocarbon contamination that may be in the tunnel.

### 8.3 Duplicate Sample

- 8.3.1 A duplicate emission sample is collected each analysis day for quality purposes.

### 8.4 Data Comparisons

- 8.4.1 The VOC sampling protocol provides the same gas sample used in the official calculation of results from the emission test. The hydrocarbon (HC) data can be compared to the GC data.
- 8.4.2 The primary bag gas data are printed out on every test report automatically along with other engine operating data. The dilution ratio and work for the entire test is also printed out on each report from the precise CVS flow data. Accordingly, there is generally no need to dilute the Tedlar bag samples before GC analysis.

## 8.5 Leak Checks

### 8.5.1 Sampling Train

8.5.1.1 To leak check any part of the sampling train, the suspected leak area should be isolated and pressurized or put under vacuum. A gauge may then be used to check if a leak exists.

### 8.5.2 Tedlar Bags

8.5.2.1 Tedlar bags may be leak checked by filling the bag and leaving it under pressure overnight. If the bag deflates, it should not be used for sample collection.

## 8.6 Flow Rates

8.6.1 The flow rate into the CVS bags is controlled by the Horiba CVS with needle valves. The flow rate over the 20 minute transient test cycle is 5 L/min, while the flow rate over the 40 minute steady state test cycle is 2.5 L/min.



## 1.0 Scope and Application

- 1.1 This SOP is based on CARB Method 1002 and describes the use of gas chromatography (GC) coupled with flame ionization detection (FID) for the determination of C<sub>2</sub>-C<sub>5</sub> hydrocarbons (light-end hydrocarbons) in the ppbC range from automotive source samples. The compounds listed below may be determined by this method:

Compound	CAS Registry No.
ethene	00074-85-1
ethyne	00074-86-2
ethane	00074-84-0
propene	00115-07-1
propane	00074-98-6
1,2-propadiene	00463-49-0
1-propyne	00074-99-7
2-methylpropane	00075-28-5
2-methylpropene (isobutene)	00115-11-7
1-butene	00106-98-9
1,3-butadiene	00106-99-0
n-butane	00106-97-8
trans-2-butene	00624-64-6
2,2-dimethylpropane	00463-82-1
1-butyne	00107-00-6
cis-2-butene	00590-18-1
3-methyl-1-butene	00563-45-1
2-methylbutane	00078-78-4
2-butyne	00503-17-3
1-pentene	00109-67-1
2-methyl-1-butene	00563-46-2
n-pentane	00109-66-0
2-methyl-1,3-butadiene	00078-79-5
trans-2-pentene	00646-04-8
3,3-dimethyl-1-butene	00558-37-2
cis-2-pentene	00627-20-3
1-buten-3-yne	00689-97-4
2-methyl-2-butene	00513-35-9
1,3-cyclopentadiene	00542-92-7
2,2-dimethylbutane	00075-83-2
cyclopentene	00142-29-0
4-methyl-1-pentene	00691-37-2
3-methyl-1-pentene	00760-20-3
cyclopentane	00287-92-3
2,3-dimethylbutane	00079-29-8
1-methyl-tert-butyl-ether	01634-04-4
4-methyl-cis-2-pentene	00691-38-3
2-methylpentane	00107-83-5
4-methyl-trans-2-pentene	00674-76-0
3-methylpentane	00096-14-0

2-methyl-1-pentene	00763-29-1
1-hexene	00592-41-6
n-hexane	00110-54-3

- 
- 1.2 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs. Analysts should also be skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 Method Summary

- 2.1 This method provides GC/FID conditions for the detection of the target analytes. Exhaust samples are introduced to the GC from Tedlar bags by means of gas sampling valves. Separation of the sample hydrocarbons takes place in a 50 m 0.32 mm ID PLOT fused silica column. Quantitative analysis is performed by the FID using an external standard approach. The computerized GC data acquisition system identifies the hydrocarbons and concentrations are determined by peak area response factors.
- 2.2 Prior to the use of this method, appropriate sample collection techniques must be used.
- 2.2.1 Samples are collected from the exhaust in Tedlar bags. Dilutions may apply and must be accounted for in final calculations.

## 3.0 Health and Safety

- 3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.
- 3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

## 4.0 Sample Preservation, Containers, Handling, and Storage

- 4.1 Samples are collected and stored in 1 L Tedlar bags.
- 4.2 Tedlar bags may not be exposed to heat or excessive light. Black Tedlar bags may be used to eliminate photochemically induced reactions.
- 4.3 Samples must be analyzed within 24 hours of collection.

## 5.0 Interferences and Potential Problems

5.1 To maximize sample integrity, Tedlar bags should not leak or be exposed to excessive light or heat. Tedlar bags must be shielded from direct sunlight to avoid photochemically induced reactions of any reactive hydrocarbons.

5.1.1 The compound 1,3-butadiene, resulting mostly during cold-start testing by the combustion of olefins, is extremely unstable as it easily polymerizes in the presence of oxygen. Therefore all samples analyzed for 1,3-butadiene must be run within 1 hour of collection.

5.2 Any component present in the sample with a retention time very similar to that of a target hydrocarbon would interfere or coelute. If separation cannot be achieved, confirmation of identification should be done using a different column for separation, or an alternate detector, e.g., mass spectrometer (MS), photoionization detector (PID), etc.

## 6.0 Equipment/Apparatus

### 6.1 GC/FID

6.1.1 Gas Chromatograph (GC) – Varian CP-3800 with programmable oven temperatures, 10 mL fixed volume injection loop for automated transfer of gaseous samples from the Tedlar bag to the GC, and analytical column interfaced with a flame ionization detector (FID).

6.1.1.1 GC Column – Varian CP 7515 PLOT fused silica, 50 m x 0.32 mm ID, or equivalent.

6.1.2 Data System – Dell-PC computer with Varian Star software capable of continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2 Nitrogen, compressed and liquid. Minimum purity of 99.998 %.

6.3 Helium, compressed. Minimum purity of 99.995 %.

6.4 Hydrogen, compressed. Minimum purity of 99.995 %.

6.5 Air, compressed. “Zero” grade (<1 ppmC total hydrocarbon contamination), or better.

6.6 Tedlar bags: SKC, Inc., 5 to 10 L in capacity, or equivalent.

6.7 Super Syringe: Fisher Scientific, 1 L.

## 7.0 Reagents

7.1 NIST-certified SRM or secondary NIST-traceable standards shall be used in all tests. A secondary standard is obtained by a comparison between a SRM and a candidate standard.

### 7.2 Calibration Standard

7.2.1 The quantitative calibration standard for all target hydrocarbons is propane. Lehner/Martin, Inc. Propane Std. in zero air – 2955 ppbC, or equivalent

### 7.3 Control Standard

7.3.1 Quality control standard, containing at least ethane, propane, n-butane, 2-methylpropene and 1,3-butadiene at concentrations between 200 and 3000 ppbC based on a propane standard. This standard is used as a daily update of control charts and a daily determination of marker retention time windows.

Scott-Marrin, Inc. 23 Component custom blend in nitrogen, or equivalent

7.4 A high concentration standard (higher than the calibration standard), containing the target hydrocarbons listed in Section 7.3.1, is used for linearity determinations. The high concentration standard must have concentrations verified against a NIST-traceable propane standard.

7.5 A low concentration standard (5 to 10 times the estimated MDL), containing the target hydrocarbons listed in Section 7.3.1, is used for MDL determinations. The low concentration standard must have concentrations verified against a NIST-traceable propane standard.

7.5.1 In lieu of a low concentration standard, a higher concentration standard may be diluted.

## 8.0 Procedure

### 8.1 Sample collection

8.1.1 Specific sample collection procedures can be found in the VOC Sampling Protocol.

8.1.2 In general, samples are collected from the integrated bag samples that have been continuously filled during the 1200 second transient test cycle. One bag is filled with dilution air, while the other is filled with dilute exhaust. A Teflon diaphragm pump is T-connected through a valve to the constant volume sampling system to fill a Tedlar bag with either dilution air or dilute exhaust.

### 8.2 GC chromatographic conditions:

Injection volume: 10 mL fixed loop  
Injector temperature: -180 C (hold 7.10 min) to 250 C (hold 61.25 min) @ 200 C/min  
Helium carrier flow: 3 mL/min  
Nitrogen aux. flow: 27 mL/min  
Hydrogen flow: 30 mL/min  
Air flow: 300 mL/min  
Column temperature: 0 C (hold 10 min) to 200 C (hold 20 min) @ 5 C/min  
Detector temperature: 250 C

8.3 Samples in Tedlar bags are connected to one of sixteen ports on the autosampler and the analytical process then begins.

8.4 The sample is introduced into the carrier gas stream through the injection valve.

8.5 Each separated analyte exits the column into the FID where a response is generated.

8.6 Hydrocarbon concentrations are calculated in parts per billion of carbon (ppbC) by Varian's Star software from the NIST-traceable propane calibration standard.

- 8.7 Analytes with concentrations higher than demonstrated in the instruments range of linearity must be diluted and rerun.
- 8.8 Peak identification and integration are checked and corrected if necessary by the analyst.
- 8.9 Target compounds that coelute are reported as the major component.
- 8.10 The PLOT fused silica analytical column is heated to 200 C to prevent carry over and assure all compounds are eluted before the next run.

## 9.0 Calculations

- 9.1 The target hydrocarbon concentrations, in ppbC, are calculated by the data system using propane as an external standard.

$$\text{Concentration}_{\text{sample}} \text{ (ppbC)} = \text{Peak Area}_{\text{sample}} \times \text{Response Factor}$$

where the response factor (RF) is calculated during daily calibration by:

$$\text{RF} = \frac{\text{Concentration of propane standard (ppbC)}}{\text{area of propane peak}}$$

## 10.0 Quality Control/Quality Assurance

### 10.1 Instrument Blank Run

- 10.1.1 An instrument blank of pure nitrogen is run each analysis day. All target hydrocarbon concentrations from the blank analysis must be below the method detection limit (MDL) before the analysis may proceed.

- 10.1.1.1 If the blank shows a peak greater than the MDL in the region of interest, the source of contamination must be investigated and remedied.

### 10.2 Calibration Run

- 10.2.1 The calibration standard is analyzed each analysis day to generate the response factor used to quantify the sample concentrations.

### 10.3 Control Standard Run

- 10.3.1 The quality control standard is analyzed at least once each analysis day. Measurements of all compounds specified in Section 7.3.1 must fall within the control limits to ensure the validity of the sample analyses that day. To meet this requirement, it may be necessary to inspect and repair the GC, and rerun the calibration and/or control standards.

## 10.4 Control Charts

10.4.1 A quality control chart is maintained for each component of the control standard listed in Section 7.3.1, and is performed for new instruments, after making instrument modifications that can affect recovery, and at least once every year. The control charts, used on a daily basis, establish that the method is “in control.” The following describes how to construct a typical control chart:

1. Obtain at least 20 daily control standard results;
2. Calculate the control standard mean concentration and standard deviation for the target hydrocarbon; and
3. Create a control chart for the target hydrocarbon by placing the concentration on the Y-axis and the date on the X-axis. Establish upper and lower warning limits at either two standard deviations (2s) or 5 percent, whichever is greater, above and below the average concentration.
4. A control standard measurement is considered to be out-of-control when the analyzed value exceeds the control limit or two successive control standard measurements of the same analyte exceed the warning limit.
5. If 20 control standard measurements are not yet available to create a control chart (e.g., the control standard was expended and replaced prior to obtaining 20 points with the new standard), measurements must be within 15% relative standard deviation (RSD) of the certified concentration. If the control standard is not a NIST standard, the cylinder should be certified by the laboratory against a NIST standard.

The measured concentrations of all target hydrocarbons contained in the control standard must be within the control limits (in control) for the sample results to be considered acceptable.

## 10.5 Duplicates

10.5.1 A duplicate analysis of one sample is performed at least once per analysis day. The relative percent difference (RPD) is calculated for each duplicate run:

$$\text{RPD (\%)} = \frac{\text{Difference between duplicate and original measurement}}{\text{Average of duplicate and original measurement}} \times 100$$

For each compound specified in Section 7.3.1, the allowable RPD depends on the average concentration level for the duplicate runs, as shown in the following table:

<u>Average Measurement for the Duplicate Runs</u>		<u>Allowable RPD (%)</u>
1 to 10	times MDL	100
10 to 20	“ “	30
20 to 50	“ “	20
Greater than 50	“ “	15

If the results of the duplicate analyses do not meet these criteria for all compounds specified in Section 7.3.1, the sample may be reanalyzed. If reanalysis is not feasible or if the criteria are still not met on reanalysis, all sample results for that analysis day are invalid.

## 10.6 Linearity

10.6.1 A multipoint calibration to confirm instrument linearity is performed for the target hydrocarbons in the control standard for new instruments, after making instrument modifications that can affect linearity, and at least once every year. The multipoint calibration consists of at least five concentration or mass loading levels (using smaller or larger volume sample sizes of existing standards is acceptable), each above the MDL, distributed over the range of expected sample concentration. A linear regression analysis is performed using concentration and average area counts to determine the regression correlation coefficient (r). The r must be greater than 0.995 to be considered sufficiently linear for one-point calibrations.

## 10.7 MDL

10.7.1 The MDL for the target hydrocarbons in the control standard must be determined for new instruments, after making instrument modifications that can affect linearity and/or sensitivity, and at least once every year. To make the calculations, it is necessary to run at least seven replicate determinations at a concentration of 5 to 10 times the estimated MDL. The MDL is calculated using the following equation:

$$\text{MDL} = t \times s$$

where s is the standard deviation of the replicates and t is the t-factor for 99 percent confidence for a one-sided normal (Gaussian) distribution. The number of degrees of freedom is equal to the number of replicates, minus one. An abbreviated t-table is:

<u>Degrees of Freedom</u>	<u>t-value</u>
4	3.7
5	3.4
6	3.1
7	3.0

10.7.1.1 The maximum allowable MDL for each compound is 1 ppbC. The calculated laboratory MDL must be equal to or lower than the maximum allowable MDL. All peaks identified as target compounds that are equal to or exceed the maximum allowable MDL must be reported. If the calculated laboratory MDL is less than the maximum allowable MDL, the laboratory may choose to set its reporting limit at the maximum allowable MDL, the calculated laboratory MDL, or any level in between.

10.7.1.2 For the purpose of calculating the total mass of all species, the concentrations of all compounds below the MDL are considered to be zero.

## 10.8 Method 1002/1003 Crossover Check

10.8.1 A crossover check is analyzed at least once each analysis day, and is performed by choosing a compound from a sample to be measured and compared by both Method 1002 and 1003. The crossover compound shall be a compound that can reasonably be expected to be found and measured by both methods in the laboratory performing the analysis. The maximum relative percent difference (RPD) allowed from the results obtained by the two methods is 15%.

## 11.0 References

- 11.1 California Environmental Protection Agency, Air Resources Board, Method 1002, Revision IV, July 2002



## 1.0 Scope and Application

- 1.1 This SOP is based on CARB Method 1003 and describes the use of gas chromatography (GC) coupled with flame ionization detection (FID) for the determination of C<sub>6</sub>-C<sub>12</sub> hydrocarbons (mid-range hydrocarbons) in the ppbC range from automotive source samples. The compounds listed below may be determined by this method:

Compound	CAS Registry No.
n-hexane	00110-54-3
trans-3-hexene	13269-52-8
cis-3-hexene	07642-09-3
trans-2-hexene	04050-45-7
3-methyl-trans-2-pentene	00616-12-6
2-methyl-2-pentene	00625-27-4
3-methylcyclopentene	01120-62-3
cis-2-hexene	07688-21-3
1-ethyl-tert-butyl-ether	00637-92-3
3-methyl-cis-2-pentene	00922-62-3
2,2-dimethylpentane	00590-35-2
methylcyclopentane	00096-37-7
2,4-dimethylpentane	00108-08-7
2,2,3-trimethylbutane	00464-06-2
3,4-dimethyl-1-pentene	07385-78-6
1-methylcyclopentene	00693-89-0
benzene	00071-43-2
3-methyl-1-hexene	03404-61-3
3,3-dimethylpentane	00562-49-2
cyclohexane	00110-82-7
2-methylhexane	00591-76-4
2,3-dimethylpentane	00565-59-3
cyclohexene	00110-83-8
3-methylhexane	00589-34-4
trans-1,3-dimethylcyclopentane	01759-58-6
cis-1,3-dimethylcyclopentane	02532-58-3
3-ethylpentane	00617-78-7
trans-1,2-dimethylcyclopentane	00822-50-4
1-heptene	00592-76-7
2,2,4-trimethylpentane	00540-84-1
trans-3-heptene	14686-14-7
n-heptane	00142-82-5
2-methyl-2-hexene	02738-19-4
3-methyl-trans-3-hexene	03899-36-3
trans-2-heptene	14686-13-6
3-ethyl-2-pentene	00816-79-5
2,4,4-trimethyl-1-pentene	00107-39-1
2,3-dimethyl-2-pentene	10574-37-5
cis-2-heptene	06443-92-1
methylcyclohexane	00108-87-2

2,2-dimethylhexane	00590-73-8
2,4,4-trimethyl-2-pentene	00107-40-4
ethylcyclopentane	01640-89-7
2,5-dimethylhexane	00592-13-2
2,4-dimethylhexane	00589-43-5
1,2,4-trimethylcyclopentane	02815-58-9
3,3-dimethylhexane	00563-16-6
2,3,4-trimethylpentane	00565-75-3
2,3,3-trimethylpentane	00560-21-4
toluene	00108-88-3
2,3-dimethylhexane	00584-94-1
2-methylheptane	00592-27-8
4-methylheptane	00589-53-7
3-methylheptane	00589-81-1
(1a,2a,3b)-1,2,3-trimethylcyclopentane	15890-40-1
cis-1,3-dimethylcyclohexane	00638-04-0
trans-1,4-dimethylcyclohexane	02207-04-7
2,2,5-trimethylhexane	03522-94-9
trans-1-methyl-3-ethylcyclopentane	02613-65-2
cis-1-methyl-3-ethylcyclopentane	16747-50-5
1-octene	00111-66-0
trans-4-octene	14850-23-8
n-octane	00111-65-9
trans-2-octene	13389-42-9
trans-1,3-dimethylcyclohexane	02207-03-6
cis-2-octene	07642-04-8
2,3,5-trimethylhexane	01069-53-0
2,4-dimethylheptane	02213-23-2
cis-1,2-dimethylcyclohexane	02207-01-4
2,6-dimethylheptane	01072-05-5
ethylcyclohexane	01678-91-7
3,5-dimethylheptane	00926-82-9
ethylbenzene	00100-41-4
2,3-dimethylheptane	03074-71-3
m-&p-xylene	00108-38-3
4-methyloctane	02216-34-4
2-methyloctane	03221-61-2
3-methyloctane	02216-33-3
styrene (ethenylbenzene)	00100-42-5
o-xylene	00095-47-6
1-nonene	00124-11-8
n-nonane	00111-84-2
(1-methylethyl)benzene	00098-82-8
2,2-dimethyloctane	15869-87-1
2,4-dimethyloctane	04032-94-4
2,6-dimethyloctane	02051-30-1
n-propylbenzene	00103-65-1
1-methyl-3-ethylbenzene	00620-14-4
1-methyl-4-ethylbenzene	00622-96-8

1,3,5-trimethylbenzene	00108-67-8
1-methyl-2-ethylbenzene	00611-14-3
1,2,4-trimethylbenzene	00095-63-6
n-decane	00124-18-5
(2-methylpropyl)benzene	00538-93-2
(1-methylpropyl)benzene	00135-98-8
1-methyl-3-(1-methylethyl)benzene	00535-77-3
1,2,3-trimethylbenzene	00526-73-8
1-methyl-4-(1-methylethyl)benzene	00099-87-6
2,3-dihydroindene (indan)	00496-11-7
1-methyl-2-(1-methylethyl)benzene	00527-84-4
1,3-diethylbenzene	00141-93-5
1,4-diethylbenzene	00105-05-5
1-methyl-3-n-propylbenzene	01074-43-7
1-methyl-4-n-propylbenzene	01074-55-1
1,2-diethylbenzene	00135-01-3
1-methyl-2-n-propylbenzene	01074-17-5
1,4-dimethyl-2-ethylbenzene	01758-88-9
1,3-dimethyl-4-ethylbenzene	00874-41-9
1,2-dimethyl-4-ethylbenzene	00934-80-5
1,3-dimethyl-2-ethylbenzene	02870-04-4
n-undecane (hendecane)	01120-21-4
1,2-dimethyl-3-ethylbenzene	00933-98-2
1,2,4,5-tetramethylbenzene	00095-93-2
1-methyl-2-n-butylbenzene	01595-11-5
1,2,3,5-tetramethylbenzene	00527-53-7
1-(1,1-dimethylethyl)-2-methylbenzene	01074-92-6
1,2,3,4-tetramethylbenzene	00488-23-3
n-pentylbenzene	00538-68-1
1-(1,1-dimethylethyl)-3,5-dimethylbenzene	00098-19-1
naphthalene	00091-20-3
n-dodecane	00112-40-3
n-tridecane	00629-50-5

- 
- 
- 1.2 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs. Analysts should also be skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 Method Summary

- 2.1 This method provides GC/FID conditions for the detection of the target analytes. Exhaust samples are introduced to the GC from Tedlar bags by means of gas sampling valves. Separation of the sample hydrocarbons takes place in a 60 m 0.32 mm ID WCOT fused silica column. Quantitative analysis is

performed by the FID using an external standard approach. The computerized GC data acquisition system identifies the hydrocarbons and concentrations are determined by peak area response factors.

2.2 Prior to the use of this method, appropriate sample collection techniques must be used.

2.2.1 Samples are collected from the exhaust in Tedlar bags. Dilutions may apply and must be accounted for in final calculations.

### **3.0 Health and Safety**

3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

### **4.0 Sample Preservation, Containers, Handling, and Storage**

4.1 Samples are collected and stored in 1 L Tedlar bags.

4.2 Tedlar bags may not be exposed to heat or excessive light. Black Tedlar bags may be used to eliminate photochemically induced reactions.

4.3 Samples must be analyzed within 24 hours of collection.

### **5.0 Interferences and Potential Problems**

5.1 To maximize sample integrity, Tedlar bags should not leak or be exposed to excessive light or heat. Tedlar bags must be shielded from direct sunlight to avoid photochemically induced reactions of any reactive hydrocarbons.

5.2 Any component present in the sample with a retention time very similar to that of a target hydrocarbon would interfere or coelute. If separation cannot be achieved, confirmation of identification should be done using a different column for separation, or an alternate detector, e.g., mass spectrometer (MS), photoionization detector (PID), etc.

### **6.0 Equipment/Apparatus**

6.1 GC/FID

6.1.1 Gas Chromatograph (GC) – Varian CP-3800 with programmable oven temperatures, 30 mL fixed volume injection loop for automated transfer of gaseous samples from the Tedlar bag to the GC, and analytical column interfaced with a flame ionization detector (FID).

6.1.1.1 GC Column – Varian CP 8870 WCOT fused silica, 60 m x 0.32 mm ID, or equivalent.

- 6.1.2 Data System – Dell-PC computer with Varian Star software capable of continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.
- 6.2 Nitrogen, compressed and liquid. Minimum purity of 99.998 %.
- 6.3 Helium, compressed. Minimum purity of 99.995 %.
- 6.4 Hydrogen, compressed. Minimum purity of 99.995 %.
- 6.5 Air, compressed. “Zero” grade (<1 ppmC total hydrocarbon contamination), or better.
- 6.6 Tedlar bags: SKC, Inc., 5 to 10 L in capacity, or equivalent.
- 6.7 Super Syringe: Fisher Scientific, 1 L

## 7.0 Reagents

- 7.1 NIST-certified SRM or secondary NIST-traceable standards shall be used in all tests. A secondary standard is obtained by a comparison between a SRM and a candidate standard.
- 7.2 Calibration Standard
  - 7.2.1 The quantitative calibration standard for all target hydrocarbons is propane.  
Lehner/Martin, Inc. Propane Std. in zero air – 2955 ppbC, or equivalent
- 7.3 Control Standard
  - 7.3.1 Quality control standard, containing at least n-hexane, benzene, toluene, n-octane, ethylbenzene, m&p-xylene, o-xylene and n-decane at concentrations between 200 and 2000 ppbC based on a propane standard. This standard is used as a daily update of control charts and a daily determination of marker retention time windows.  
Scott-Marrin, Inc. 23 Component custom blend in nitrogen, or equivalent
- 7.4 A high concentration standard (higher than the calibration standard), containing the target hydrocarbons listed in Section 7.3.1, is used for linearity determinations. The high concentration standard must have concentrations verified against a NIST-traceable propane standard.
- 7.5 A low concentration standard (5 to 10 times the estimated MDL), containing the target hydrocarbons listed in Section 7.3.1, is used for MDL determinations. The low concentration standard must have concentrations verified against a NIST-traceable propane standard.
  - 7.5.1 In lieu of a low concentration standard, a higher concentration standard may be diluted.

## 8.0 Procedure

## 8.1 Sample collection

8.1.1 Specific sample collection procedures can be found in the VOC Sampling Protocol.

8.1.2 In general, samples are collected from the integrated bag samples that have been continuously filled during the 1200 second transient test cycle. One bag is filled with dilution air, while the other is filled with dilute exhaust. A Teflon diaphragm pump is T-connected through a valve to the constant volume sampling system to fill a Tedlar bag with either dilution air or dilute exhaust.

## 8.2 GC chromatographic conditions:

Injection volume: 30 mL fixed loop  
Injector temperature: -180 C (hold 7.10 min) to 250 C (hold 61.25 min) @ 200 C/min  
Helium carrier flow: 3 mL/min  
Nitrogen aux. flow: 27 mL/min  
Hydrogen flow: 30 mL/min  
Air flow: 300 mL/min  
Column temperature: 10 C (hold 9 min) to 250 C (hold 13 min) @ 5 C/min  
Detector temperature: 300 C

8.3 Samples in Tedlar bags are connected to one of sixteen ports on the autosampler and the analytical process then begins.

8.4 The sample is introduced into the carrier gas stream through the injection valve.

8.5 Each separated analyte exits the column into the FID where a response is generated.

8.6 Hydrocarbon concentrations are calculated in parts per billion of carbon (ppbC) by Varian's Star software from the NIST-traceable propane calibration standard.

8.7 Analytes with concentrations higher than demonstrated in the instruments range of linearity must be diluted and rerun.

8.8 Peak identification and integration are checked and corrected if necessary by the analyst using the following procedure and criteria:

1. The primary peak identification is done by the computer using the relative retention times based on reference calibration runs.
2. Confirm that the relative peak heights of the sample run ("fingerprint") match the typical fingerprint seen in past sample runs.
3. Compare the relative retention times of the sample peaks with those of reference runs.
4. Any peak with a reasonable doubt is labeled 'Unidentified'.

8.9 Target compounds that coelute, with the exception of m&p-xylene, are reported as the major component. Due to the difficulty in separation of m-xylene and p-xylene, they are reported together as m&p-xylene.

8.10 The WCOT fused silica analytical column is heated to 250 C to prevent carry over and assure all compounds are eluted before the next run.

8.10.1 After running a particularly "dirty" sample, the analyst should run a blank before proceeding to the next sample as there may be sample carry over, or flush the sampling system with air.

## 9.0 Calculations

- 9.1 The target hydrocarbon concentrations, in ppbC, are calculated by the data system using propane as an external standard.

$$\text{Concentration}_{\text{sample}} \text{ (ppbC)} = \text{Peak Area}_{\text{sample}} \times \text{Response Factor}$$

where the response factor (RF) is calculated during daily calibration by:

$$\text{RF} = \frac{\text{Concentration of propane standard (ppbC)}}{\text{area of propane peak}}$$

## 10.0 Quality Control/Quality Assurance

### 10.1 Instrument Blank Run

- 10.1.1 An instrument blank of pure nitrogen is run each analysis day. All target hydrocarbon concentrations from the blank analysis must be below the method detection limit (MDL) before the analysis may proceed.

10.1.1.1 If the blank shows a peak greater than the MDL in the region of interest, the source of contamination must be investigated and remedied.

### 10.2 Calibration Run

- 10.2.1 The calibration standard is analyzed each analysis day to generate the response factor used to quantify the sample concentrations.

### 10.3 Control Standard Run

- 10.3.1 The quality control standard is analyzed at least once each analysis day. Measurements of all compounds specified in Section 7.3.1 must fall within the control limits to ensure the validity of the sample analyses that day. To meet this requirement, it may be necessary to inspect and repair the GC, and rerun the calibration and/or control standards.

### 10.4 Control Charts

- 10.4.1 A quality control chart is maintained for each component of the control standard listed in Section 7.3.1, and is performed for new instruments, after making instrument modifications that can affect recovery, and at least once every year. The control charts, used on a daily basis, establish that the method is "in control." The following describes how to construct a typical control chart:

1. Obtain at least 20 daily control standard results;
2. Calculate the control standard mean concentration and standard deviation for the target hydrocarbon; and

3. Create a control chart for the target hydrocarbon by placing the concentration on the Y-axis and the date on the X-axis. Establish upper and lower warning limits at either two standard deviations (2s) or 5 percent, whichever is greater, above and below the average concentration.
4. A control standard measurement is considered to be out-of-control when the analyzed value exceeds the control limit or two successive control standard measurements of the same analyte exceed the warning limit.
5. If 20 control standard measurements are not yet available to create a control chart (e.g., the control standard was expended and replaced prior to obtaining 20 points with the new standard), measurements must be within 15% relative standard deviation (RSD) of the certified concentration. If the control standard is not a NIST standard, the cylinder should be certified by the laboratory against a NIST standard.

The measured concentrations of all target hydrocarbons contained in the control standard must be within the control limits (in control) for the sample results to be considered acceptable.

## 10.5 Duplicates

- 10.5.1 A duplicate analysis of one sample is performed at least once per analysis day. The relative percent difference (RPD) is calculated for each duplicate run:

$$\text{RPD (\%)} = \frac{\text{Difference between duplicate and original measurement}}{\text{Average of duplicate and original measurement}} \times 100$$

For each compound specified in Section 7.3.1, the allowable RPD depends on the average concentration level for the duplicate runs, as shown in the following table:

<u>Average Measurement for the Duplicate Runs</u>		<u>Allowable RPD (%)</u>
1 to 10	times MDL	100
10 to 20	“ “	30
20 to 50	“ “	20
Greater than 50	“ “	15

If the results of the duplicate analyses do not meet these criteria for all compounds specified in Section 7.3.1, the sample may be reanalyzed. If reanalysis is not feasible or if the criteria are still not met on reanalysis, all sample results for that analysis day are invalid.

## 10.6 Linearity

- 10.6.1 A multipoint calibration to confirm instrument linearity is performed for the target hydrocarbons in the control standard for new instruments, after making instrument modifications that can affect linearity, and at least once every year. The multipoint calibration consists of at least five concentration or mass loading levels (using smaller or larger volume sample sizes of existing standards is acceptable), each above the MDL, distributed over the range of expected sample concentration. A linear regression analysis is performed using concentration and average area counts to determine the regression correlation coefficient (r). The r must be greater than 0.995 to be considered sufficiently linear for one-point calibrations.

## 10.7 MDL

10.7.1 The MDL for the target hydrocarbons in the control standard must be determined for new instruments, after making instrument modifications that can affect linearity and/or sensitivity, and at least once every year. To make the calculations, it is necessary to run at least seven replicate determinations at a concentration of 5 to 10 times the estimated MDL. The MDL is calculated using the following equation:

$$\text{MDL} = t \times s$$

where *s* is the standard deviation of the replicates and *t* is the *t*-factor for 99 percent confidence for a one-sided normal (Gaussian) distribution. The number of degrees of freedom is equal to the number of replicates, minus one. An abbreviated *t*-table is:

<u>Degrees of Freedom</u>	<u>t-value</u>
4	3.7
5	3.4
6	3.1
7	3.0

10.7.1.1 The maximum allowable MDL for each compound is 1 ppbC. The calculated laboratory MDL must be equal to or lower than the maximum allowable MDL. All peaks identified as target compounds that are equal to or exceed the maximum allowable MDL must be reported. If the calculated laboratory MDL is less than the maximum allowable MDL, the laboratory may choose to set its reporting limit at the maximum allowable MDL, the calculated laboratory MDL, or any level in between.

10.7.1.2 For the purpose of calculating the total mass of all species, the concentrations of all compounds below the MDL are considered to be zero.

## 10.8 Method 1002/1003 Crossover Check

10.8.1 A crossover check is analyzed at least once each analysis day, and is performed by choosing a compound from a sample to be measured and compared by both Method 1002 and 1003. The crossover compound shall be a compound that can reasonably be expected to be found and measured by both methods in the laboratory performing the analysis. The maximum relative percent difference (RPD) allowed from the results obtained by the two methods is 15%.

## 11.0 References

11.1 California Environmental Protection Agency, Air Resources Board, Method 1003, Revision IV, July 2002



## 1.0 Scope and Application

- 1.1 This exhaust gas sampling protocol is specifically designed for sampling dilute exhaust generated from heavy-duty diesel engines being operated over the EPA transient cycle or steady state emission test as described in 40 CFR Part 86.
- 1.2 For transient cycle operation, this official testing protocol involves continual sample integration of all gaseous emissions along with pertinent engine and ambient variables for 1200 seconds (20 minutes). For eight-mode steady state operation, this official testing protocol involves modal sample collection for a total of 20 minutes. Modes 1, 2, 3 and 8 are collected for 3 minutes of the 5 minute mode, while modes 4, 5, 6 and 7 are collected for 2 minutes.
- 1.3 Dilute exhaust samples are collected from the dilution tunnel.

## 2.0 Method Summary

- 2.1 Samples analyzed for aldehyde and ketone compounds (carbonyls) are collected continuously by pulling the dilute exhaust from the dilution tunnel, using a Teflon diaphragm pump, through a series of two DNPH cartridges.
- 2.2 The absorbing solution (2,4-DNPH) complexes the carbonyl compounds into their diphenylhydrazone derivatives.
- 2.3 Once received by the laboratory, cartridges are eluted with 5 mL acetonitrile and analyzed via high performance liquid chromatography (HPLC).

## 3.0 Health and Safety

- 3.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.
- 3.2 All sampling should be done while using proper protective equipment to minimize exposure to vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

## 4.0 Sample Preservation, Containers, Handling, and Storage

- 4.1 Samples are collected and stored in DNPH-impregnated cartridges.
- 4.2 DNPH cartridges must be sealed and refrigerated, at a temperature less than 40° F, upon receipt from manufacturer, until ready for use.
- 4.3 If samples are not analyzed the same day as collected, they must be refrigerated at a temperature below 40° F.
- 4.4 Refrigerated samples are stable for up to 30 days.

**5.0 Interferences and Potential Problems**

5.1 To decrease the chance of background contamination variables and oxygenated impurities, DNPH-impregnated cartridges are used rather than impingers.

**6.0 Equipment/Apparatus**

6.1 Sep-Pak® DNPH-impregnated cartridges: Waters Corporation, or equivalent

6.2 Digital flow meter: Dwyer Instruments, Inc., or equivalent.

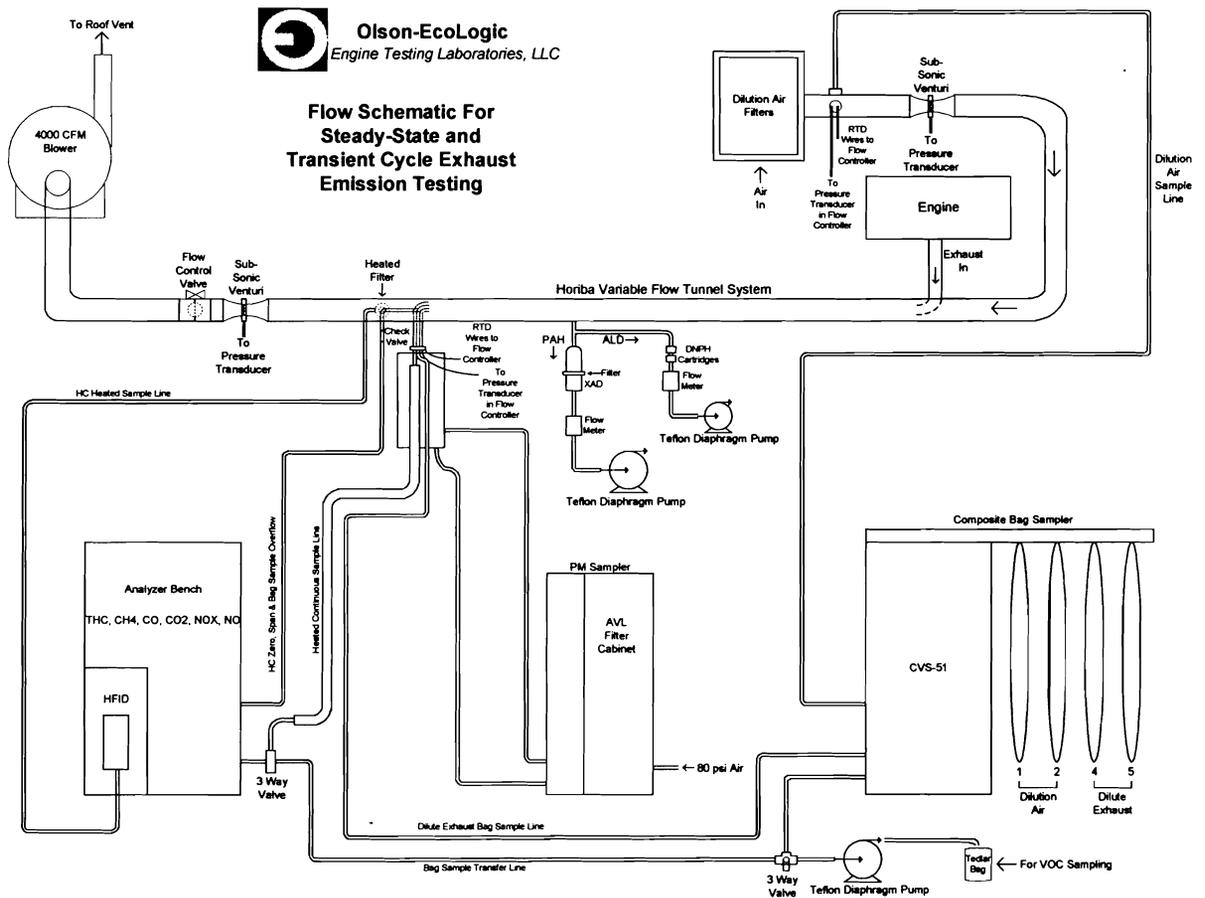
6.3 Teflon diaphragm vacuum-pressure pump: MFG Corp., or equivalent.

6.4 Sampling Train Schematic

6.4.1 8' Gas line from the tunnel to the PAH/ALD sampling system is as follows:

6.4.1.1 Siltek®/Sulfinert® treated ½" 316L grade stainless steel tubing: Restek, or equivalent.

6.4.2 After the "T" connection, all ALD sampling line is ¼" Teflon tubing.



## 7.0 Procedure

### 7.1 Sample collection

7.1.1 Two DNPH cartridges connected into the sample flow path.

7.1.2 Dilute exhaust samples are collected onto the DNPH cartridges by turning on the power to the Teflon diaphragm pump.

7.1.2.1 For transient test cycle sample collection, the Teflon diaphragm pump is turned on for the duration of the 1200 second emission test.

7.1.2.2 For steady state cycle sample collection, the Teflon diaphragm pump is turned on for 3 minutes at the end of modes 1, 2, 3 and 8; and 2 minutes at the end of modes 4, 5, 6 and 7.

7.1.3 The flow rate through the DNPH cartridges should be 1 L/min.

7.1.3.1 Flow rate should be continuously monitored and adjusted if necessary during sampling.

7.1.4 The dilution and work is noted for each dilute exhaust sample taken for final calculations.

7.1.5 DNPH cartridges are refrigerated immediately after sample collection, below a temperature of 40° F, until analyzed by HPLC.

## 8.0 Calculations

8.1 Volumetric flow conversions are as follows:

$$1 \text{ SCFM} = 0.0283 \text{ m}^3/\text{min} = 28.317 \text{ L}/\text{min}$$

## 9.0 Quality Control/Quality Assurance

### 9.1 Tunnel Blank Sample

9.1.1 A tunnel blank sample must be collected each analysis day. If the tunnel blank shows a peak greater than the method detection limit (MDL) in the region of interest, the source of the contamination must be investigated and remedied. Do not subtract the blank value from the sample value.

### 9.2 Field Blank Sample

9.2.1 At least one cartridge per batch is analyzed as a field blank. If the cartridge blank shows a peak greater than the method detection limit in the region of interest, the source of the contamination must be investigated and remedied.

### 9.3 Leak Checks

#### 9.3.1 Sampling Train

9.3.1.1 A leak check must be performed each analysis day to ensure correct sampling system flow rates.

9.3.1.2 To leak check any part of the sampling train, the suspected leak area should be isolated and pressurized or put under vacuum. A gauge may then be used to check if a leak exists.

9.3.1.3 Flow rate must be kept within 0.2 L/min of the set sampling flow rate of 1 L/min.

### 9.4 Flow Rates

9.4.1 The flow rate into the DNPH cartridge is controlled with a digital flow meter. The flow rate over the transient or steady state emission test cycle is 1 L/min.

## 10.0 References

10.1 Desert Research Institute, DRI Carbonyl Sampler, 1-710.3, Revision 03, June 1997.

## 1.0 PURPOSE/APPLICABILITY

This analytical method applies to dinitrophenylhydrazine-impregnated cartridges through which air samples have been passed for the collection of carbonyl compounds. Carbonyl compounds react rapidly with 2,4-dinitrophenylhydrazine (DNPH) in acidic media to form yellow to orange-colored hydrazones. The color depends on the type of carbonyl compound, and the amount of the product formed is dependent on the quantity of carbonyl compound provided to the reagent.

Samples are collected by drawing a known volume of air through the DNPH-impregnated cartridges. These exposed cartridges are then returned to the laboratory for isolation, separation, and quantification of the hydrazone products by high-performance liquid chromatography (HPLC). The ambient air concentration of various carbonyl compounds is determined from the quantity of the associated hydrazones found in the exposed cartridges and the volume of air samples. Typically C<sub>1</sub>-C<sub>6</sub> carbonyl compounds, including benzaldehyde, are measured effectively by this technique, with a detection limit of ~ 0.1 ppbv.

This method follows the procedure described in EPA Method TO-11A (January 1997, EPA/625/R-96/010b).

## 2.0 MATERIALS/APPARATUS

### 2.1 DNPH Cartridges

Waters Sep-Pak XPoSure Aldehyde Samplers are purchased from Waters (WAT047205) and sampled directly from the manufacturer without need for laboratory preparation.

## 3.0 SAMPLE ANALYSIS

Aldehydes collected in the cartridge (as the hydrazones) are eluted with acetonitrile and the eluent is analyzed using reverse phase high performance liquid chromatograph (HPLC) (Fung and Grosjean, 1981). Gradient elution is used with a sufficiently polar mobile phase for the separation of acetone, acrolein, and propanal. The mobile phase polarity is then decreased steadily to allow the elution of the higher aldehydes.

### **3.1 Sample Shipping/Storage**

The cartridges are returned to the laboratory in secondary aluminum envelopes provided by the manufacturing, labeled with unique Project Media Identification (PMI) numbers, in a cooler at 4°C. In the laboratory, they are stored in a refrigerator until analysis. The time between sampling and extraction should not exceed two weeks. Sample elutes are stable at 4 °C for up to one month.

### **3.2 Sample Preparation**

Uncap the cartridge and place it in a small test tube holder. Elute the cartridges with 2 ml of acetonitrile into a volumetric flask and transfer into a septum vial. Cap the vial and write the cartridge number on the side using a permanent marker. If not analyzed the same day, place the extracts in a clean refrigerator.

### **3.3 Standard Solutions**

Prepare stock solutions of the hydrazone standards provided by AccuStandard in acetonitrile at carbonyl concentration of 5 µg/mL.

Dilute the stock standards to obtain working solutions in the range of 0.1 to 10 µg/mL concentrations for most applications. Higher concentrations may be needed occasionally if the air carbonyl concentrations exceed ~20 ppb during sampling. At least three concentrations of working standards bracketing the sample concentrations should be prepared for the calibration.

A secondary standard from Restek is used to verify the calibration.

### **3.5 Instrument Conditions**

The HPLC instrument is a Waters 2695 Alliance Separation Module with a photo diode array (PDA) detector with Empower software. Data are collected between a wavelength range of 190-450 nm.

## Water Alliance 2695 Gradient Acquisition Method for Carbonyls

Solvent A: 100% HPLC Water  
Solvent B: 100% Optima Acetonitrile  
Solvent C: 100% Optima Methanol  
Column: Polaris C18-A, 4.6x15, 3um  
Detector: PDA, 360 nm

Table 1. Gradient elution solvent conditions.

Time (min)	Flow (mL)	%A	%B
0.01	1.50	70.0	30.0
1.00	1.50	70.0	30.0
20.00	1.50	60.0	40.0
32.00	1.50	55.0	45.0
41.00	1.50	37.0	63.0
45.00	1.50	37.0	63.0
50.00	1.50	30.0	70.0
55.00	1.50	70.0	30.0

The column is conditioned with 50:50 water/methanol for 10 minutes at the end of each sequence.

### 3.6 Calibrations

Following the Millennium<sup>32</sup> 3.20 PDA document, five calibration standards are analyzed using concentrations in Table 2 (see Figures 2 for example of calibration curve). The curve is forced through zero and is acceptable with a  $R^2 \geq 0.95$ . A secondary standard from Restek is used to verify the calibration.

Once the linear response factor has been documented, an intermediate concentration standard near the anticipated level of each component (but at least ten times the detection limit) is used for a daily calibration check standard. A sample chromatogram is shown in Figure 1.

### 3.7 Data Acquisition and Processing

The calibration equation below is used to determine the amount of carbonyls in the samples (this step is performed by the HPLC baseline data system).

Table 2. Calibration Levels

	Level 1	Level 2	Level 3	Level 4	Level 5
Conc. (ug/mL)	1	0.5	0.25	0.1	0.025
Formaldehyde	7.036	3.518	1.759	0.703	0.351
Acetaldehyde	5.13	2.565	1.282	0.513	0.256
Acetone	4.1	2.05	1.025	0.41	0.205
Acrolein	4.366	2.183	1.091	0.436	0.218
Propionaldehyde	4.113	2.056	1.028	0.411	0.2055
Crotonaldehyde	3.57	1.785	0.892	0.357	0.178
2-Butanone (MEK)	3.5	1.75	0.875	0.35	0.175
Methacrolein	3.57	1.785	0.892	0.357	0.178
n-Butyraldehyde	3.51	1.755	0.877	0.351	0.1755
Benzaldehyde	2.7	1.35	0.675	0.27	0.135
Valeraldehyde	3.122	1.561	0.78	0.312	0.156
Glyoxal	0.92	0.46	0.23	0.046	0.023
m-Tolualdehyde	2.506	1.253	0.626	0.25	0.125
Hexaldehyde	3.116	1.558	0.779	0.311	0.155

### 3.8 Calculations

$$C_A = \frac{W_d}{V_m \text{ (or } V_s)} \times 1000$$

where:

$C_A$  = concentration of analyte (ng/L) in the original sample  
 $W_d$  = total quantity of analyte ( $\mu\text{g}$ ) in sample, blank corrected

$$W_d = W \times V_E$$

where:

$W$  = Concentration of analyte in the cartridge ( $\mu\text{g}$ )  
 $V_E$  = final volume (ml) of sample extract  
 $V_m$  = total sample volume (L) under ambient conditions  
 $V_s$  = total sample volume (L) at 25 °C and 760 mm Hg

The analyte concentrations can be converted to ppbv using the following equation:

$$C_A \text{ (ppbv)} = C_A \text{ (ng/L)} \times \frac{24.4}{MW_A}$$

where:

$C_A$ (ppbv) = concentration of analyte in parts per billion by volume  
 $C_A$ (ng/L) is calculated using  $V_s$

$MW_A$  = molecular weight of analyte.

### 3.9 Quality Control

After calibration, an intermediate concentration calibration check standard is analyzed every 10 samples with a  $\pm 10\%$  recovery.

#### 3.9.1 Blanks

At least one field blank or 10% of the field samples, whichever is larger, should be shipped and analyzed with each group of samples. The field blank is treated identically to the samples except that no air is drawn through the cartridge.

Instrument blanks are analyzed after high concentrations.

#### 3.9.2 Method Precision and Accuracy

10% of the samples are analyzed twice for replicate precision which typically falls within  $\pm 10\%$ .

### 4.0 REFERENCES

Fung, K., and D. Grosjean (1981). "Determination of Nanogram Amounts of Carbonyls as 2,4, dinitrophenylhydrazones by High Performance Liquid Chromatography." *Analy. Chem.*, 53, 168.

U.S. EPA, "Method TO-11A: Determination of Formaldehyde in Ambient Air Using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC) [Active Sampling Methodology]," EPA/625/R-96/010b, in *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*.

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DRI STANDARD OPERATING PROCEDURE

Page: 6 of 9

Date: 4/11/2005

Title: Analysis of Carbonyl Compounds  
by High Performance Liquid Chromatography

Number: 2-710.4

Revision: 04

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Winberry, W.T., Jr., N.T. Murphy, and R.M. Riggan (1988). *Method TO11 in Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*. EPA/600/4-89/017, U.S. Environmental Protection Agency, Research Triangle Park, NC.



# Sample Report

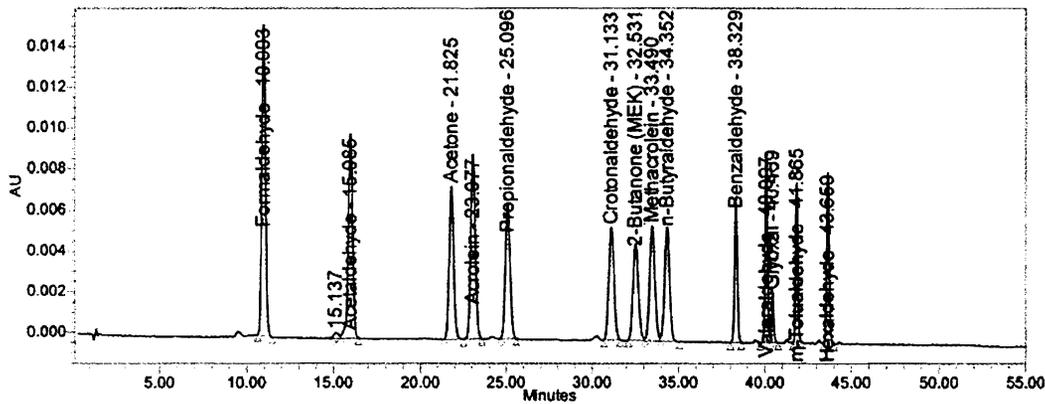
Reported by User: Katarzy na Rempala Project Name: API\_Winter2004

# of Results: 5

## SAMPLE INFORMATION

Sample Name: 0404lev el2  
Sample Type: Standard  
Vial: 3  
Injection #: 1  
Injection Volume: 20.00 ul  
Run Time: 55.0 Minutes  
Sample Set Name: API\_040505

Acquired By: Katy  
Date Acquired: 4/6/2005 3:56:28 PM  
Acq. Method Set: Polaris\_3um\_MS  
Date Processed: 4/7/2005 11:09:16 AM  
Processing Method: Polaris0405PM  
Channel Name: Extract 360.0  
Proc. Chnl. Descr: PDA 360.0 nm



	Peak Name	RT	Area	Amount	Units	PDA Match1 Spect. Name	Match1	PDA Match2 Spect. Name	Match2
1	Formaldehy de	10.993	233572	3.518	ug/ml	Formaldehy de	0.851		
2	Acetaldehy de	15.985	176681	2.565	ug/ml	Acetaldehy de	5.705	n-Buty raldehy de	5.709
3	Acetone	21.825	137633	2.050	ug/ml	Acetone	1.322	2-Butanone (MEK)	1.870
4	Acrolein	23.077	167748	2.183	ug/ml	Acrolein	1.085	Methacrolein	3.072
5	Propionaldehy de	25.096	129540	2.056	ug/ml	n-Buty raldehy de	1.448	Acetaldehy de	1.653
6	Crotonaldehy de	31.133	114907	1.785	ug/ml	Crotonaldehy de	1.065	Methacrolein	6.077
7	2-Butanone (MEK)	32.531	102244	1.750	ug/ml	2-Butanone (MEK)	1.621	Acetone	2.927
8	Methacrolein	33.490	122150	1.785	ug/ml	Methacrolein	3.741	Acrolein	5.489
9	n-Buty raldehy de	34.352	112621	1.755	ug/ml	n-Buty raldehy de	3.275	Valeraldehy de	3.330
10	Benzaldehy de	38.329	82427	1.350	ug/ml	Benzaldehy de	1.003	m-Tolualdehy de	3.987

Figure 1a. Chromatograph of Level 2 standard.



## Sample Report

Reported by User: Katarzy na Rempala Project Name: API\_Winter2004

# of Results: 5

	Peak Name	RT	Area	Amount	Units	PDA Match1 Spect. Name	Match1	PDA Match2 Spect. Name	Match2
11	Valeraldehy de	40.097	97770	1.561	ug/ml	Valeraldehy de	5.956	n-Buty raldehy de	6.388
12	Gly oxal	40.469	21352	0.460	ug/ml				
13	m-Tolualdehy de	41.865	70294	1.253	ug/ml	m-Tolualdehy de	0.975	Benzaldehy de	3.809
14	Hexaldehy de	43.659	84775	1.558	ug/ml	Hexaldehy de	1.304	Valeraldehy de	1.531

Figure 1b. Chromatograph of Level 2 standard.

**Empower**

LC Calibration Report ver1

Reported by User: Katarzyna Rempala (Katy)

Project Name: API\_Winter2004

Processing Method: **Polaris0405PM**

Project Name: API\_Winter2004

Processing Method ID 1175

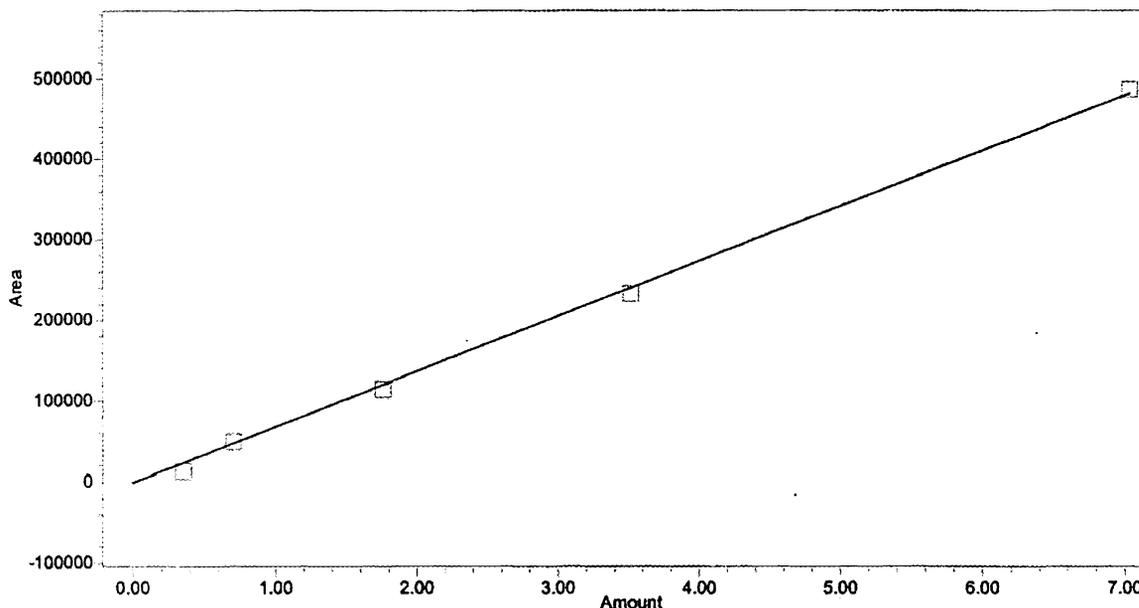
System: W2695

Calibration ID: 1159

Channel: Extract 360.0

Date Calibrated: **4/7/2005 11:13:37 AM**

Proc. Chnl. Descr. PDA 360.0 nm



Name: Formaldehyde; RT: 11.360; Fit Type: Linear thru Zero; Cal Curve Id: 1160; R: 0.999224; R<sup>2</sup>: 0.998448; Weighting: None; Equation: Y = 6.84e+004 X

Figure 2. Calibration curve for formaldehyde.

---

**Attachment 9**

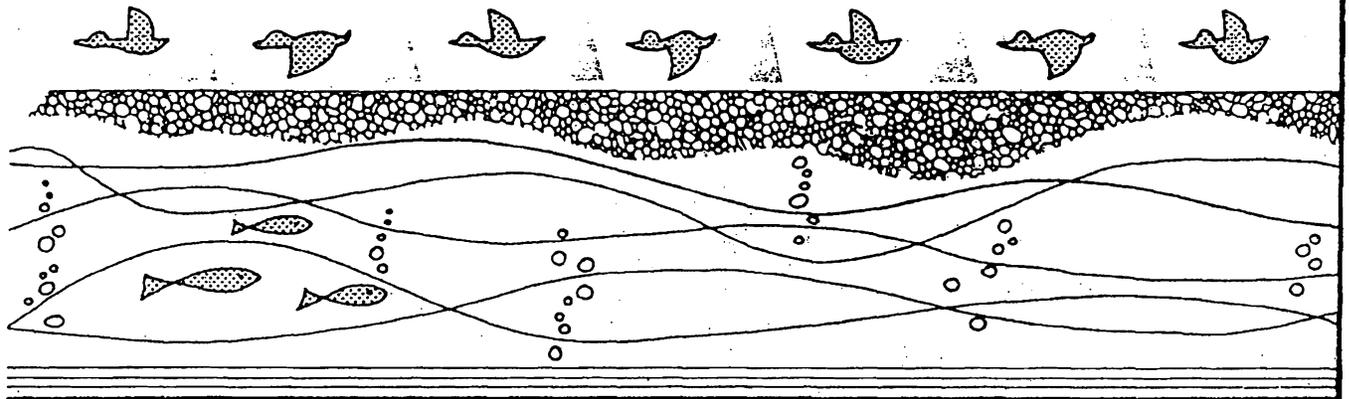
**Oil Spill Recovery Using Elastol, Report 1**



Environment  
Canada

Environnement  
Canada

# SPILL TECHNOLOGY NEWSLETTER



An informal quarterly newsletter published by the  
Technology Development and Technical Services Branch  
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The Spill Technology Newsletter was started with modest intentions in 1976 to provide a forum for the exchange of information on spill countermeasures and other related matters. We now have over 2000 subscribers in over 40 countries.

To broaden the scope of this newsletter, and to provide more information on industry and foreign activities in the field of spill control and prevention, readers are encouraged to submit articles on their work and views in this area.

## INTRODUCTION

The first article of this issue is by Rick McKelvey of the Canadian Wildlife Service. Rick summarizes the oiled-bird problem and gives details on how to build a portable hot-water system for cleaning birds. The second article summarizes spill statistics for Atlantic Canada over the past fourteen years. The trends and significant occurrences are reviewed.

The third article is by Merv Fingas and Ed Tennyson who review their joint U.S. and Canadian project to evaluate Elastol and Demoussifier, two new spill treating agents. Both agents functioned well over a series of tests ranging from laboratory to large field scale.

## EVALUATION OF TWO NEW OIL SPILL CHEMICAL ADDITIVES: ELASTOL AND DEMOUSSIFIER

Submitted by: Merv Fingas  
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### Introduction

A new oil spill treating agent, Elastol, has been developed for enhancing the recovery potential of oil. When added to oil, the powder renders oil visco-elastic making it adhesive to oil spill recovery equipment. Elastol is composed of a non-toxic polymer, polyisobutylene and is hydrophobic and not water soluble. A major study was undertaken jointly by the U.S. Minerals Management Service and Environment Canada to evaluate this new spill additive. Laboratory testing was done and studies were conducted in large-scale test tanks and in a major field exercise off Canada's east coast.

At the same time, another new spill treating agent, demoussifier, was tested in large outdoor tanks and at sea. This product, which also consists of a mixture of long-chain polymers which have no measurable toxicity to humans or to aquatic life, was developed at Environment Canada's River Road Labs. The product breaks up water-in-oil emulsions and prevents their formation.

### Laboratory Testing of Elastol

The laboratory work on Elastol involved several different tests. The effect on a suite of different oils was determined by measuring the time to initiate change and the degree of elasticity formed. These oils included: Prudhoe Bay, Alberta Sweet Mix Blend, Norman Wells, Bent Horn, Hibernia, Tarsiut, Atkinson, Amauligak crudes, diesel fuel and a Bunker C mix. All oils displayed viscoelastic properties when treated with doses of 600 to 6000 ppm Elastol. In general, more viscous oils tended to attain a higher degree of elasticity than non-viscous oils, but did so over a longer period of time. No simple correlation could be established between an oil property and Elastol effectiveness. Elastol effectiveness is enhanced by mixing and by higher temperatures, although the latter may be the effect of decreasing oil viscosity.

Under low mixing energy conditions, oils exhibited some degree of elasticity within 15 minutes of Elastol application. A high degree of elasticity was not observed until after one hour. Less viscous oils took less time to reach maximum elasticity and viscous oils more time. If left to weather, Elastol-treated oil became more elastic with the increasing viscosity of the oil. In fact, some samples left for 30-day periods became elastic as rubber bands sold for stationery purposes. This effect has been ascribed to the effect of the increasing viscosity of the oil with weathering (evaporation) and not the progressive reaction of the Elastol.

Elastol causes a minor reduction in the rate of oil evaporation, but not significant enough to reduce its flash point. Elastol reduces slick spreading to a limited degree, especially at high concentrations. This effect, about 20%, is not believed to have a significant useful benefit by itself in real applications. When Elastol is applied in very large doses, >1%, the slick would actually contract somewhat, but again, the effect would not be beneficial in a field situation.

The addition of Elastol either had no effect or an inhibiting effect on the formation of water-in-oil emulsions, except in the case of the Armauligak and Tarsiut oils from the Beaufort Sea region. In two cases, the application of Elastol to emulsified oil actually led to measurable de-emulsification. Application of Elastol to stable water-in-oil emulsions sometimes had little effect. Testing with commercial de-emulsifiers and the Environment Canada "demoussifier", showed that Elastol had no effect on the operation of these chemicals and that they could be used together.

Elastol reduces chemical dispersant effectiveness by as much as one order of magnitude. Elastol also reduces natural dispersion of oil into water by as much as three orders of magnitude. This property, while superficially appearing negative, is actually quite useful. If Elastol was used in situations where the aquatic life is very sensitive and important, it could reduce water concentrations of the oil in the water to threshold levels.

Elasticity was measured using a die swell apparatus in which oil is pushed through a small opening and the fluid responds by swelling to a size corresponding to its elasticity. This is measured by photographing the swell, measuring it with a vernier caliper and comparing untreated versus treated oil to yield a ratio which is described as "elasticity" in this paper. The instrument displayed good sensitivity to polymer concentration and to the degree of observed elasticity. This instrument could also be used in field conditions and is relatively insensitive to debris and water in the oil.

#### Tank Scale Testing of Elastol and Demoussifier

An application device was developed for each of the two products, as commercial devices do not exist for delivering treatments at the low ratios required. Elastol would be tested at 500 to 5000 ppm and demoussifier would be tested at 150 to 2000 ppm. A search of commercial devices revealed that nothing suitable was available off-the-shelf but that sandblaster-type equipment could be satisfactorily modified. A commercial blaster (Sears) was modified so that it could spray low quantities. One modification was necessary for the solid Elastol, and another for the liquid demoussifier. The modified applicator was tested on each product to ensure that uniform spacial distribution was achieved and that application rates could be controlled over the necessary range by adjusting the air pressure when applying the product from a boat travelling at approximately 3 knots. A series of test tank runs were performed to ensure that results obtained previously with hand distribution techniques and with pre-mixing were duplicated with the new applicators. Success was achieved in all cases, and no detrimental effects were observed during application of either product, such as herding and other phenomena that have decreased the field effectiveness of dispersants so dramatically (Bobra et al., 1988).

Part of this study involved large-scale tank testing of both products using the Esso tank in Calgary, Alberta. The tank measures 15 x 19 m with a depth of 0.8 to 2 m. Two test days were devoted to demoussifier and two to Elastol. Testing was performed in two boomed areas inside the tank. This permitted the simultaneous testing of a control and a treated slick under identical conditions. The demoussifier prevented the formation of water-in-oil emulsions on both test days and did so at ratios as low as 1:2000 (500 ppm).

Elastol was added to a test crude oil at 4000 ppm and the test slick was released several hours later when the oil was highly elastic. Despite this high elasticity, the oil was not thick enough to burn. The oil was recovered by a rotating disk skimmer and the effect of Elastol was to increase the recovery rate of this unit significantly. In fact, the pump could not keep pace with all the oil being recovered. On the fourth day of testing, crude oil was treated with 2000 ppm of Elastol and recovered with a skimmer. The recovery rate was again high and exceeded the capacity of the pump to remove it. On this particular day, the oil in the untreated boom had formed an emulsion. This was treated with demoussifier as was the Elastol-treated slick. The demoussifier broke the emulsion in the untreated slick and no emulsion formed in the treated slick, nor were any other effects noted. During the first two trial days, the use of demoussifier reduced the effectiveness of the recovery operation significantly. It was concluded, therefore, that on a preliminary basis, demoussifier and Elastol could be used together to enhance recovery and eliminate emulsion.

The tank scale tests showed that there were no scaling effects for either the Elastol or the demoussifier. Both products worked well for the intended purpose. Elastol increased the visco-elasticity of the oil and greatly increased the recovery by the oil skimmer. Elastol, however, did not reduce the spreading or increase the thickness of the slick sufficiently to allow in-situ burning. Demoussifier prevented the formation of water-in-oil emulsion and also broke emulsion already formed. Although demoussifier causes the oil to be less adhesive and lowers the recovery rate of skimmers, the two products can be applied together to achieve positive results.

### Large-scale Field Testing

The tests conducted in the tank were repeated on five-barrel slicks during a field trial 50 miles offshore of Nova Scotia (Seakem, 1988). Five slicks were laid for each of the products and each product was tested both premixed and by application-at-sea, to confirm that application effects were not a factor. The treatments and results of the trial are summarized in Table 1.

The demoussifier trials were performed by laying down a five-barrel oil slick, treating it with the product at the specified ratio, taking samples at subsequent intervals and measuring the water content and the viscosity. One slick was left untreated throughout as a control and another slick was left to form mousse (water-in-oil emulsion) and then treated at the 240-minute interval to test the demoussifier's ability to break emulsion at sea. As can be seen by dramatic reduction in viscosity (105 000 to 22 600 cSt; 1050 to 226 cm<sup>2</sup>/s) over the 30-minute period between samples, the product worked well in breaking up the emulsion.

The product also worked well over the five-hour test period to prevent the formation of emulsions. This is illustrated in Figure 1 which also shows that there is a

TABLE I TREATMENTS AND RESULTS OF TRIALS

Trials	Slick	Treatment (ppm)	Sample 1				Sample 2					
			Time (min.)	Viscosity (cSt)*	Water Content	Elasticity	Comments	Time (min.)	Viscosity (cSt)*	Water Content	Elasticity	Comments
Demoussifier	1	1000	60	10 000	84%			300	84 250	90%		No mousse noted
	2	250	60	2 700	54%			300	62 250	93%		No mousse noted
	3	control	60	6 350	88%			270	320 000	95%		Heavy mousse
	4	post-4000	60	2 200	72%			pre-290 post-270	105 000 22 600	90% 78%		Heavy mousse Moderate mousse Treatment broke mousse
	5	pre-1000	15	970	32%			280	38 500	80%		No mousse formed
Elastol	6	3000	130	29 300		1.33	Moderately elastic	280	300 000		1.35	Highly elastic
	7	1000	145	32 250		1.28	Low elasticity	280	228 000		1.33	Moderately elastic
	8	control	135	187 000		0.99	No elasticity, widespread	290	242 000		0.99	No elasticity, widespread
	9	9000	120	93 000		1.99	High elasticity	330	696 000		2.63	Super elastic
	10	pre-3000	115	170 500		1.35	Moderate elasticity	315	156 000		1.57	Highly elastic

\* 1 cSt =  $1 \times 10^{-2}$  cm<sup>2</sup>/s

strong correlation between the viscosity and the amount of treatment. The greater the treatment, the less the viscosity, because of the lesser water content. The water content was universally high, even in those slicks that visibly did not form water-in-oil emulsions. Although water content is indicative of the formation of water-in-oil emulsification, the stability of the emulsion would have to be determined because the unstable emulsions lost water slowly. The water content of the slicks is interesting in that all the slicks laid over the two day test period rapidly took up water, including those slicks that were treated with Elastol. This was noted despite the fact that the oil viscosity was higher, although not as high as that expected from an emulsion, and the oil did not have the appearance of an emulsion. The appearance of the unemulsified oil is also significant, the water droplets were often of sufficient size to be seen. An emulsion is reddish-brown in colour, has a high viscosity and the water droplets are too small to be seen.

The Elastol tests were performed in an analogous manner, with one control slick laid and one slick being pretreated to test the effect of at-sea treatment. The slicks were sampled periodically, and both viscosity and elasticity were measured immediately on board the ship.

The high elasticity of the treated slicks was significantly higher than that of the untreated slicks and corresponded to that experienced in the laboratory, in fact, as shown in Figure 2, it actually exceeded laboratory results at the higher doses. This unexpected result is probably due to the better mixing achieved in the field situation. Interestingly, the dose and elasticity in the field appear to be linear, a phenomenon that had not been noted previously.

The elasticity of the oil was sufficient to cause stringing of the product when samples were recovered. This is indicative of a very high state of elasticity and would result in high oil recovery rates if a skimmer was used. The elasticity appeared to be uniform throughout the slicks despite the typical uneven distribution of treating agent at sea.

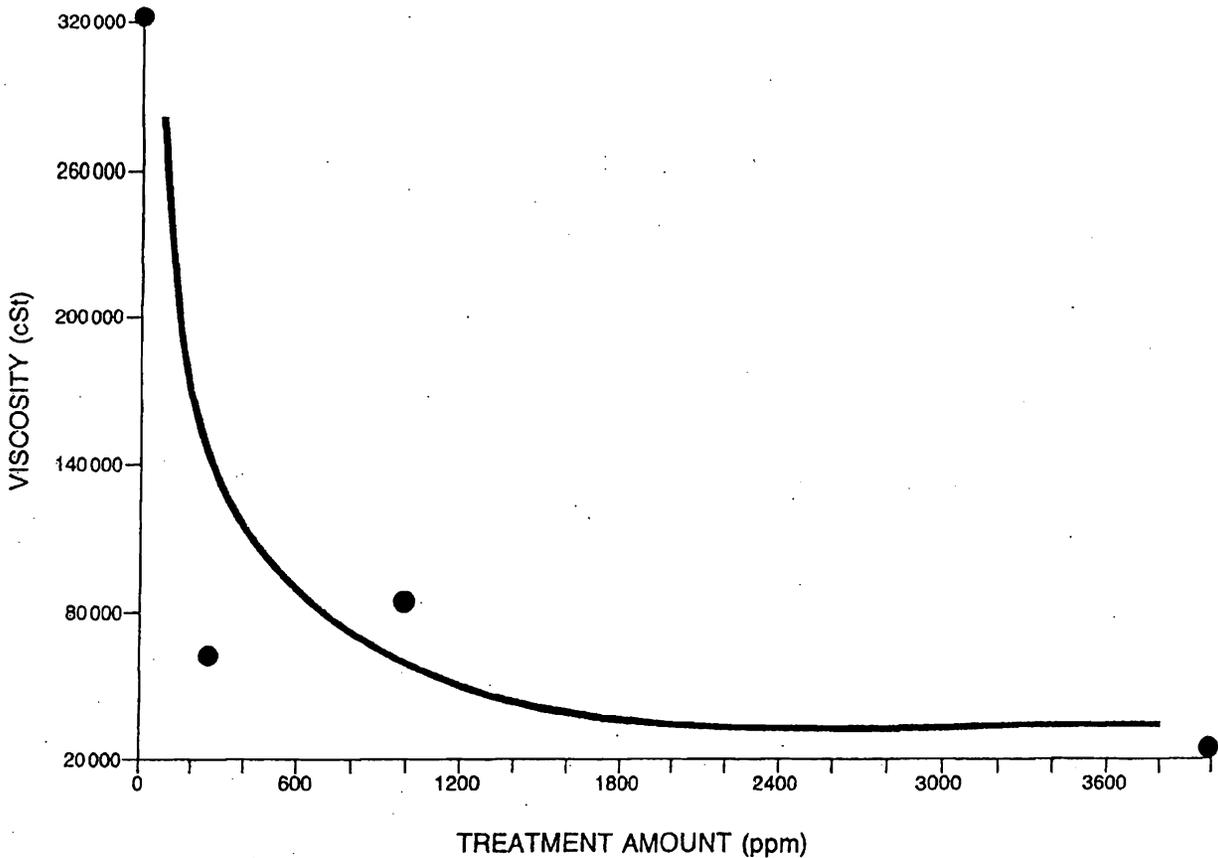


FIGURE 1 THE EFFECT OF DEMOUSSIFIER APPLICATION ON VISCOSITY

The slicks were monitored by a remote sensing aircraft, but the analysis of slick areas was not complete at the time of writing. Slicks treated with Elastol, however, appeared to be smaller to shipboard observers and the size of the slick appeared to correlate well with the amount of Elastol. In fact, one was able to distinguish slicks by their size, with the 9 000-ppm-treated slick being the smallest.

#### Summary and Conclusions

1. Elastol functioned well in the laboratory, test tank and in field situations; it caused oil to become viscoelastic in all applications.
2. Elastol is able to float with and mix with oil so that application is not critical as it is with dispersants.
3. Demoussifier has the same application insensitivity as Elastol.

4. The effects of Elastol improves oil skimmer recovery.
5. Elastol retards slick spreading; however, this effect, for physical reasons, is not sufficient for countermeasures purposes such as in-situ burning of oil on water.
6. The demoussifier prevented emulsion in the test slicks over the five-hour test period.
7. The demoussifier broke water-in-oil emulsions in 10 to 15 seconds after application.
8. Results of field application such as herding and loss of effectiveness, seen with dispersants, were not noted at all with either product.
9. Water content is not a good indicator of mousse formation as all slicks at the offshore trial accumulated a large amount of water. Stable mousse formation is indicated by a stable water content, small water droplet size, red colouring and a very high viscosity.

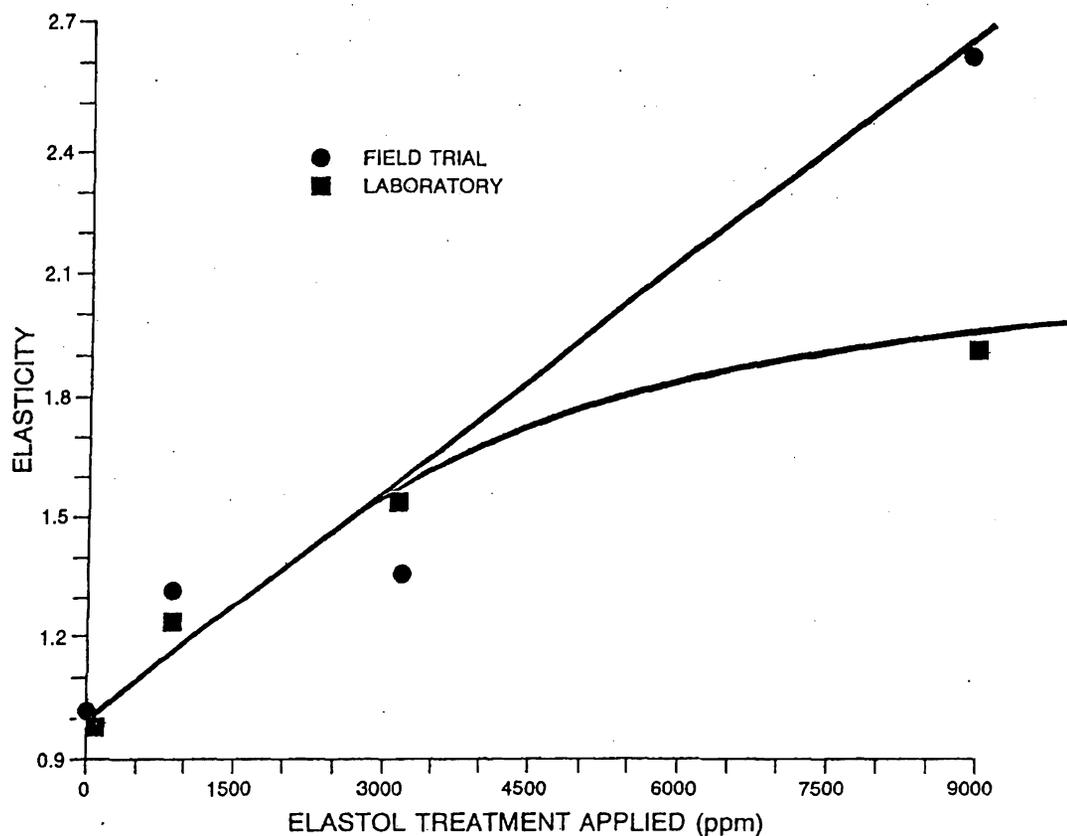


FIGURE 2 ELASTICITY OF OILS AFTER ELASTOL TREATMENT

**References**

Bobra, M.A., P.I. Kawamura, M. Fingas and D. Velicogna, Laboratory and Tank Test Evaluation of Elastol, Environment Canada Manuscript Report Number EE-94, Ottawa, Ontario (1987).

Bobra, M.A., P.I. Kawamura, M. Fingas and D. Velicogna, Mesoscale Application and Testing of and Oil Spill Demulsifying Agent and Elastol, Environment Canada Manuscript Report Number EE-104, Ottawa, Ontario (1988).

Seakem Oceanography, Field Test of Two Spill Treating Agents, Environment Canada, Manuscript Report Number EE-XX, in press (1988).

**Attachment 10**

**Oil Spill Recovery Using Elastol, Report 2**



**MEMORANDUM**

**TO:** All  
**FROM:** Al Hadermann  
**DATE:** July 7, 1992  
**SUBJECT:** Elastol and Corals and Seagrasses (with footnote)

Anitra Thorhaug\* has tested Elastol powder and various weathered oils for toxicity to seagrasses and corals. Tests were conducted on species in 50 gallon glass tanks containing 100,000 ml of seawater. Tests were run at 0.00125%, 0.0075% and 0.01235% oil, i.e., approximately 1.25 ml, 7.5 ml and 12.5 ml. When Elastol was added it was used at 10 mg, or 0.010 g. This corresponds to treatment concentrations of approximately 8,000, 1,333 and 800 ppm for 1.25 ml, 7.5 ml, and 12.5 ml of oil, respectively. Although the Elastol was not used as a slurry, and overtreatment obviously occurred at the 12.5 ppm oil level, the results showed that Elastol treatment reduced the toxicity of the oil.

The exposure times to oil and oil and Elastol were 6 and 10 hours for coral and seagrasses, and 10 hours for mangroves. Agitation was provided by air bubbling. The seawater was replaced after the incubation (exposure) time.

Dr. Thorhaug reported that:

"The corals continued with their polyp feeding patterns with the Elastol-oil mixture in the tank. This was an extraordinary result compared to the other compounds used in the test which were a variety of dispersed-oil products."

"The quantitative data for the various concentrations and various oils show that the oil plus the Elastol at concentrations at 125 ppm were appreciably less toxic than oil alone for corals and seagrasses."

Since oil alone at 125 ppm did not affect mangroves, no basis for improvement with Elastol was seen.

In Summary, Dr. Thorhaug states:

"The toxicity of oil alone was definitely greater than the Elastol treatments in almost all the concentrations tested."

\*Anitra Thorhaug is a researcher at Florida International University, P.O. Box 490559, Miami, FL 33149.

ELASTOSOL, an oil spill control agent: Toxicity effects on tropical and subtropical seagrass, corals, fish and mangroves.

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The toxicity of other than dispersant oil spill control agents (solidifiers, gels, sorbent and bioremediation agents) for cleaning oil spills has received very little attention (NRC, 1989; Thorhaug, 1991; IMO, 1991). This is the beginning of an attempt to give the same attention to the toxicity of these products to critical tropical/subtropical matrix organisms and fisheries species.

The product elastosol contains polyisobutylene and an oleophilic surfactant. Although it is proprietary, it has undergone chemical analysis and field testing (Fingas, 1989; Bobras, 1987a&b, 1988a&b; Fingas and Tennyson, 1988). A wide variety of crude oils became elasticized within 15 minutes more-or-less after elastosol treatment. These were retained in solidified condition up to 30 days with viscoelastic properties maintained over this time period. The properties appear to be that slicks are prevented from spreading and can be picked up by skimmers efficiently. With sensitive ecosystems, the major use of elastosol clearly will be relatively small confined spills probably at ports, marinas, oil refineries or in bays or estuaries. Thus, the tolerance of critical habitat organisms to elastosol is important.

#### METHODS:

##### Toxicity studies

Laboratory investigations. Standardized procedures used by the U.S. Environmental Protection Agency (EPA) and various agencies (FAO, UNESCO, UNEP) for testing tropical matrix organisms were employed so that standardized test results could be produced. The hope was to compare results with temperate results, along with UNEP European dispersed-oil toxicity data.

Table 1. Elastosol concentrations versus toxicity of seagrasses, corals and mangroves. 7 to 12 specimens at each concentration per treatment.

SPECIES	TIME IN HRS	CONC OIL	% MORTAL.		OIL ALONE	OIL TYPE	CONC. OIL	% MORTAL.		CONC. OIL	% MORTAL.		% MORTAL. OIL ALONE	
			W/ELAST.	CONTROL				CONTROL	ELAST		CONTROL	ELAST		
<b>SEAGRASS</b>														
Thal. test.	4	125	15	11	20	SW	75	10	7	10	12.5	0	0	10
Hal. wri.	4	125	44	9	22	SW	75	11	8	15	12.5	0	0	12
Syr. fil.	4	125	12	10	34	SW	75	12	4	18	12.5	0	0	14
<b>CORAL</b>														
Por. por.	4	125	15	0	52	SW	75	0	0	12	12.5	0	0	0
Mont. ann.	4	125	0	0	32	SW	75	0	0	0	12.5	0	0	0
Acrop.	4	125	0	29	100	SW	75	0	0	0	12.5	0	0	0
<b>MANGROVE</b>														
Rhizi.	10	125	0	0	0	SW	75	0	0	0	12.5	0	0	0
Avicenn	10	125	0	0	0	SW	75	0	0	0	12.5	0	0	0
Lang.	10	125	0	0	0	SW	75	0	0	0	12.5	0	0	0

Thal. test. = *Thalassia testudinum*; Hal. wri = *Halodule wrightii*; Syr. fil. = *Syringodium filiforme*; Por. por. = *Porolithes porolithes*; Mont. ann. = *Montastrea annularis*; Acrop. = *Acropora palmata*; Rhizi. = *Rhizophora mangle*; Avicenn. = *Avicennia*; Lang. = *Languncularia*

Selection of critical matrix organisms for tropical shorelines was a choice based on (1) importance to the food web, (2) rate and difficulty of replacement once decimated, (3) shoreline stabilization characteristics, and (4) usefulness to the nation (fisheries, tourism). Local experts such as Jeremy Woodley, director of the Discovery Bay Marine Laboratory and preeminent Jamaican coral reef expert, were asked to choose indicator species for the project.

Standardization among techniques for various groups was attempted so that the results of each group could be compared. Thus, the standardized portion will be explained first.

**Laboratory conditions.** The procedure was described in detail by Thorhaug and Marcus (1985). Fifty-gallon glass aquaria were used in out-of-door conditions (such as fluctuating light, temperatures, and running water) and, except for the mangroves and seagrasses, were protected from rain and wind. Temperatures ranged from 26° to 30°C, with fluctuation of 1°C ± 0.5°C. Seawater ranged from 30‰ to 33‰ (except for the mangroves). In seagrasses, three inches of beach sand was at the bottom. Corals tanks were bare. For seagrasses, a minimum of 15 blade groups of each species was placed in each tank (1 tank per treatment). Great care was taken in transport and holding conditions for the coral and mangrove specimens. Time for equilibration of specimens was allowed. 100,000 cc of seawater was placed in each treatment.

**Laboratory procedures.** Oil-only was a treatment in each set. Time for weathering of oil was 24 hours at 28°C in shallow plastic pans. The control tank was handled exactly the same as treatments. The dispersant was applied to oil floating on the tank surface with 1 minute of vigorous stirring. The dispersed oil formed a brown cloud of free-floating material, visually homogeneous throughout the tank (depended somewhat on concentration and dispersant). The coral and seagrasses were not touching the oil floating on the top; the mangroves were in contact on the bark. All specimens were in contact with the dispersant mousse.

The application of elastosol was 12.5 ppm oil, 75 ppm oil and 125 ppm oil with 10 mg (1 package) of elastosol. The exposure times were 6 and 10 hours for corals and seagrasses, 10 hours for mangroves. The treatment was immediately removed, and the water cleaned and changed in all treatments.

The specimens were incubated up to 14 days (coral, seagrass) and 9 months (mangroves). The observation period included daily or bi-weekly readings of health. Seagrasses had young green blades tagged and numbered. These were graded for discoloration, spotting, wilting and other morbidly symptoms. Corals were graded by bleaching, spotting, browning, wilted polyps, extruded polyps, and mucous secretion. Mangroves were graded by leaf drop, twig drop, root death and discoloration, leaf coloration, leaf spots, and leaf wilt. These symptoms of mortality and illness were made quantitative by experts and organisms were graded. The tables showing the results are not of health but of mortality.

Morphologically, the results of elastosol included the observation that the oil flowing on the surface of the tanks within a few minutes of treatment with elastosol congealed into long lines. There appeared to be no film on the glass sides of the aquaria or on the water. The mixture of elastosol and oil was complete.

There were a series of biological and morphological observations.

- 1) The corals continued with their polyp feeding patterns with the elastosol-oil mixture in the tank. This was an extraordinary result compared to the other compounds used in the test which were a variety of dispersed-oil products. Color and vigor of the polyps of all species was retained.
- 2) The elastosol had no apparent effect on seagrass or mangrove vigor or color.

The quantitative data for the various concentrations and various oils show that the oil plus the elastosol at concentrations of 125 ppm were appreciably less toxic than oil alone for corals and seagrasses. Mangroves had no effect from elastosol. Oil alone did not affect mangroves at these concentrations. Corals were the most sensitive to oil. The response of decreased toxicity by application of elastosol is seen dramatically at 125 ppm oil in corals where the control had a higher mortality than the oil plus the elastosol. The mangroves were far more tolerant of oil, plus oil and clean-up product of any kind than were seagrasses or corals.

#### CONCLUSIONS:

The herder or solidifier Elastosol does not appear to have marked toxic effects on the tropical/subtropical matrix organisms studied, even at high concentrations. Oil alone at the higher concentrations does have a toxic effect on seagrasses and corals. The results of dispersants at these same concentrations were highly toxic on the seagrasses, corals and mangroves for many of the products tested.

The elastosol in all tests was the least toxic of a series of oil spill clean-up formulations. The toxicity of oil alone was definitely greater than the elastosol treatments in almost all the concentrations tested.

The difficulty of application of elastosol would indicate use for a smaller spill such as a harbor or estuarine spills. The fact of very low toxicity corals would make this extremely well-adapted to spills near or over coral reefs. It may also be very helpful to control oil when spills are within mangrove swamps. Certainly, spills which cannot be mechanically handled or chemically dispersed may warrant the expense and application methods for elastosol.

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**Attachment 11**

**Oil Spill Recovery Using Elastol, Report 3**



## The 1987 Newfoundland Oil Spill Experiment

### An Overview

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### Abstract

A joint Canadian-United States exercise involving the intentional spilling of approximately 20,000 gallons of specially-treated crude oil was conducted off Newfoundland in September 1987 to evaluate the containment and recovery capabilities of three state-of-the-art booms and skimmers. As part of the exercise, data were collected on a specially-instrumented oil spill boom in an attempt to verify a proposed performance test procedure for open-ocean oil spill booms. A visco-elastic chemical additive was used, after the equipment evaluation was completed to enhance recovery operations. Additional observations were made on the persistence of spilled oil slicks in advanced sea states. The containment and recovery effort was one of the most successful on record and was conducted in winds and sea states commonly thought to be beyond existing capabilities.

### Introduction

An intentional oil spill of 18,000 U.S. gallons was conducted on September 24, 1987, offshore of St. John's Newfoundland to evaluate the containment and recovery capability of three booms and skimmers. The spill also provided an opportunity to verify a nonpolluting performance evaluation procedure for offshore oil containment booms. The spill was conducted approximately 25 nautical miles east of St. John's. Ocean dumping permit requirements included SSW currents and westerly winds to minimize chance of shoreline contact; water depths of at least 100 meters; the site had to be at least 25 nautical miles from shore, and the area had to be within 2 to 3 hours steaming from St. John's. The center of the area selected was 47 degrees, 40 minutes North and 52 degrees, 03 minutes West.

A crude similar to the typical high wax Grand Banks crude was unavailable. Brent crude from the North Sea was allegedly treated by adding 1 percent slack wax by volume to yield an oil of similar physical properties to the Grand Banks crudes. The modified oil was to have a density of 839.8 kg/m<sup>3</sup> and a viscosity of 20 m Pas at 12 degrees C (Ross, 1987a). Recent information indicates that insufficient slack wax was added to reach the 1 percent by volume target level.

Meteorological conditions were recorded on the Canadian Coast Guard (CCG) Cutter "Grenfell" at 15 minute intervals. These include corrected wind velocities and air and water temperatures.

A wave rider was deployed at the test site but failed to function during the exercise. Consequently, sea conditions were estimated, with reasonable agreement, by various trained observers.

The test plan called for the deployment of three booms as follows: A 250 meter length of the specially instrumented Oil and Hazardous Material Simulated Environmental Test Tank (OHMSEIT) boom would be deployed in normal catenary. Approximately 20,000 gallons of treated Brent Crude would be spilled by the command/recovery ship Terra Nova Sea into the catenary. The oil would be held in the boom for approximately 1 hour while freeboard and draft data and visual observations of oil retention were recorded. During this period, 200 meters of the Canadian Coast Guard's RO-BOOM would be deployed behind the OHMSEIT boom. The tow speed would be increased to significant loss speed (0.5-1.0 knot). One end of the boom would then be released and the oil discharged into the RO-BOOM. Oil would be held in the RO-BOOM for approximately 1 hour while the oil retention capabilities were observed. The St. John's Coast Guard Vikoma Ocean Pack boom (400 meters) would be deployed behind the RO-BOOM during the observation period. The last procedure involving lost tow speeds would be repeated with the RO-BOOM, and the oil would be released into the Vikoma.

Oil would be retained in the Vikoma for approximately 1 hour. The Terra Nova Sea would then commence skimmer evaluations. Two skimmers, the Framo ACW400, and an innovative Coast Guard Heavy Oil Skimmer (HOS) would be evaluated for 20 minutes each and the remaining contained oil would be recovered by the skimmer with the better performance (Ross, 1987b).

The intent of the OHMSEIT boom deployment would be to verify the hypothesis that a boom's ability to contain oil is correlated with its ability to sea keep or comply with wave-induced surface motion. If this hypothesis could be verified and quantified, future performance evaluations of offshore containment booms could be restricted to measuring seakeeping capabilities in a range of sea states. No further spills of the 20,000 gallon size of light and heavy oils would be required, in a range sea states, to evaluate each type of boom. Ocean dumping permits are difficult to obtain and intentional oil spill exercises of this magnitude approach the million dollar funding level. Intentional spills also constitute a risk of potential damage to the immediate environment. Clearly a cost-effective and nonpolluting evaluation procedure for offshore equipment is necessary to develop a predictive capability for the performance of offshore response equipment. Wind conditions desired were sea state 2 to 4, and winds from 10 to 20 knots.

#### Practice Run - September 21, 1987

The ships and smaller vessels sailed at 6:00 a.m. and proceeded to a location 5.5 miles off Torbay point. The CCG Grenfell then deployed the OHMSEIT boom and passed one end to the CCG cutter 212 which took the boom in tow. The CCG cutter 206 then attempted to pick up the trailing end of the boom, a job which took 45 minutes. As soon as the 206 had the end of the boom secured, the two cutters attempted to tow the boom, in a "U" configuration, into a position astern of the Terra

Nova Sea. This resulted in the OHMSETT boom immediately beginning to twist on itself, and 1 hour was lost in straightening the boom. Eventually the boom was positioned relative to the Terra Nova Sea and simulated discharge of the oil was carried out. Data collection, without oil, for almost 1 hour followed.

While the OHMSETT boom was being deployed and positioned, the RO-BOOM was deployed from the COG Sir Humphrey Gilbert and passed to COG cutter 214. This procedure took almost 2 hours and, the RO-BOOM was rapidly positioned with respect to the OHMSETT boom because both the Cutter 214 and a Boston Whaler were able to tow the boom at speeds of 5 knots. With the RO-BOOM in position, the COG cutters 212 and 206 commenced to maneuver, presumably to form a "J." The COG cutter 206 then snagged the OHMSETT cable in her screws and cut it, disabling herself. This resulted in delay in the exercise while the cable was freed and, because COG cutter 214 and the Boston Whaler were now approaching closely, the Cutter 212 took the boom and Cutter 206 in tow and cleared the area so as not to impede the exercise.

During the period in which the RO-BOOM was being deployed, the COG Grenfell deployed the Vikoma boom. Once the simulated oil release was over, the Terra Nova Sea took the other end of the boom and the vessels formed a Catenary. After holding position relative to the RO-BOOM for a period, the ships formed a "J" with the boom and practiced deploying the skimmers.

All ships returned to harbour by 5:00 p.m.

#### Period Between the Dry Run and the Trial

Several meetings of the senior people involved in the exercise occurred between September 21 and September 23, 1987. It was decided to remove COG cutter 206 from the exercise. The Boston Whaler was able to tow and hold the boom in sea state 1, but it was recognized that this would be difficult, with oil, in the desired weather. The Newfoundland Fisheries department had provided a vessel, the Bernier, and it was decided to use her and a second chartered OSV to tow the RO-BOOM.

Concern was registered that the weather might, surprisingly, be too calm on September 24, the day which looked best for the actual trial. It was, therefore, decided to interchange the OHMSETT and RO-BOOMS to take advantage of the higher winds and waves expected later in the day.

The long time required to deploy the RO-BOOM from the Sir Humphrey Gilbert led to the decision to deploy that boom from the second OSV, the Triumph Sea. Repairs were carried out on the OHMSETT boom cabling and the boom with instrumentation, was functional by 5:00 p.m. on September 23.

Since the Terra Nova Sea had oil recovery tanks on board, it was decided to dispense with the dumb barge and to release the oil directly from that OSV.

Trial with Oil on September 24, 1987

The Triumph Sea and the Bernier sailed at 3:00 a.m. All other vessels sailed at 4:00 a.m., and everyone was on station by 6:45 a.m. The Triumph Sea commenced deployment of the RO-BOOM enroute and by 7:30 a.m., the boom was ready to receive oil. Once the OHMSEIT boom was deployed, at 8:15 a.m., the oil was pumped into the RO BOOM with supervision from the helicopter and a small boat.

All oil was in the boom by 9:00 a.m.

It was decided to give the news media a chance to view the test from the air and this was done from 9:00 a.m. to 10:00 a.m. During this period, the OCG cutters 212 and 214 attempted to pull the OHMSEIT boom into proper position astern of the RO-BOOM and keep it there. Every attempt to move the boom, in a catenary, in the 15 knot winds blowing at the time resulted in the boom twisting. As a result the OHMSEIT boom fell progressively further astern of the RO-BOOM.

At 10:30 a.m. the oil in the end of the pocket of the RO-BOOM was 30 cm thick, the wind was 15 knots, and some splash-over and significant drainage under the boom were occurring. The vessels, therefore, formed a "J" and released one end of the boom to allow the oil to flow into the OHMSEIT boom. The OHMSEIT boom was approximately 1 km astern, and the vessels were having trouble towing the boom in a catenary without the boom twisting. It was, therefore, decided to direct the cutters with the OHMSEIT boom, to the oil, by helicopter, keeping the boom in a straight tow. After 20 minutes, the cutters were adjacent to the thick oil, and, after a further 20 minutes approximately 80 percent of the thick oil was in the OHMSEIT boom. The remaining 20 percent was contained in the Vikoma boom. Data collection on the OHMSEIT boom started as soon as the oil was captured and continued for 56 minutes. The OCG cutters then stopped across the path of the vessels towing the Vikoma boom and released one end of the boom. The oil spilled into the Vikoma boom catenary, and one OCG cutter trailed the OHMSEIT boom at the throat of the catenary allowing the waves to wash the oil into the Vikoma boom.

The OCG Grenfell and Terra Nova Sea towed the oil filled Vikoma boom for approximately 1 hour. During this time, the wind had freshened to 15 knots to 20 knots. The boom was moving at over 1.1 knots relative to the sea, and some oil was being lost (approximately 3 mm thick). The OCG Grenfell then attempted to move ahead to form the "J" for the skimmers. Not being very maneuverable, she quickly reached 3.4 knots and the oil was lost.

With the oil now lost, the weather abated slightly. The RO boom was still streaming astern of the Triumph Sea so it was decided that she and the Bernier would form the boom into a catenary and attempt to recover the oil downwind. The helicopter had been lost to the exercise for approximately 1 hour at this time. As soon as it returned, it was refueled and sent up to assist. In the interim a small boat was used to guide the tow vessels into the heaviest portions of the slick. When the helicopter was over the thick oil, it was apparent that the vessels with the RO-BOOM were adjacent to some of the oil and unable to see it. They were, therefore, directed

from the air and small boat, and managed to collect 80 to 90 percent of the thick oil which was on the surface at the time.

Oil was successfully contained and recovery was attempted using the three skimmers on board the recovery-command vessel. The first skimmer, the Heavy Oil Skimmer (HOS), was deployed and no measurable recovery was observed. The oil used was modified by adding petroleum wax so that it would resemble a typical Grand Banks crude oil. This type of oil is uncharacteristic of most crudes in that it possesses low adhesive properties. Therefore, oleophilic skimmers, which depend upon the adhesive nature of the recovered oil, do not perform well with high paraffin-based oils. This was again proven with the oleophilic HOS skimmer. The skimmer was recovered after several trained observers were satisfied that the HOS skimmer had had sufficient evaluation time in the contained slick. Problems with the ability of the support arms, used to suspend both the HOS and Framo skimmers, to adjust to the roll of the vessel and short period wave action resulted in both skimmers being frequently submerged so that oil and water were washed into the sump of the skimmer.

The second skimmer the "Framo ACW-400" was deployed. The overall rate of oil recovery of the Framo was 60 gallons per minute with unknown amounts of the recovery resulting from frequent partial submergence.

At this point, it was decided by the On-scene Commander that the skimmer evaluation was complete. Additional measures were needed to ensure recovery of the remaining contained oil because the weather was deteriorating and night was falling. Accordingly, approximately 7 pounds of the visco-elastic agent "Elastol" were spread from an 8-ounce styrofoam coffee cup into the estimated 7,400 gallons of oil and oil water emulsion in the containment boom. Elastol was added because previous research funded by the Minerals Management Service and Environment Canada had shown that the elastic and adhesive properties of the oil could be increased by addition of the agent, thus making the oil more readily capturable with these types of skimmers. The Framo ACW-400 was retrieved from the slick as the "Elastol" was added and because of the operational constraints on the recovery operation, due to the weather and lateness of the day, the skimmer was not redeployed.

The weir-type skimmer, Pharos Marine GI-185, was deployed into the treated slick and recovered near capacity rates of 85 gallons per minute of oil and oil emulsion with no free water. This recovery rate was higher than anticipated and may have been even higher if the oil had been untreated. Treatment significantly increased the viscosity of the oil. The skimmer was removed from the slick, cleaned, and stored on board.

The HOS skimmer was redeployed and failed to function because of a piece of steel in the return pump. The skimmer was recovered, the steel piece removed, and then the skimmer was redeployed.

The HOS skimmer yielded a recovery rate of 50 gallons per minute with a portion of the oleophilic fabric on one of its two drums damaged. Debris was collected at this time in the ventury system used to measure recovery fluid flow rates. The debris may have contributed to the subsequent failure of the HOS skimmer return hose. No flow-rate measurements were taken before the failure. Visual observations on the amount of oil adhering to the oleophilic fabric of the HOS skimmer indicated that recovery rates were significantly increased by the addition of "Elastol."

Operations were suspended because of the advancing sea states and increasing darkness.

An overflight of the area, by helicopter, was carried out during the skimming operation. This revealed a sheen approximately 2.5 by 0.5 nautical miles with 3 patches of brown oil. It is estimated that no more than 260 gallons of oil remained in the thick patches. A further flight 18 hours later showed that only small brown patches and sheen remained, and this was rapidly dispersing.

#### Lessons Learned or Relearned

- o Thorough proficiency with the recovery equipment to be used is essential.  
Routine practice is required.
- o Large volumes of oil are necessary to realistically evaluate performance of offshore response equipment.
- o The use of helicopters to direct the placement of tow vessels and the use of small vessels to monitor and advise on boom conditions are essential to maximize the efficiency of conventional recovery operations.
- o Accurate measurements of the meteorological and sea conditions are necessary for accurate analysis of the evaluations.
- o The requirements for slow-speed towing and maneuvering of large containment booms necessitate the use of vessels with variable pitched propellers, thrusters, and good seamen in control.
- o It was not possible to form a recovery configuration with two vessels while towing upwind. The third vessel was necessary for recovery in normal catenary.
- o The upper meteorological and sea state limits for downwind containment and recovery were not reached during this test.
- o Upwind collection proved impossible when winds approached 15 knots. This is consistent with many past observations for containment operations conducted upwind.
- o Steps should be taken to avoid loss of contained oil by shipboard discharges such as cooling water impinging upon the slick.
- o Tankage should be available for recovery of several times as much fluid as discharged to account for the oil and water emulsions and free water recovered.
- o Analysis of the correlation of the ability of a boom to seakeep with its ability to contain oil is continuing.

- o Recovery of high wax oils similar to Newfoundland crudes in 10 degrees C water is significantly enhanced by the use of "Elastol."

Disclaimer

Mention of specific products in this paper does not constitute or infer endorsement or acceptance by the Minerals Management Service, the Conservation and Protection, or the authors.

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**Attachment 12**

**SOIL PENETRATION**

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Ludwigshafen/Rhein, Federal Republic of Germany

Laboratory tests of ELASTOLT.M. (Oil Spill Combat Agent)

H.M. Laun and R. Hingmann

I. Introduction

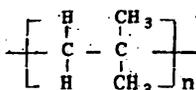
ELASTOLT.M.\* was developed by GTA with the assistance of BASF. It is manufactured in the form of a white powder with particle sizes between 100  $\mu\text{m}$  and 1000  $\mu\text{m}$  (Fig.1).



Fig.1: The powder form of ELASTOLT.M. simplifies application.

\* in USA registered trademark of General Technology Applications (GTA) Inc., Manassas, Virginia; in other countries of Elastogran Polyurethane GmbH, a company of BASF group

The powder contains about 50% by weight of Polyisobutylene (PIB) of extremely high molar mass ( $M_w = 6 \cdot 10^6$  g/mol). PIB is a non-toxic polymer that only consists of carbon and hydrogen atoms. The chemical composition is



Fully extended the polymer chain would reach a length of about 17  $\mu\text{m}$ . Granules of polymeric material are coated with water-insoluble  $\text{Ca}_5(\text{PO}_4)_3\text{OH}$  salt in order to obtain an easily spreadable, non-agglomerating powder. Only the PIB-component is soluble in oil. When dissolved the macromolecules give rise to a distinct viscoelasticity as well as a drastic increase in the elongational viscosity of the solution even at PIB concentrations of only a few hundred ppm.

These properties make ELASTOL<sup>T.M.</sup> a very interesting oil spill treating agent [1]. The performance of skimmers is improved, the resistance to spreading and break-up is greater, and the speed of collecting barriers can be increased.

In this work we report laboratory tests with ELASTOL<sup>T.M.</sup> applied to different oils at various concentrations. The intention is on one hand to give quantitative data on both the rheological properties of the ELASTOL<sup>T.M.</sup> treated oils and on the dissolving kinetics of the powder. On the other hand we try to illustrate the basic mechanisms that are responsible for the observed properties of the solution. These properties are mainly due to the stretching and orientation of the dissolved polymer chain. These are purely physical effects which work independently of possibly present chemically active additives.

In Table 1 the fractionated oils and crude oils used in this investigation are listed.  $\eta_0$  is the zero shear rate viscosity of the oils at 25°C.

**Table 1:** List of oils used in this investigation and their zero shear rate viscosities  $\eta_0$  at 25°C.

fractionated oils	$\eta_0$ [mPa·s]
petroleum	1.42
fuel oil	3.65
diesel oil (Columbia diesel)	6.9
spindle oil	7.0
transmission lubricant/petroleum 1/1 (mixture)	10.5
transformer oil	13.0
MARCOL <sup>T.M.</sup> 82 (Exxon)	21.6
fine mechanics lubricant	34.3
vacuum pump oil	190
transmission lubricant	713

crude oils	$\eta_0$ [mPa·s]
Quaibo	4.6
Arabmed	18.0
Venezolana/Quaibo 61/39 (mixture)	70.0

## II. Rheological properties of ELASTOL<sup>T.M.</sup> oil solutions

### 1. Dependence of the solution viscosity on the type of oil

The oils listed in Table 1 were treated with both 2 000 ppm and 10 000 ppm (0.2 and 1% by weight) ELASTOL<sup>T.M.</sup>. The solutions were produced by rolling the powder-oil mixtures in closed glass bottles at about 120 rpm and 23°C for a time period of 30 to 70 hours.

The viscosity of the resulting solutions was determined by means of a high precision Couette Rheometer (Contraves LS 30). This instrument can measure shear stresses ranging from about  $3 \cdot 10^{-4}$  Pa to 4 Pa in a shear rate range between  $3 \cdot 10^{-3} \text{ s}^{-1}$  and  $100 \text{ s}^{-1}$ .

In general the PIB solutions are non-Newtonian, viz. the viscosity of the fluid is dependent on the shear rate. In our measurements, therefore, shear rates small enough to get the constant viscosity  $\eta_s$  at the limit of small shear rates (zero shear rate viscosity) were used. The data are listed in Table 2.

Table 2: Viscosity increase due to ELASTOL<sup>T.M.</sup> (dissolved by rolling) in various oils for powder concentrations of 2 000 and 10 000 ppm. Viscosities were measured at 25 °C.

oil	$\eta_0$ [mPa·s]	c = 2 000 ppm		c = 10 000 ppm	
		$\eta_s$ [mPa·s]	$\eta_r$	$\eta_s$ [mPa·s]	$\eta_r$
petroleum	1.42	3.02	2.13	23.3	16.3
fuel oil	3.65	7.54	2.07	52.3	14.3
spindle oil	7.0	15.1	2.16	97.1	13.9
transmission lubricant/ petroleum 1/1	10.45	17.1	1.64	83.4	7.9
transformer oil	13.0	22.7	1.75	122.6	9.4
MARCOL <sup>T.M.</sup> 82	21.6	36.1	1.67	178	8.2
vacuum pump oil	190	238.4	1.25	908	4.8
transmission lubricant	713	845.6	1.19	1656	2.3
Quaibo	4.6	8.5	1.85	46.2	10.0
Arabmed	18.0	24.0	1.33	74.6	4.1
Venezolana/ Quaibo 61/39	70	83.7	1.20	241	3.4

In the case of the most viscous oils (vacuum pump oil, transmission lubricant) and a powder concentration of 10 000 ppm the PIB could not entirely be dissolved even after a rolling time of > 70 h. For these two samples the  $\eta_s$ -values only represent lower limits of the viscosity of a homogeneous solution.

The relative viscosities  $\eta_r$

$$\eta_r = \eta_s / \eta_0 \quad (1)$$

also listed in Table 2 are the ratios of solution viscosity and oil viscosity.  $\eta_r$  increases with increasing powder concentration. It is also seen that the relative viscosity increase due to ELASTOL<sup>T.M.</sup> becomes smaller with increasing viscosity of the oil. For 1% concentration we get a relative viscosity of 16.3 for petroleum, compared to 4.8 for vacuum pump oil.

In Figure 2 and Figure 3 the relative viscosities are plotted versus the logarithm of the oil viscosity for 2 000 ppm and 10 000 ppm powder, respectively. It is interesting to note that for the

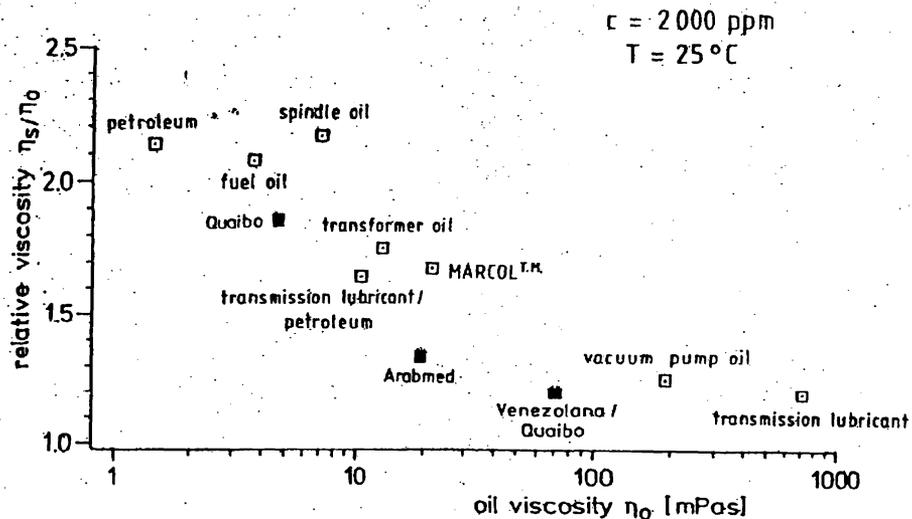


Fig.2: Relative viscosities  $\eta_r$  for various oils as obtained after rolling powder oil mixtures with an ELASTOL<sup>T.M.</sup> concentration of 2 000 ppm at 25 °C. Open symbols represent fractionated oils, full symbols crude oils.

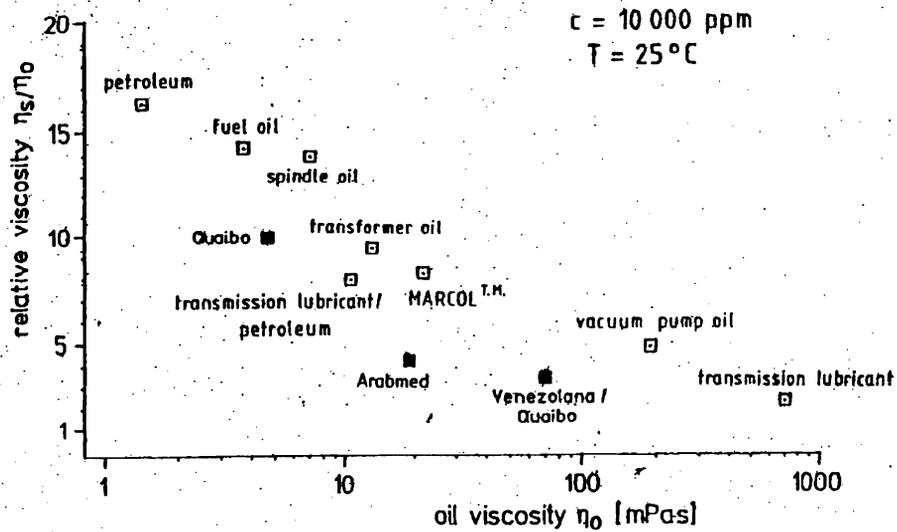
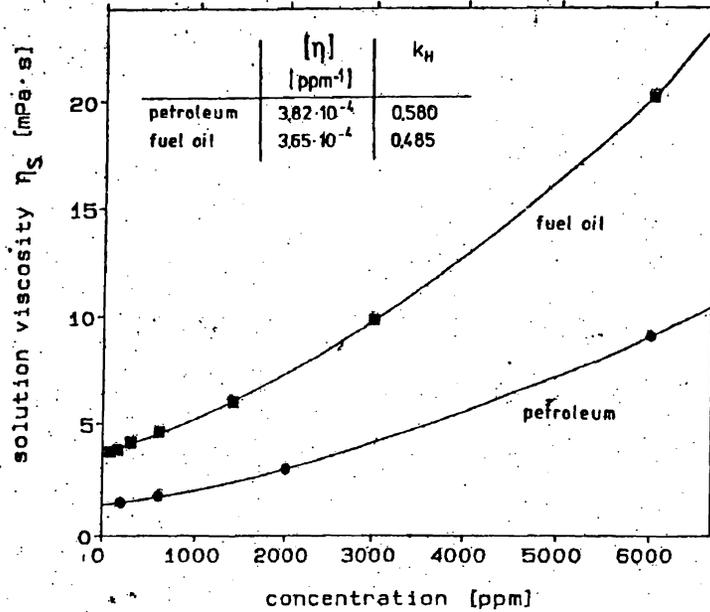


Fig.3: Same as Fig.1 for an ELASTOL<sup>T.M.</sup> concentration of 10 000 ppm.

1/1 mixture of petroleum and transmission lubricant as well as for the crude oils, the relative viscosities are significantly smaller compared to fractionated oils of similar viscosity. A possible explanation for the observed effect might be the reduction of the coil extension in the oil mixtures due to changes in the average polymer-solvent interaction.

## 2. Dependence of solution viscosity on powder concentration

Zero shear rate viscosities  $\eta_s$  were measured on solutions in petroleum and fuel oil at various concentrations. For this purpose base solutions having 6 000 ppm ELASTOL<sup>T.M.</sup> were prepared and subsequently diluted by adding solvent. The resulting data are shown in Figure 4.



**Fig.4:** Solution viscosity  $\eta_s$  versus ELASTOL<sup>T.M.</sup> concentration for petroleum and fuel oil. Full lines represent the fit by the Huggins equation (see text) using the parameters given in the inserted table.

The viscosity of the solutions increases stronger than proportional to the powder concentration in the range tested. The experimental results are nicely described by a polynomial of order 2 (full lines) as proposed by Huggins [2]:

$$\eta_s = \eta_0 (1 + [\eta]c + k_H [\eta]^2 c^2) \quad (2)$$

$[\eta]$  being the intrinsic viscosity and  $k_H$  the Huggins constant. The values determined by a best fit to the data points are given in the diagram.

### 3. Viscoelasticity of ELASTOLT.M. solutions

The solutions were submitted to small amplitude oscillatory shear in a rotational rheometer using bi-cone geometry. The apparatus is schematically depicted in Figure 5. A gap angle of  $\alpha = 3.8^\circ$  and angular frequencies  $\omega = 2\pi f$  ( $f$  frequency of the oscillation) in the range of 0.2 to 18  $s^{-1}$  were used. The shear amplitude was  $\hat{\gamma} = 0.5$ .

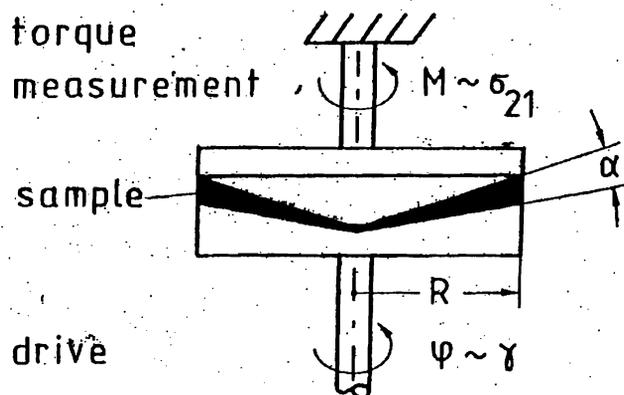


Fig.5: Schematic drawing of the bi-cone geometry used for small amplitude oscillatory shear.

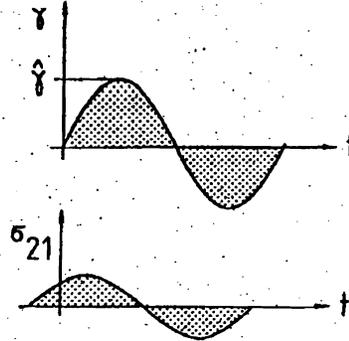
The resulting shear stress  $\sigma_{21}$  is oscillating at the same frequency but is phase shifted compared to the shear strain  $\gamma$  [3] (see Fig.6). It can be decomposed into Sine and Cosine components by means of a frequency response analyzer.

The direct output of the measurements are the storage modulus  $G'$  and the loss modulus  $G''$  as functions of the angular frequency  $\omega$ . In our experiments the contribution of inertia forces to  $G'$  has been corrected for analytically.

shear strain  $\gamma$ :

$$\gamma = \hat{\gamma} \sin \omega t$$

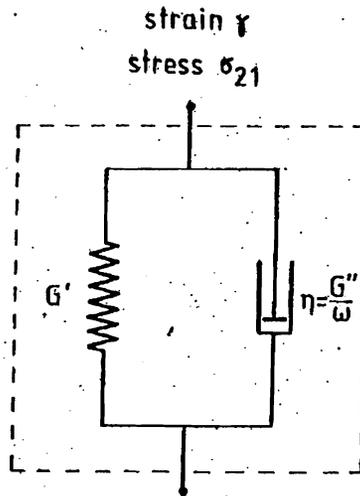
shear stress  $\sigma_{21}$ :



$$\sigma_{21} = \hat{\gamma} [ \underbrace{G' \sin \omega t}_{\text{storage modulus}} + \underbrace{G'' \cos \omega t}_{\text{loss modulus}} ]$$

**Fig.6:** Decomposition of the phase shifted oscillating shear stress into two components yields the storage modulus  $G'$  and the loss modulus  $G''$ .

In general the response of solutions to oscillatory shear can be described by an elastic spring  $G'$  and a dashpot  $\eta = G''/\omega$  arranged in parallel [3] (Fig.7). A purely viscous fluid of viscosity  $\eta$  can be represented by the dashpot alone and the shear stress is proportional to the shear rate  $\dot{\gamma}$  which yields a phase shift of 90 degrees between stress and strain. The dissipated energy per cycle is proportional to the loss modulus  $G''$ . An elastic material (e.g. ideal rubber) can be represented by the spring. Here the stress is proportional to the strain (no phase shift). The storage modulus  $G'$  represents the spring constant and is proportional to the stored energy per cycle. A viscoelastic material has both components. The quantity  $G'/G''$  is equal to the ratio of stored and dissipated energy.

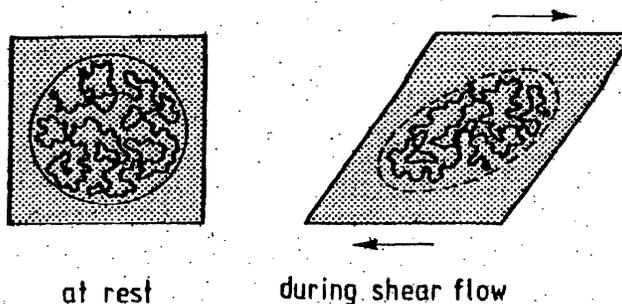


$$\sigma_{21} = G' \gamma + \eta \dot{\gamma} = G' \gamma + \frac{G''}{\omega} \dot{\gamma}$$

**Fig.7:** Representation of the response a viscoelastic fluid to oscillatory shear by a spring and dashpot arranged in parallel ( $G'$  and  $G''$  are frequency dependent).

The behaviour of long polymer chains dissolved in a viscous fluid (PIB macromolecules dissolved in oil in our case) can be understood as follows: at rest the molecules have a random coil like equilibrium conformation (left side of Fig.8). In a flow field the random coils immobilize part of the solvent giving rise to the observed viscosity increase with increasing polymer concentration.

If the equilibrium coil conformation remains undisturbed the solution would behave in a purely viscous manner.

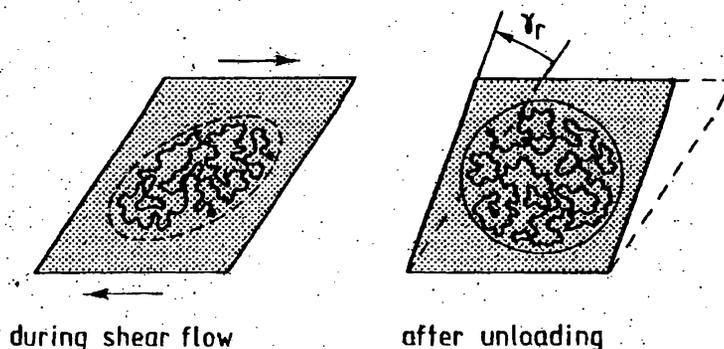


**Fig.8:** Equilibrium conformation (random coil) at rest and deformed state during flow of a macromolecule in solution (schematic).

In reality, during flow the molecule attains an ellipsoidal conformation (right side of Fig.8) and will partially be oriented parallel to the direction of flow. This deformation of the coil, however, is reversible. When the flow is stopped the molecule will re-establish its equilibrium conformation. This process takes some time and is governed by a characteristic retardation time  $\tau$ . An estimate for the characteristic time is possible based on the moduli  $G'$  and  $G''$ :

$$\tau = G' / \omega G'' \quad (3)$$

It must also be mentioned that, if the solution is not kept under shape constraint after removal of the shear deformation, the re-attainment of the equilibrium random coil conformation will result in a reversed shear of the sample (Fig.9). The total recoverable strain  $\gamma_r$ , although difficult to measure on dilute solutions, may also be used as a direct measure of the elasticity of the sample besides the ratio  $G'/G''$ .



**Fig.9:** Recoverable shear strain of a viscoelastic solution after unloading due to the desorientation of deformed molecules.

Coming back to our measurements of the moduli  $G'$  and  $G''$  it is important to note that small amplitude oscillatory shear measures the viscoelasticity of the solution at small deviations from the equilibrium (coil-like) conformation of the dissolved PIB molecules.

As an example Figure 10 shows the measured dynamic moduli of a 16 000 ppm solution of ELASTOLT.M. in petroleum. The pronounced viscoelasticity of the solution is evident from the fact that besides the loss modulus a distinct storage modulus can be measured. The ratio  $G'/G''$  increases with growing angular frequency.

The viscosity  $|\eta^*|$  represented by the full symbols is calculated from the moduli according to

$$|\eta^*| = \frac{1}{\omega} \sqrt{G'^2 + G''^2} \quad (4)$$

This quantity is very close to the value of the viscosity in steady shear flow if  $\omega$  is equal to the shear rate [4]. Thus, the decrease of  $|\eta^*(\omega)|$  with increasing angular frequency reflects the non-

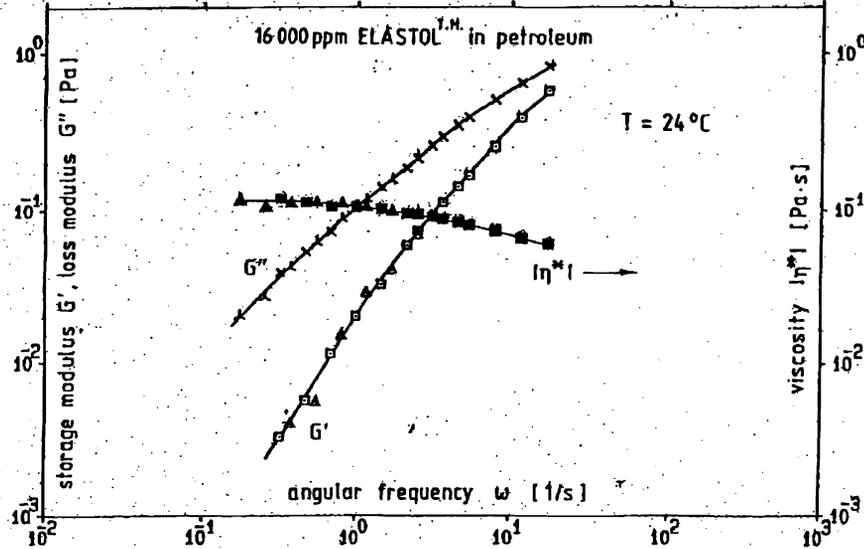
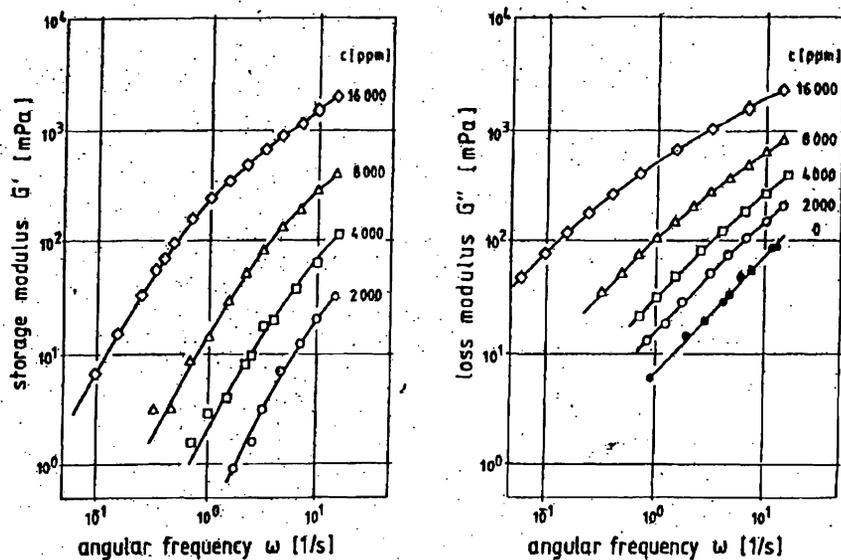


Fig.10: Storage and loss moduli (open symbols) and viscosity (full symbols) versus angular frequency of an ELASTOL<sup>T.M.</sup> solution in petroleum at 16 000 ppm and 24°C.

Newtonian behaviour of the solution, viz. the shear viscosity decreases with increasing shear rate. Again, this behaviour can be understood in terms of Fig.8. Deformed and partially oriented macromolecules give rise to a viscosity decrease depending on the shear rate.

Figure 11 shows the concentration dependence of the moduli in diesel oil. The pure oil (full circles) is Newtonian and does not have a measurable storage modulus. In that case  $G''$  increases proportional to  $\omega$ , the ratio  $G''/\omega$  being equal to the viscosity  $\eta$  of the oil [3].

With increasing ELASTOL<sup>T.M.</sup> concentration the storage modulus grows stronger than the loss modulus. For  $\omega = 1 \text{ s}^{-1}$  (0.13 Hz) a fourfold increase in concentration (4 000 ppm to 16 000 ppm) yields a  $G''$  growth of less than a factor of 20 whereas the storage modulus

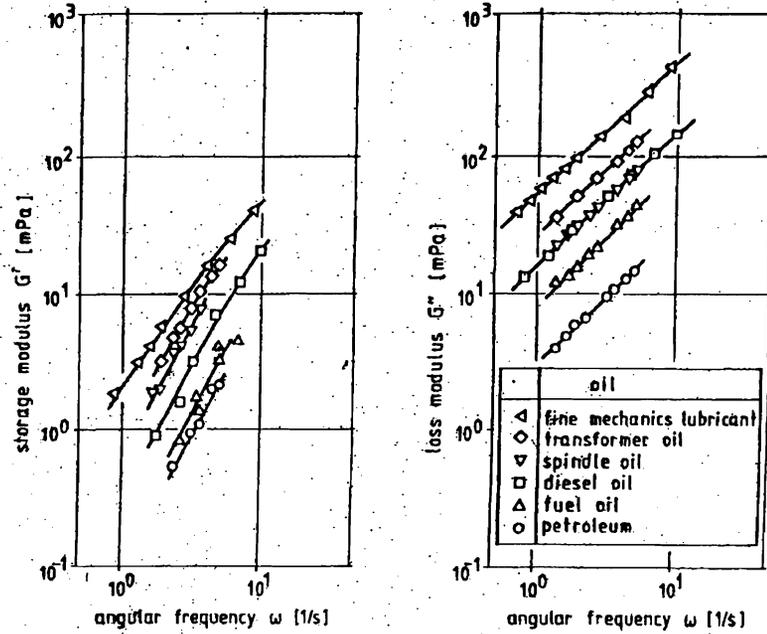


**Fig.11:** Storage modulus (left diagram) and loss modulus (right diagram) versus angular frequency of ELASTOLT.M. solutions in diesel oil at various concentrations and 24°C. The pure oil does not have a measurable storage modulus.

increases approximately 100-fold. As a result, the ratio  $G'/G''$  at a given frequency (cf. Table 3), taken as a quantitative measure of the viscoelasticity, increases considerably. It is also seen that in the same sequence the increase of  $G''$  is less than proportional to  $\omega$ .

A comparison of the dynamic moduli for a constant ELASTOLT.M. concentration of 2 000 ppm in various oils of different viscosities is shown in Figure 12.

Table 3 gives a compilation of the moduli measured on various solutions. Here, the  $G'$  and  $G''$  are compared at a constant angular frequency of  $\omega = 1.26 \text{ s}^{-1}$  ( $f = 0.2 \text{ Hz}$ ) and 24°C. Besides the



**Fig.12:** Storage modulus (left diagram) and loss modulus (right diagram) versus angular frequency for ELASTOLT.M. solutions in various oils at a constant concentration of 2 000 ppm and 24 °C.

viscosity  $|\eta^*|$  (Equ. (4)) and the ratio  $G'/G''$  this table also gives the characteristic relaxation time  $\tau$  (Equ. (3)). For each oil both  $G'/G''$  and  $\tau$  increase with growing concentration. The most pronounced changes are observed in the characteristic time.

**Table 3:** Dynamic moduli and viscoelastic properties of various solutions measured at  $\omega = 1.26 \text{ s}^{-1}$  by means of small amplitude oscillatory shear.

	c [ppm]	G* [mPa]	G' [mPa]	$ \eta^* $ [mPa·s]	G'/G*	$\tau$ [ms]
petroleum	0	1.6	-	1.3	-	-
./.	8 000	23	1.6	18	0.07	55
./.	16 000	125	25.6	102	0.21	140
fuel oil	0	4.8	-	3.8	-	-
./.	4 000	12	0.35	8.8	0.03	23
./.	8 000	24	1.1	19	0.05	36
diesel oil	0	-	-	-	-	-
./.	2 000	20	0.4	16	0.02	16
./.	4 000	39	3.2	31	0.08	65
./.	8 000	125	21.6	105	0.17	137
./.	16 000	590	290	523	0.49	390
spindle oil	0	8.9	-	7.1	-	-
./.	2 000	18	0.65	14	0.04	29
./.	4 000	33	2.1	26	0.06	51
./.	8 000	85	12.6	68	0.15	118
transformer oil	0	16	-	13	-	-
./.	2 000	34	2.3	27	0.07	54
fine mechanics lubricant	0	42	-	33	-	-
./.	2 000	67	3.0	53	0.04	35

#### 4. Drawability (elongational viscosity) of ELASTOL<sup>T.M.</sup> solutions

One of the most striking effects of ELASTOL<sup>T.M.</sup> dissolved in oil is the dramatic increase in resistance to elongational flows. When the solution is stretched the PIB molecules are oriented which yields an elongational viscosity greater than three times the shear viscosity [5]. This behaviour is best demonstrated in the ductless siphon test [6] (Fig.13).

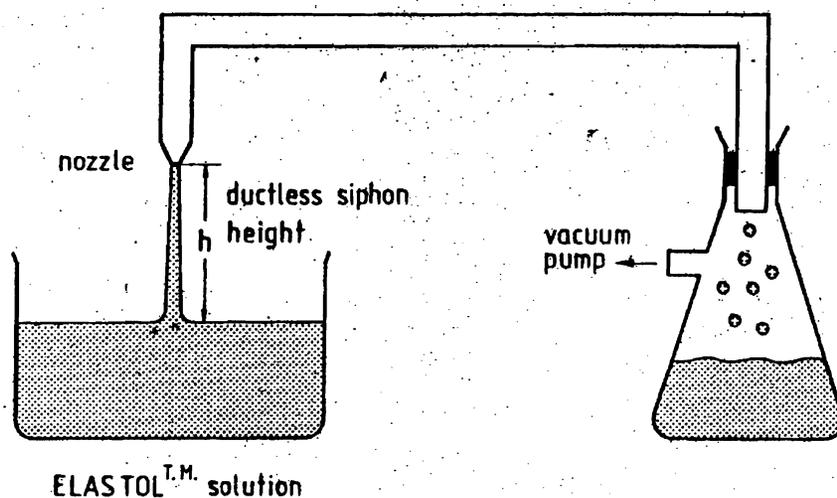


Fig.13: Schematic drawing of the ductless siphon test method. The ductless siphon height is gradually increased until break of the filament at  $h_{max}$ .

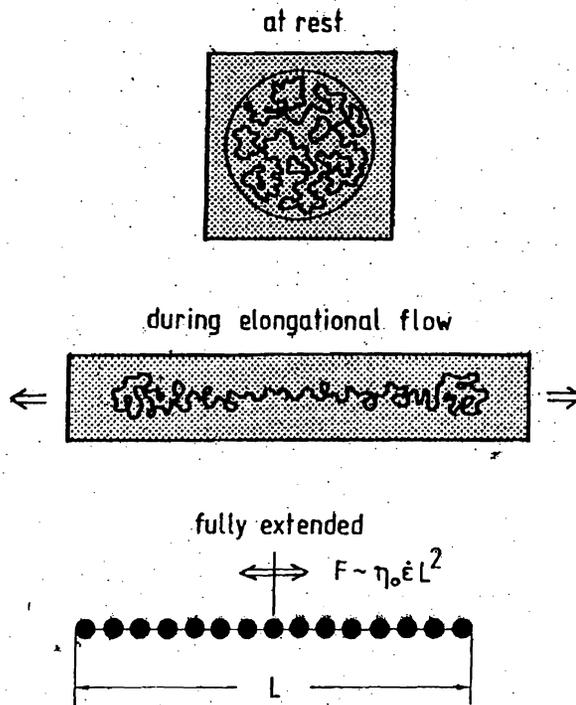
A pipe being connected to a vacuum pump carries a nozzle at its other end. The solution is contained in a beaker. When the nozzle is brought into contact with the meniscus of the solution the latter is sucked out of the beaker. The distance  $h$  between nozzle and solution meniscus is increased either by slowly lowering the container position or just due to the decreasing meniscus height for a fixed beaker position. The solution will still flow upward until at a maximum ductless siphon height  $h_{max}$  the filament breaks.

For an untreated oil, in general, the maximum ductless siphon height will be in the order of 1 mm. With ELASTOLT.M.  $h_{\max}$  is much greater and may reach values of half a meter or more. This behaviour is of tremendous importance for the performance of collecting devices used for oil spill combat.

A photograph of the spinline in the ductless siphon test is shown in Fig.14 for a 4 000 ppm solution in fine mechanics lubricant (with red dye) and a nozzle diameter of 1 mm. The distance between the nozzle and the solution meniscus was 20 cm.



Fig.14: Ductless siphon test on a 4 000 ppm solution in fine mechanics lubricant. Nozzle diameter 1 mm.



**Fig.15:** Stretching of the polymer chain when the solution is subjected to an elongational flow field (schematic).

The physical reason for the high elongational viscosity is schematically depicted in Fig.15. At rest the molecule has a coil like conformation, but in an extensional flow field, the molecule is stretched. The degree of stretching is dependent on the rate of strain of the solution and the duration of extensional flow. The sum of friction forces due to the surrounding solvent molecules has an opposite sign on both sides of the center of mass. Therefore, the extensional forces acting on the molecule are maximal in the middle of the chain and the orientation of chain segments parallel to the direction of strain is most pronounced in that region.

For the extreme case of a fully extended chain (bottom diagram in Fig.15) it is easy to show that the force  $F$  acting in the center of the molecule is proportional to the chain length  $L$ , the solvent viscosity  $\eta_0$ , and the strain rate  $\dot{\epsilon}$  [7]. The force  $F$  may even become higher than the chemical bond of the backbone. Under that condition the molecule will break [7].

It is obvious that in the case of long molecules the resistance to elongational flow is mainly governed by the force  $F$  that can be transmitted by each single molecule from one volume element to another and the concentration of dissolved macromolecules. In summary, we expect the elongational viscosity  $\mu$  [5] of the solution

$$\text{elongational viscosity } \mu = \frac{\text{tensile force/cross section}}{\text{extension rate}} \quad (5)$$

to increase with strain rate, solvent viscosity, and polymer concentration.

It should also be noted here that the stretched molecules store energy since the orientation process is fully reversible (chain rupture excluded). When the strain rate is set to zero the deformation of the molecule will decay with time until the coil conformation is re-established. Without shape constraint this process will cause a shrinkage (recoverable strain) of an elongated solution filament.

The measured ductless siphon heights  $h_{\max}$  of petroleum, fuel oil, and spindle oil versus ELASTOLT.M. concentration are plotted in Figure 16. We observe a monotone increase of  $h_{\max}$  with increasing powder concentration in petroleum. For fuel oil and spindle oil, however,  $h_{\max}$  goes through a reproducible minimum. It appears that the concentration corresponding to the filament length minimum is shifted to smaller values when the oil viscosity is increased. Most probably this effect is not caused by a minimum in the true resistance of the filament to stretching but by problems in the nozzle flow due to the shear viscosity which increases with concentration.

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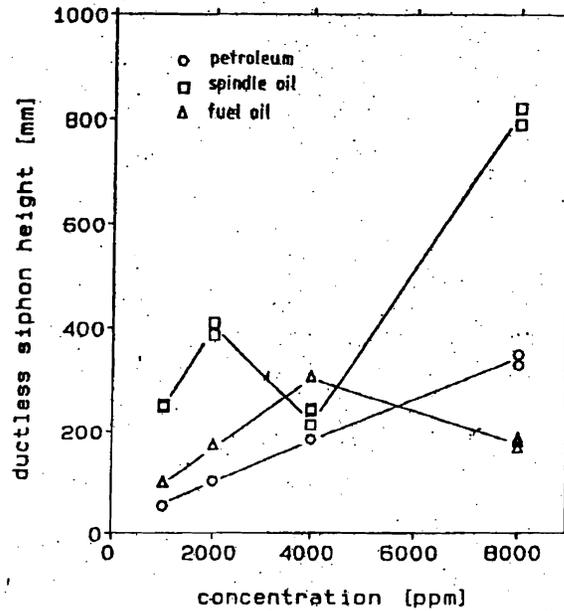


Fig.16: Maximum ductless siphon heights at room temperature for three oils versus ELASTOL<sup>T.M.</sup> concentration (nozzle diameter 1 mm).

Thus, for a direct comparison of the effect of ELASTOL<sup>T.M.</sup> on the drawability in various oil solutions one should restrict the measurements to concentrations below the minimum. In that range the behaviour may be approximated by the power law

$$h_{max} = K_{DS} c^{\alpha} \quad (6)$$

the exponent  $\alpha$  having values in the range of 0.75 to 0.95.

Table 4: Ductless siphon heights  $h_{max}$  at filament break for various oils and at an ELASTOL<sup>T.M.</sup> concentration of 2 000 ppm.

oil	$\eta_0$ [mPa·s]	$h_{max}$ [mm]
petroleum	1.42	100
fuel oil	3.8	187
spindle oil	7.0	380
transformer oil	13.0	471
fine mechanics lubricant	34.3	805
petroleum + SEPAROL <sup>T.M.</sup> AF 27	-	99

Table 4 gives a compilation of the measured  $h_{max}$  values in various oils for a constant ELASTOL<sup>T.M.</sup> concentration of 2 000 ppm. The maximum ductless siphon height strongly increases with increasing oil viscosity. Therefore, the higher the oil viscosity, the lower the concentration of ELASTOL<sup>T.M.</sup> required to obtain a given value of  $h_{max}$ .

As already mentioned, the drawability of ELASTOL<sup>T.M.</sup> solutions is an exclusively physical phenomenon. The application of chemical additives (for instance emulsion breakers) should have no influence on the ductless siphon heights. This was experimentally verified for petroleum treated with 2 000 ppm ELASTOL<sup>T.M.</sup> and 100 ppm SEPAROL<sup>T.M.</sup> AF 27 (emulsion breaker). The same value of  $h_{max}$  as given in Table 4 for the pure petroleum was observed.

The drawability increase due to ELASTOL<sup>T.M.</sup> is of great advantage in collecting oil floating on a water surface by means of a vacuum skimmer. A field test with crude oil from ERM (= refinery Mannheim) spread on water in a training basin gave the following results: In the case of untreated oil 90% water and only 10% oil were collected. After the application of 6 000 ppm ELASTOL<sup>T.M.</sup> powder 95% oil and only 5% water were collected [8]. Fig.17 shows that the oil film is pulled from the water surface without rupture and can easily be sucked by the vacuum skimmer over a distance of about 15 cm.

\* registered trademark of BASF Aktiengesellschaft

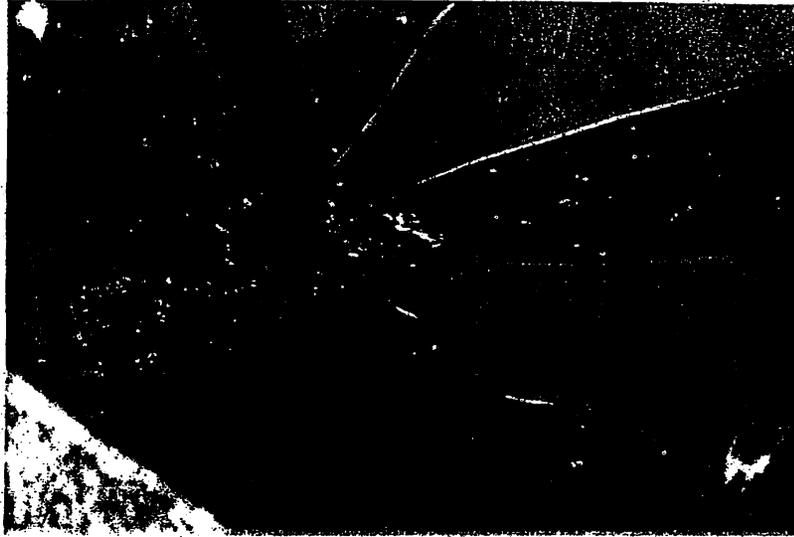
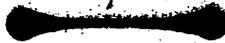
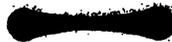
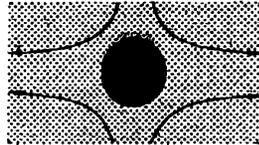


Fig.17: Example of the improved performance of a vacuum skimmer in collecting crude oil (ERM) from a water surface [8].

#### 5. Consequences of ELASTOL<sup>T.M.</sup> on droplet formation

Fig.18 shows schematically the break up of an oil droplet into two smaller droplets due to an elongational flow field. Once a neck is formed the neck is elongated until break of the strap. The formation of smaller droplets continues until the rheological forces can no longer overcome the stabilizing effect of the interfacial tension.

With dissolved macromolecules, however, it is possible to prevent the formation of very small droplets [6]. Here, the limiting drop size is governed by the resistance of the strap to stretching. When the elongational viscosity in the neck is increased the break up process will come to an end at a bigger droplet size.



**Fig.18:** Formation of smaller droplets in an elongational flow field (schematic).

The consequence for oil/water mixtures is demonstrated by Fig.19. Two glass bottles were partially filled with water. In the right bottle pure Heizöl was used whereas in the left bottle the oil was treated with 10 000 ppm ELASTOL<sup>T.M.</sup>. The closed bottles were shaken by hand for a given time period. After a settling time of about 10 seconds we observe in the right bottle a milky emulsion of very small oil droplets in water. The water and oil phases are hardly separated. In the case of the ELASTOL<sup>T.M.</sup> treated oil (right) we get a coarse emulsion by shaking. The separation of water and oil is nearly complete after 10 seconds.



Fig.19: Diesel oil/water mixtures after shaking and a settling time of 10 seconds. Pure oil (right), oil treated with 10 000 ppm ELASTOL<sup>T.M.</sup> (left).

### III. Dissolving behaviour of ELASTOL<sup>T.M.</sup> powder

#### a) General behaviour

The efficiency of ELASTOL<sup>T.M.</sup> in treating oil spills is strongly dependent on the dissolving speed of the powder after being spread onto oil layers floating on water. To simulate this situation on a laboratory scale a beaker of 150 mm diameter was partially filled with water. Oil was layered onto the water surface to a thickness of 5 mm. ELASTOL<sup>T.M.</sup> powder corresponding to 10 000 ppm of the weight of oil was "homogeneously" spread on the oil layer.

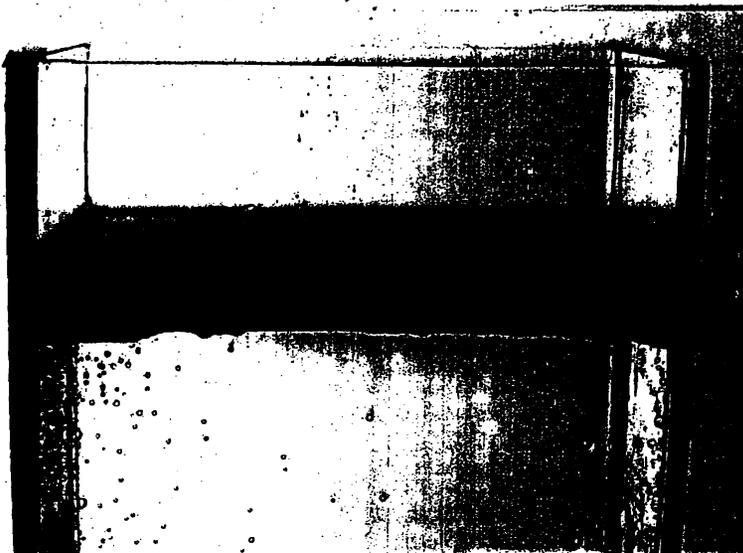


Fig.20: Side view of an oil layer floating on water. ELASTOL<sup>T.M.</sup> was spread on top of the oil and sedimented to the oil/water interface from where it dissolves.

After application, the powder rapidly sedimented to the water/oil interface (Fig.20). From there, the PIB component gradually dissolved in the oil phase, whereas the water-insoluble salt remained in particulate form at the interface. As a measure of the

effective solution concentration we determined the oil solution viscosity as a function of time. For this purpose small samples were withdrawn at different time intervals using a syringe. Precautions were taken not to collect undissolved PIB.

b) Solution viscosity with and without agitation

Figure 21 shows the solution viscosity versus logarithmic time as measured for fuel oil at a powder concentration of 10 000 ppm. In a first test series depicted by full circles the dissolving of ELASTOL<sup>T.M.</sup>.

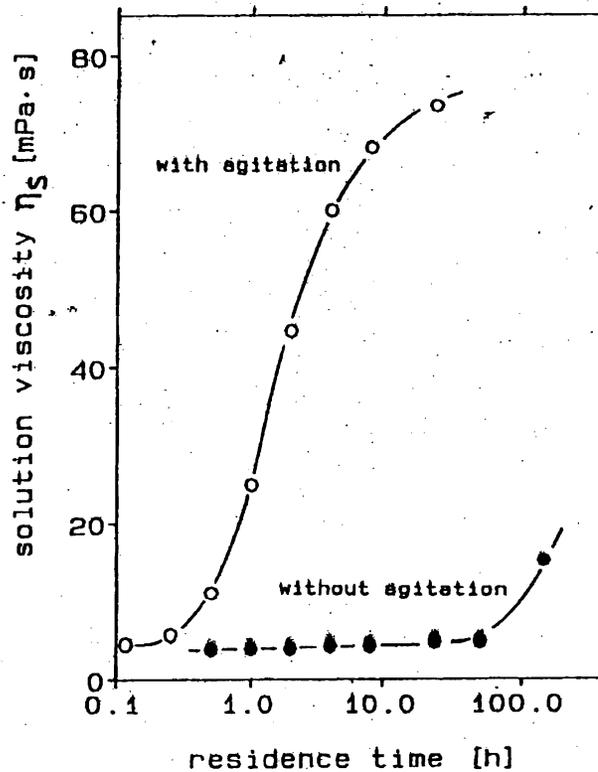
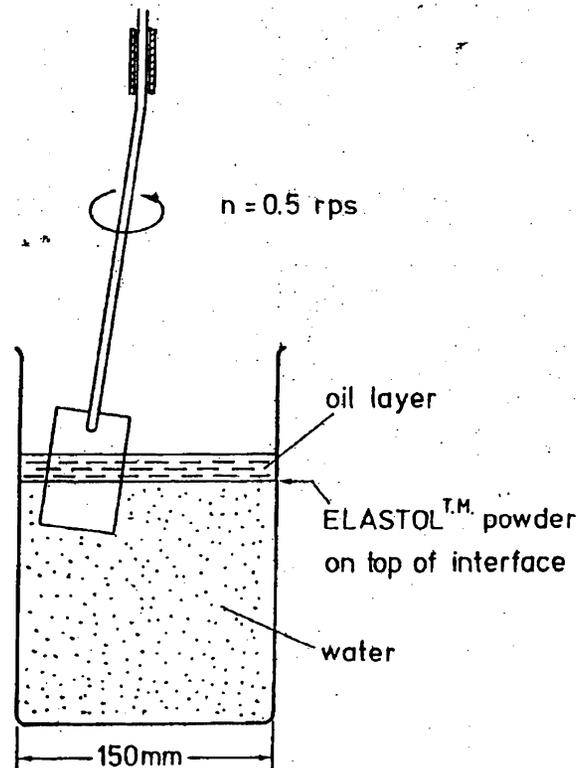


Fig.21: Relative solution viscosities of fuel oil layers (5 mm thickness) floating on top of water versus time elapsed after application of ELASTOL<sup>T.M.</sup> by spraying (concentration 10 000 ppm related to the total weight of oil) with (open circles) and without (full circles) gentle stirring.

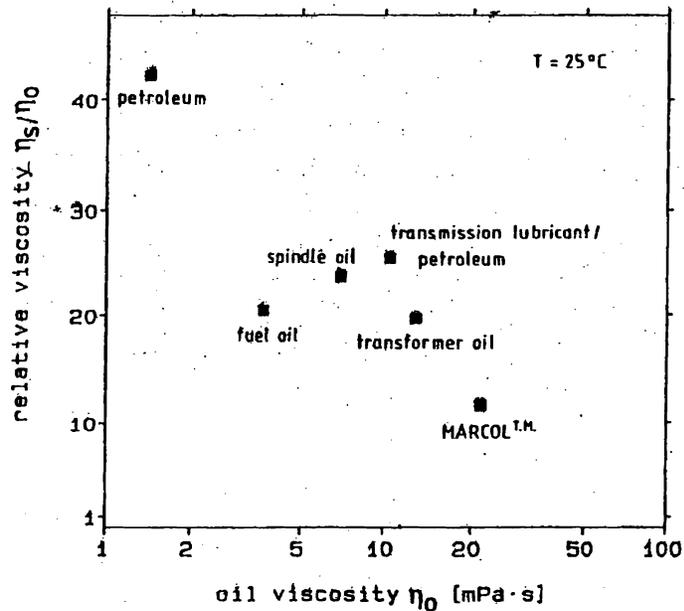
took place without any agitation. Obviously, in that case a significant increase of the solution viscosity is only observed at times greater than 100 hours.

In a second series of tests a gentle agitation was attained by stirring. A schematic drawing of the arrangement is shown in Figure 22. The rotary speed of the stirrer was 0.5 revolutions per second. As can be seen from Fig.21 the gentle agitation drastically increases the speed of dissolving (open circles). A significant increase in the oil solution viscosity was observed after only 0.5 hours.



**Fig.22:** Experimental arrangement for the dissolving tests with stirring.

Similar dissolving tests with stirring were performed with various oils. The relative solution viscosities  $\eta_r$  (solution viscosity related to the oil viscosity) versus the oil viscosity  $\eta_0$  are plotted in Figure 23. The data obtained by this method are more complex to interpret than those obtained by rolling (cf. Figs. 2 and 3). This is because partial evaporation of the oil has to be taken into account which is different for the various solvents. This explains why viscosities from the stirring experiment may be higher than those for solutions prepared by rolling in closed glass bottles.



**Fig. 23:** Relative viscosities of the various oil layers measured at the end of a 24 h stirring period. The initial powder concentration was 10 000 ppm related to the total weight of the oil on water.

c) Comparison of dissolving speeds

By comparing the viscosities determined for solutions prepared by rolling and the results obtained in the dissolving tests with gentle stirring (neglecting the influence of partial evaporation) we can evaluate the time necessary to dissolve an effective concentration of 2 000 ppm ELASTOL<sup>T.M.</sup> out of a total powder concentration of 10 000 ppm. The necessary times are plotted in Figure 24 as a function of the oil viscosities  $\eta_0$  (double-log representation). The increasing dissolving time is approximately proportional to the oil viscosity.

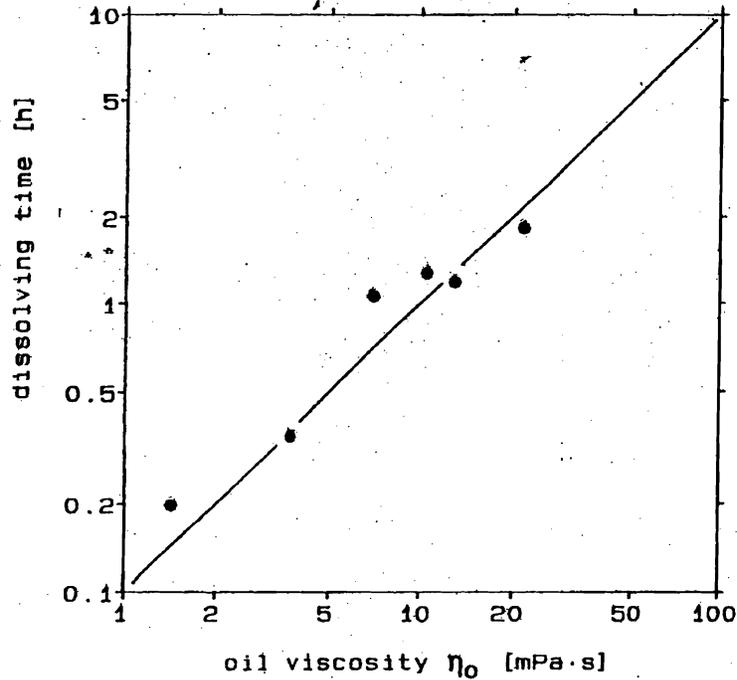


Fig.24: Dissolving time for 2 000 ppm ELASTOL<sup>T.M.</sup> out of an initial powder concentration of 10 000 ppm versus oil viscosity. The effect of partial evaporation during stirring is neglected here.

The data in Fig.24 can at least approximately be generalized to estimate the effect of temperature on the dissolving time. For instance, if the viscosity of the oil increases by a factor  $x$  when the temperature is lowered by  $\Delta T$  compared to room temperature, the necessary dissolving time of the powder will be longer by the same factor  $x$ .

d) Dissolving speeds at various concentrations

The effect of powder concentration on the speed of dissolving is demonstrated in Table 5 for petroleum. ELASTOL<sup>T.M.</sup>-concentrations of 10 000 ppm (as in Fig.24) and of 4 000 ppm were used. It is clearly seen that the necessary time to dissolve 20% of the initial powder is significantly shorter for the smaller concentration (11.8 and 8.4 min, respectively). This means that the dissolving times given in Fig.24 for 10 000 ppm powder cannot be generalized for other concentrations. However, since for practical applications the ELASTOL<sup>T.M.</sup> concentrations will presumably be lower, we can regard the data of Fig.24 to represent an upper limit of dissolving times for the relatively weak kind of agitation used in our tests.

Table 5: Comparison of dissolving times for two different ELASTOL<sup>T.M.</sup> concentrations in petroleum (evaporation neglected) with stirring as in Fig.22.

powder concentration [ppm]	dissolved fraction [ppm]	necessary dissolving time [min]	solution viscosity [mPa·s]
4 000	800	8.4*	1.93
4 000	4 000	39	5.51
10 000	800	6.0	1.93
10 000	2 000	11.8*	2.99
10 000	10 000	78	18.9

\* 20%-values

Another result that can be deduced from Table 5 concerns the dissolving time for a given absolute concentration of the solution, e.g. 800 ppm. Whereas for 4 000 ppm applied powder a dissolving time of 8.4 min is necessary the same concentration will be achieved within only 6.0 min if 10 000 ppm of powder are spread onto the oil layer. This result is not at all surprising but has to be kept in mind for practical applications.

#### IV. Water/crude oil emulsions

The formation of extremely stable water-in-crude oil emulsions, often called "chocolate mousse", is a major problem in combatting oil spills at sea. These emulsions are highly viscous and their water content can be as high as 90%. They are difficult to collect and even more difficult to dispose of. According to Bredie et al. [9] the formation of these mousses depends on the presence of both wax and asphaltenes in the crude oil. These authors suggest that wax/asphaltene crystals may stabilize small water droplets in the oil, leading to a dramatic increase in viscosity.

In order to produce "chocolate mousse" on a laboratory scale we performed experiments with various water-in-oil mixtures. In one series of tests different oil-water mixtures were treated with a shaker ("red devil") in closed bottles for 15 min. As expected the formation of a stable emulsion could not be observed for dewaxed and deasphaltenized oils like transformer oil or MARCOL<sup>T</sup>-M. 82. After a settling time of several minutes the water and oil phases separated. For the crude oils listed in Table 2, however, stable water-in-oil emulsions were easily produced by shaking. Even after a settling period of 24 hours no phase separation was observed.

The most stable "mousse" could be produced with Arabmed ( $\eta_0 = 18 \text{ mPa}\cdot\text{s}$ ) for which a sudden rise in viscosity of the emulsion was observed with increasing water content. For a 70% water-in-oil emulsion a viscosity of  $20 \text{ mPa}\cdot\text{s}$  was measured, whereas for 80% (90%) water content  $6.1$  ( $8.1$ )  $\text{Pa}\cdot\text{s}$  were observed.

When the same experiments were repeated on Arabmed treated with  $2\,000 \text{ ppm ELASTOL}^{\text{T.M.}}$  the sudden rise in viscosity in going from 70% to 80% water content was reduced by at least a factor of 4. For both 80% and 90% water content viscosities of about  $1.5 \text{ Pa}\cdot\text{s}$  were measured indicating that  $\text{ELASTOL}^{\text{T.M.}}$  improves the pumpability of "chocolate mousse".

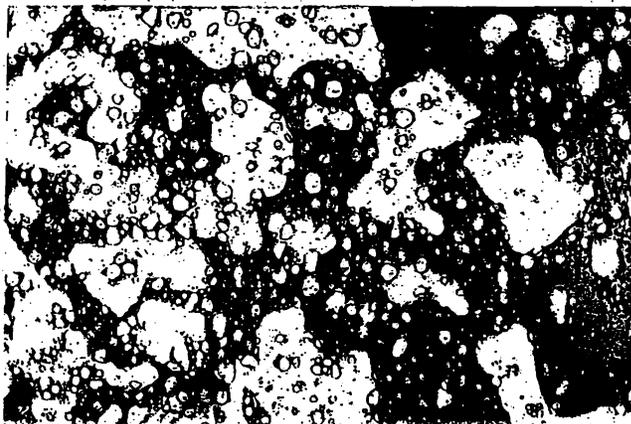


Fig.25: Stiff "chocolate mousse" obtained by shaking Arabmed with 90% water for 15 min in the red devil (right). Crude oil treated with  $2\,000 \text{ ppm ELASTOL}^{\text{T.M.}}$  (left).

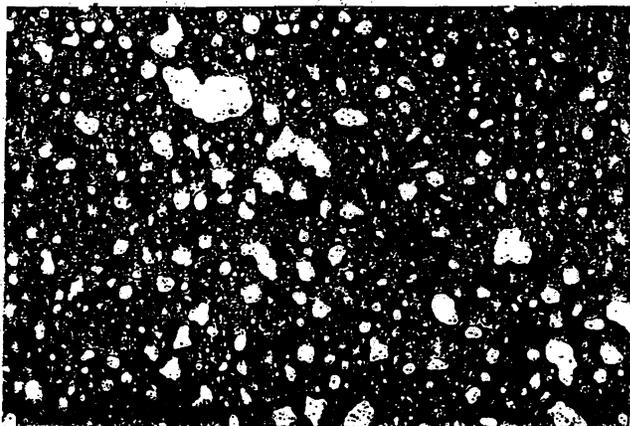
Fig.25 demonstrates that without  $\text{ELASTOL}^{\text{T.M.}}$  a stiff and stable emulsion with yield stress forms. When treated with  $\text{ELASTOL}^{\text{T.M.}}$ , however, the Arabmed/water mixture was fluid even at very low shear stresses and had to be kept in a container. Also a tendency to phase separation between water and oil was evident

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a)



b)



**Fig.26:** Light micrographs of 90% water-in-oil emulsions with Arabmed (magnification: 66 fold)  
a) without ELASTOL<sup>T.M.</sup>      b) with 2 000 ppm ELASTOL<sup>T.M.</sup>

Microscopic analysis of emulsions produced from Arabmed and 90% water was carried out (Fig.26). Panel (a) shows the "chocolate mousse" formed with untreated Arabmed whereas Panel (b) demonstrates the effect of inclusion of 2 000 ppm ELASTOL<sup>T.M.</sup>. Both pictures illustrate the water-in-oil character of this emulsion, but for ELASTOL<sup>T.M.</sup> treated Arabmed the size of the water droplets is significantly smaller.

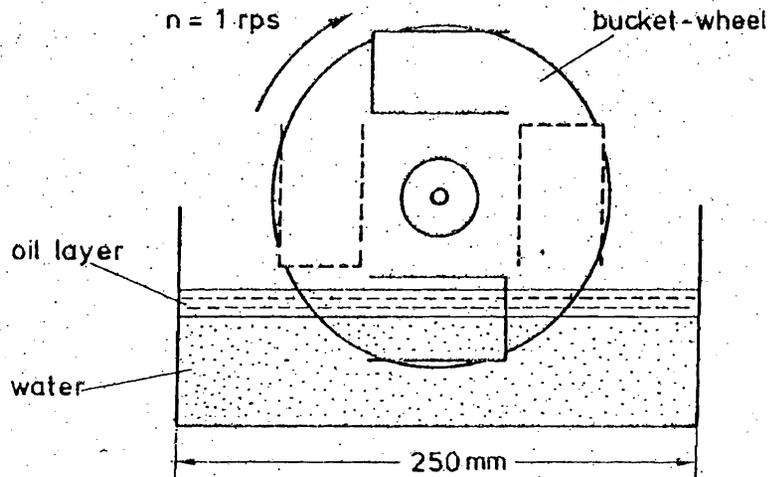


Fig.27: Experimental arrangement for the formation of "chocolate mousses" from crude oil layers floating on water.

In order to simulate the formation of "chocolate mousse" from crude oil layers floating on water more realistically, the apparatus schematically depicted in Fig.27 was used. Here the fluid was repeatedly picked up by a bucket-wheel and subsequently poured out onto the surface. In this arrangement the formation of stable emulsions was observed after stirring for less than 4 hours. After 4 hours the "chocolate mousse" formed with untreated Arabmed had a viscosity of 2 Pa·s.

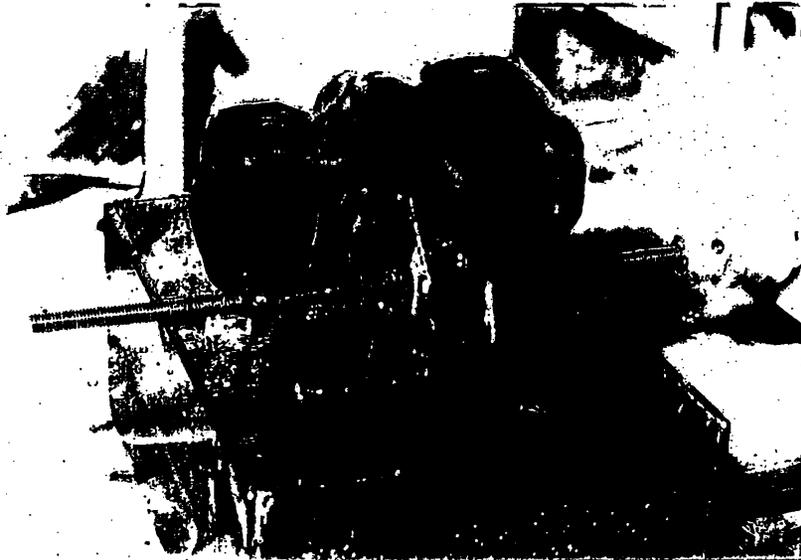


Fig.28: "Chocolate mousse" obtained with Arabmed in the bucket-wheel arrangement.

In addition, the bucket-wheel arrangement clearly shows the influence of evaporation. In Table 6 the emulsion viscosities measured after various stirring and evaporation periods are listed.

Table 6: Viscosities for "chocolate mousse" formed with Arabmed in the bucket-wheel arrangement (shear rate  $2 \text{ s}^{-1}$ ).

stirring time [h]	residence time [h]	$\eta_0$ [Pa·s] untreated crude oil	$\eta_0$ [Pa·s] with 2 000 ppm ELASTOLT.M.
4	4	2.0	1.4
8	32	7.8	2.0
12	100	12.2	2.7
16	104	5.4*	

\* Addition of 2 000 ppm ELASTOLT.M. to the "chocolate mousse" obtained from untreated Arabmed after 100 hours .

Stirring intervals of 4 hours and various rest periods have been applied. The first column gives the total stirring time and the second column the total residence time of Arabmed in the bucket-wheel test. For the untreated crude oil the first stirring interval yields a "chocolate mousse" of 2 Pa·s. With increasing residence time due to evaporation the "mousse" viscosity increased up to 12.2 Pa·s (100 h).

When 2 000 ppm ELASTOL<sup>T.M.</sup> powder were spread on the mousse a drop in the emulsion viscosity to 5.4 Pa·s was observed after another stirring period of 4 hours (last line in Table 6). This means that the pumpability of the emulsion is improved and the advantage of elasticity is obtained by application of ELASTOL<sup>T.M.</sup> even after the formation of a stiff "mousse". When ELASTOL<sup>T.M.</sup> was spread on the oil layer at the beginning of the test, viz. before the formation of a "chocolate mousse", the effect of viscosity reduction was even more pronounced (fourth column).

#### Acknowledgement

The authors are indebted to W. Reuther, J. Steidel and Ch. Kaduk for performing the measurements and to M. Reuther for the support in preparing the diagrams.

November 1987

## Appendix

### Results on ELASTOL<sup>T.M.</sup> treated oils obtained by other authors

#### a) Environmental Emergencies Technology Division, Ottawa

In an extensive study Bobra and Fingas et al. [10] investigated the effect of ELASTOL<sup>T.M.</sup> on eight different crude oils as well as diesel in both small scale laboratory experiments and large tank tests with and without waves.

In order to measure the elastic properties of ELASTOL<sup>T.M.</sup>/oil solutions these investigators made use of the die swell phenomenon: The diameter of a viscoelastic fluid extruded through a small capillary is greater than the die diameter, depending on the degree of elasticity of the solution. A distinct die swell was observed for ELASTOL<sup>T.M.</sup>/oil solutions. The effect increased with increasing mixing energy, powder concentration, and temperature.

ELASTOL<sup>T.M.</sup> had no effect on flash point and evaporation data.

It could be shown that the spreading of oil slicks on calm water is reduced by addition of larger amounts of ELASTOL<sup>T.M.</sup> (> 5 000 ppm). The reduction depends on the quantity of ELASTOL<sup>T.M.</sup> actually dissolved in the oil. In the case of Alberta Sweet Mixed Blend (ASMB) crude oil with  $\eta_0 = 9.2$  mPa·s and a powder concentration of 6 000 ppm the spreading was reduced to 1/3 of the value without ELASTOL<sup>T.M.</sup>

Concerning the formation of water-in-crude oil emulsions it is reported that ELASTOL<sup>T.M.</sup> has only a weak influence on the emulsion formation tendency, but the water content of the "chocolate mousse" is reduced considerably. For instance, 1 litre "Emulsifying Mix" (= ASMB/Bunker C 1:1) forms 10 litres of stable emulsion; after addition of 6 000 ppm ELASTOL<sup>T.M.</sup> only 4.3 litres are produced. This behaviour is to some extent still valid when ELASTOL<sup>T.M.</sup> is added after the emulsification process.

In small scale wave generating tank tests (35 litres content) it was found that ELASTOL<sup>T.M.</sup> treated oils have a lower fraction dispersed in the water phase than untreated oils. Furthermore, for most of the oils the water content of ELASTOL<sup>T.M.</sup> treated emulsions was significantly reduced (on average 65% less for 6 000 ppm). At the same time the increase in viscosity due to emulsification was strongly suppressed by ELASTOL<sup>T.M.</sup>.

The authors report that the effect of ELASTOL<sup>T.M.</sup> on viscoelasticity in the presence of salt water was only slightly smaller than with fresh water.

The application of a dispersant and ELASTOL<sup>T.M.</sup> at the same time reduced the dispersant effectivity by 70%, however, the amount of dissolved oil was still by a factor of 1 000 higher than without dispersant.

ELASTOL<sup>T.M.</sup> did not negatively affect the efficiency of an emulsion inhibitor applied at the same time.

Large scale tank tests with waves (75 l Norman Wells crude oil) showed that the dissolving speed of the ELASTOL<sup>T.M.</sup> powder is increased with growing wave height (mixing energy). Furthermore, it was observed that in the case of waves the powder may be spread in a random manner without disadvantages for the dissolving speed and the homogeneity of the final solution.

An oil spill of 75 l kept in booms (waves height 10 cm) could nearly completely (70 l) be recovered by a Morris MI-2C skimmer after the addition of 6 700 ppm ELASTOL<sup>T.M.</sup>.

The positive results of further tests with ELASTOL<sup>T.M.</sup> performed off-shore by M. Fingas (Environment Canada) will be published in the near future.

b) Berlin Model Basin, VWS Berlin

At the Versuchsanstalt für Wasserbau und Schiffbau (VWS) H.O. Gebius [11] performed containment tests in a 60 m long and 2.5 m wide basin. An amount of 5 l of oil was placed on one side of a boom which could be moved by a towing carriage at variable speeds. The quantity of interest was the boom velocity at which the first droplets appear on the backside of the boom (drainage failure).

In the case of calm water a concentration of only 1 000 ppm ELASTOLT.M. was sufficient to increase the critical velocity from 0.24 m/s to 0.48 m/s for an oil of  $\eta_0 = 22$  mPa·s viscosity (MACROLT.M. 82). For an oil of higher viscosity (PRIMOLT.M. 352,  $\eta_0 = 713$  mPa·s) the increase was not so pronounced, an effect which most probably is due to incomplete dissolving of the ELASTOLT.M. powder at the applied mixing time of 1 h.

With waves of wave length 1.5 m and 0.13 m wave height a critical boom velocity of about 0.30 m/s was measured on MARCOLT.M. 82. The application of 1 000 ppm and 3 000 ppm ELASTOLT.M. increased the critical velocity up to 0.36 and 0.42 m/s, respectively.

Acknowledgement

The authors are grateful to Dr. M. Fingas, Environment Canada, for making test results available prior to publication.

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**Attachment 13**

**Oil Dispersant Toxicity Test**





**United States Testing Company, Inc.**  
Biological Services

1415 Park Avenue  
Hoboken, New Jersey 07030  
Tel: 201-792-2400  
Fax: 201-658-0636

March 17, 1992

Jack Scambos  
Environmental Recovery Resources  
35 Smith Ridge Road  
South Salem, New York 10590

Dear Mr. Scambos:

This letter is with regard to our conversation earlier today. You were interested in further interpretation of testing performed by USTC versus your product "Elastol". The study you were concerned with was performed for General Technology Applications, Inc. in March and April of 1987 (USTC Report #06589). You wanted an interpretive summary, since none was given with the report. The numerical summary follows:

Toxicant	Artemia 48hr LC50	Fundulus 96hr LC50
Elastol	>18,000 ppm	>18,000 ppm
Elastol + Oil	>3,200 ppm	>18,000 ppm
#2 Fuel Oil	600 ppm	3,200 ppm
DSS (ref tox)	12 ppm	18 ppm

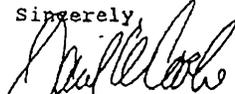
Review of the report indicates that the product demonstrated no acute toxicity to either Artemia salina (brine shrimp), or Fundulus heteroclitus (Killi fish), at exposure levels up to 18,000 ppm (the highest product concentration tested).

When mixed with #2 Fuel Oil (1:10 ratio of product to oil), the product demonstrated no toxicity to A. salina at an exposure level of 3200 ppm. At 3200 ppm, the product/oil mixture contained 320 ppm Elastol and 2,880 ppm #2 Fuel Oil. Since the toxicity of #2 Fuel Oil was determined to be 600 ppm, Elastol caused an apparent toxicity reduction.

When mixed with #2 Fuel Oil, the product demonstrated no toxicity to F. heteroclitus at an exposure level of 18,000 ppm. At 18,000 ppm, the product/oil mixture contained 1,800 ppm Elastol and 16,200 ppm #2 Fuel Oil. Since the toxicity of #2 Fuel Oil was determined to be 3,200 ppm, Elastol caused an apparent toxicity reduction.

Bear in mind that I was not involved in the above mentioned testing, and can only interpret what was reported. If you have any questions, or if I can be of further service, do not hesitate to call me.

Sincerely,

  
Daniel Cooke  
Mgr, Ecotoxicology



EST. 1880

# United States Testing Company, Inc.

## Biological Services Division

1415 PARK AVENUE • HOBOKEN, NEW JERSEY 07030 • 201-792-2400

### REPORT OF TEST

EPA Standard Dispersant  
Toxicity Tests  
of  
ELASTOL

Conducted for:

General Technology Applications Inc.  
7720 Mason King Court  
Manassas, Virginia 22110

April 14, 1987

TEST REPORT NO. 06589

SIGNED FOR THE COMPANY

Prepared by:

  
Robert Lippencott  
Biologist

V.P., Mgr, Biological Services Division

BY

  
Daniel Drozdowski

Laboratories in: New York • Chicago • Los Angeles • Houston • Tulsa • Memphis • Reading • Richland

THIS REPORT APPLIES ONLY TO THE STANDARDS OR PROCEDURES IDENTIFIED AND TO THE SAMPLE(S) TESTED. THE TEST RESULTS ARE NOT NECESSARILY INDICATIVE OR REPRESENTATIVE OF THE QUALITIES OF THE LOT FROM WHICH THE SAMPLE WAS TAKEN OR OF APPARENTLY IDENTICAL OR SIMILAR PRODUCTS. NOTHING CONTAINED IN THIS REPORT SHALL MEAN THAT UNITED STATES TESTING COMPANY, INC., CONDUCTS ANY QUALITY CONTROL PROGRAM FOR THE CLIENT TO WHOM THIS TEST REPORT IS ISSUED, UNLESS SPECIFICALLY SPECIFIED. OUR REPORTS AND LETTERS ARE FOR THE EXCLUSIVE USE OF THE CLIENT TO WHOM THEY ARE ADDRESSED, AND THEY AND THE NAME OF THE UNITED STATES TESTING COMPANY, INC. OR ITS SEALS OR INSIGNIA, ARE NOT TO BE USED UNDER ANY CIRCUMSTANCES IN ADVERTISING TO THE GENERAL PUBLIC AND MAY NOT BE USED IN ANY OTHER MANNER WITHOUT OUR PRIOR WRITTEN APPROVAL. SAMPLES NOT DESTROYED IN TESTING ARE RETAINED A MAXIMUM OF THIRTY DAYS.

A Member of the SGS Group (Societe Generale de Surveillance)

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

Project: EPA Revised Standard Oil Dispersant Toxicity Test  
(July 1984)

Sample: An oil coagulant submitted and identified by the Client as:

Elastol (fine white powder)

Procedure: Tests were in accordance with the EPA "Revised Standard Dispersant Toxicity Test", Federal Register, 49:139, p. 29204-29207.

Note: When blended and mixed with the salt water test medium, as specified by the EPA method, Elastol remained largely undissolved. Elastol-oil combinations formed into a sticky, rubbery substance.

Summary of Results:

<u>Toxicant</u>	<u>Artemia salina</u>		<u>Fundulus heteroclitus</u>	
	<u>48 hour LD<sub>50</sub> (ppm)</u>	<u>95% CI</u>	<u>96 hour LD<sub>50</sub> (ppm)</u>	<u>95% CI</u>
Elastol	>18,000	N/A	>18,000	N/A
#2 fuel oil	600		3200	
1:10 mixture, Elastol and #2 fuel oil	>3200	N/A	>18,000	N/A
DSS (reference toxicant)	12		18	

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

**Toxicity Testing Results**

Sample: Elastol

Test Dates: 3/26 - 3/30/87

Organism: Fundulus heteroclitus

Test Conc. (ppm)	% Mortality (hours)				Initial		Final	
	24	48	72	96	D.O.	pH	D.O.	pH
Control	0	0	0	0	7.0	7.8	7.0	7.9
10,000	0	0	0	0	7.0	7.8	4.0	8.0
18,000	0	0	0	0	7.0	7.8	4.0	7.9

Temperature: 22°C

Dilution water: Artificial Sea Water

Salinity: 20 ppt

Test organism: Source: Scientific Suppliers  
x̄ weight: 1 - 1.5 g  
x̄ length: 40 - 50 mm  
no./conc: 10

Summary:

LC<sub>50</sub>: >18,000 ppm

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

**Toxicity Testing Results**

Sample: Elastol #2 Fuel Oil (10:1)

Test Dates: 3/26 - 3/30/87

Organism: Fundulus heteroclitus

<u>Test Conc.</u> (ppm)	<u>% Mortality (hours)</u>				<u>Initial</u>		<u>Final</u>	
	24	48	72	96	<u>D.O.</u>	<u>pH</u>	<u>D.O.</u>	<u>pH</u>
Control	0	0	0	0	7.0	7.8	7.0	7.9
10,000	10	10	10	10	7.0	7.8	4.0	7.9
18,000	10	10	10	10	7.0	7.8	4.0	7.9

Temperature: 22°C

Dilution water: Artificial Sea Water

Salinity: 20 ppt

Test organism: Source: Scientific Suppliers  
x weight: 1 - 1.5 g  
x length: 40 - 50 mm  
no./conc: 10

LC<sub>50</sub>: >18,000 ppm

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

Toxicity Testing Results

Sample: Elastol

Test Dates: 3/26 - 3/28/87

Organism: Artemia salina

<u>Test Conc.</u> (ppm)	<u>% Mortality (hours)</u>		<u>Initial</u>		<u>Final</u>	
	<u>24</u>	<u>48</u>	<u>D.O.</u>	<u>pH</u>	<u>D.O.</u>	<u>pH</u>
Control	0	0	7.0	7.8	7.0	8.0
10,000	0	0	7.0	7.8	7.0	8.0
18,000	0	0	7.0	7.8	7.0	8.0

Temperature: 22°C

Dilution water: Artificial Sea Water

Salinity: 20 ppt

Test organism: Source: In-house culture  
age : 24 - 48 hrs  
no./conc: 100

Summary:

LC<sub>50</sub>: >18,000 ppm

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

Toxicity Testing Results

Sample: Elastol 1:10 with #2 Fuel Oil

Test Dates: 4/3 - 4/5/87

Organism: Artemia salina

<u>Test Conc.</u> (ppm)	<u>% Mortality (hours)</u>		<u>Initial</u>		<u>Final</u>	
	24	48	D.O.	pH	D.O.	pH
Control	0	0	8.0	7.8	6.5	7.9
32	3	3	8.0	7.8	6.5	7.9
100	0	0	8.0	7.8	6.5	7.9
320	0	0	8.0	7.8	6.5	7.9
1000	0	0	8.0	7.8	6.5	7.9
3200	0	3	8.0	7.8	6.5	7.9

Temperature: 21°C

Dilution water: Artificial Sea Water

Salinity: 22 ppt

Test organism: Source: In-house culture  
age : 24 - 48 hrs  
no./conc: 100\*

\*Note: n=60 organisms/conc. were used for confirmation tests.

Summary:

LC<sub>50</sub>: >3,200 ppm

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

**Toxicity Testing Results**

Sample: No. 2 Fuel Oil alone

Organism: Fundulus heteroclitus

Test Dates: 3/5/87 - 3/9/87

Test Conc. (ppm)	Percent Mortality (hours)				Initial		Final	
	(24)	(48)	(72)	(96)	D.O.	pH	D.O.	pH
Control (0)	0	0	0	0	9.0	7.7	8.0	7.6
100	0	0	0	0	8.8	7.6	8.0	7.6
1000	0	0	0	0	8.6	7.6	8.0	7.6
10000	0	0	70	100	8.0	7.6	7.0	7.6

Temperature: 19±1°C

Dilution water: Artificial Sea Water

Salinity: 20 0/00

Test organism: Source: Scientific Suppliers (Massachusetts & N.H.)

$\bar{x}$  weight: 1.0-1.5g

$\bar{x}$  length: 45 mm

no./conc.: 10

Summary:

LC<sub>50</sub>: 3,200 ppm

Laboratory Note: The above test was run for reference purposes only. In five separate tests conducted previous to this project, dating from 1983 to 1986, the toxicity of #2 fuel oil to Fundulus has been reported as >1,000 ppm to >10,000 ppm. At 10,000 ppm (1% in water) there is a significant oil slick which can directly adhere to and irritate swimming organisms.

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

**Toxicity Testing Results**

Sample: No. 2 Fuel Oil only

Organism: Artemia salina

Test Dates: 3/6/87 - 3/8/87

<u>Test Conc.</u> <u>(ppm)</u>	<u>Percent Mortality</u> <u>(hours)</u>		<u>Initial</u>		<u>Final</u>	
	<u>(24)</u>	<u>(48)</u>	<u>D.O.</u>	<u>pH</u>	<u>D.O.</u>	<u>pH</u>
Control (0)	0	0	7.0	7.6	7.0	7.6
100	0	20	7.0	7.6	7.0	7.7
1000	0	70	7.0	7.6	7.0	7.5
10000	0	100	7.0	7.6	7.0	7.4

Observation: Physical entrapment of organisms in surface oil contributed to mortality.

Temperature: 21±1°C

Dilution water: Artificial Sea Water

Salinity: 20 0/00

Test organism: Source: San Francisco Bay Type (cysts)  
age : newly hatched lab culture  
no./conc.: 100

Summary:

LC<sub>50</sub>: Approximately 600 ppm (range 1000 - 10,000)

Laboratory Note: The above test was run for reference purposes only. In five separate tests conducted previous to this project dating from 1983 to 1986, the toxicity of #2 fuel oil to Artemia has been reported in the range of 600 ppm to >3200 ppm. The organism is very small (less than 1mm) and thereby susceptible to scavenging by oil droplets or slicks. Mortality is not always attributable to chemical toxicity.

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/87

**Toxicity Testing Results**

Sample: Dodecyl sodium sulfate (reference material)

Organism: Fundulus heteroclitus

Test Dates: 3/5/87 - 3/9/87

Test Conc. (ppm)	Percent Mortality (hours)				Initial		Final	
	(24)	(48)	(72)	(96)	D.O.	pH	D.O.	pH
1	0	0	0	0	9.0	7.1	7.8	7.0
3.2	0	0	0	0	8.8	7.2	7.8	7.1
10*	0	0	0	0	8.8	7.2	7.6	7.1
32	100	-	-	-	9.0	7.1	7.8	7.1
100	100	-	-	-	9.0	7.2	8.6	7.2

\*10 ppm exhibited stress, but no death

Temperature: 20±1°C

Dilution water: Artificial Sea Water

Salinity: 20 0/00

Test organism: Source: Scientific Suppliers (Massachusetts & N.H.)

x weight: 1.0-1.5g

x length: 45 mm

no./conc.: 10

Summary:

LC<sub>50</sub>: 18 ppm

Laboratory Note: The above test was run for reference purposes only. In five separate tests conducted previously, dating from 1983 to 1986, the toxicity of DSS to Fundulus has been reported as 2.7 ppm ± 1.4 ppm.

**United States Testing Company, Inc.**

Client: Gamlen Chemical (Division of Sybron)

06589  
4/14/87

**Toxicity Testing Results**

Sample: Dodecyl sodium sulfate (reference material)

Organism: *Artemia salina*

Test Dates: 3/11/87 - 3/13/87

<u>Test Conc.</u> (ppm)	<u>Percent Mortality</u> (hours)		<u>Initial</u>		<u>Final</u>	
	<u>(24)</u>	<u>(48)</u>	<u>D.O.</u>	<u>pH</u>	<u>D.O.</u>	<u>pH</u>
1	0	15	7.0	7.8	7.0	7.8
3.2	0	25	7.0	7.8	7.0	7.8
10	0	45	7.0	7.8	7.0	7.8
32	15	60	7.0	7.9	7.0	7.9

Temperature: 21±1°C

Dilution water: Artificial Sea Water

Salinity: 20 0/00

Test organism: Source: San Francisco Bay Type (cysts)  
age : newly hatched lab culture  
no./conc.: 100

Summary:

LC<sub>50</sub> (and 95% confidence limits): 12 ppm (4.8 - 30 ppm)

Laboratory Note: The above test was run for reference purposes only. In five separate tests conducted previously, dating from 1983 to 1986, the toxicity of DSS to *Artemia* has been reported as 4.5 ppm ± 2.7 ppm.

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/8

Project: EPA Revised Standard Oil Dispersant Toxicity Test  
(July 1984)

Sample: An oil coagulant submitted and identified by the Client as:  
Elastol (fine white powder)

Procedure: Tests were in accordance with the EPA "Revised Standard Dispersant Toxicity Test", Federal Register, 49:139, p. 292 29207.

Note: When blended and mixed with the salt water test medium, as specified by the EPA method, Elastol remained largely undissolved. Elastol-oil combinations formed into a sticky, rubbery substance.

Summary of Results:

<u>Toxicant</u>	<u>Artemia salina</u>		<u>Fundulus heteroclitus</u>	
	<u>48 hour LD<sub>50</sub> (ppm)</u>	<u>95% CI</u>	<u>96 hour LD<sub>50</sub> (ppm)</u>	<u>95% CI</u>
Elastol	>18,000	N/A	>18,000	N/A
#2 fuel oil	600		3200	
1:10 mixture, Elastol and #2 fuel oil	>3200	N/A	>18,000	N/A
DSS (reference toxicant)	12		18	

**United States Testing Company, Inc.**

Client: General Technology Applications Inc.

06589  
4/14/8

Project: EPA Revised Standard Oil Dispersant Toxicity Test  
(July 1984)

Sample: An oil coagulant submitted and identified by the Client as:  
Elastol (fine white powder)

Procedure: Tests were in accordance with the EPA "Revised Standard Dispersant Toxicity Test", Federal Register, 49:139, p. 292 29207.

Note: When blended and mixed with the salt water test medium, as specified by the EPA method, Elastol remained largely undissolved. Elastol-oil combinations formed into a sticky, rubbery substance.

Summary of Results:

<u>Toxicant</u>	<u>Artemia salina</u>		<u>Fundulus heteroclitus</u>	
	<u>48 hour LD<sub>50</sub> (ppm)</u>	<u>95% CI</u>	<u>96 hour LD<sub>50</sub> (ppm)</u>	<u>95% CI</u>
Elastol	>18,000	N/A	>18,000	N/A
#2 fuel oil	600		3200	
1:10 mixture, Elastol and #2 fuel oil	>3200	N/A	>18,000	N/A
DSS (reference toxicant)	12		18	

**Attachment 14**

**Abalone Larval Development Test Memorandum**



## MEMORANDUM

TO: All  
FROM: Al Hadermann  
DATE: June 29, 1992  
SUBJECT: Abalone Larval Development Test

I read "Abalone Larval Development Short-Term Toxicity Test Protocol," which I received from the California State Water Resources Control Board. This information can be used to better understand the significance of the results on Elastol.

### The Organism

Male and female abalone (7-10 cm length) are used to provide fertilized eggs under controlled conditions. Eggs are placed in saline solutions, containing potential toxicants, before the first division occurs. Thus, all division and growth of the single cell embryo to a trochophore larva, which hatches and develops into a veliger larva, occurs in the presence of the potential toxicant. This all occurs in 48 hours. At the end of 48 hours, formalin is used to fix the larvae. The larvae are then individually inspected for evidence of normal and abnormal development. Although not inclusive, abnormal development involves shell development defects.

### Toxicity Determination

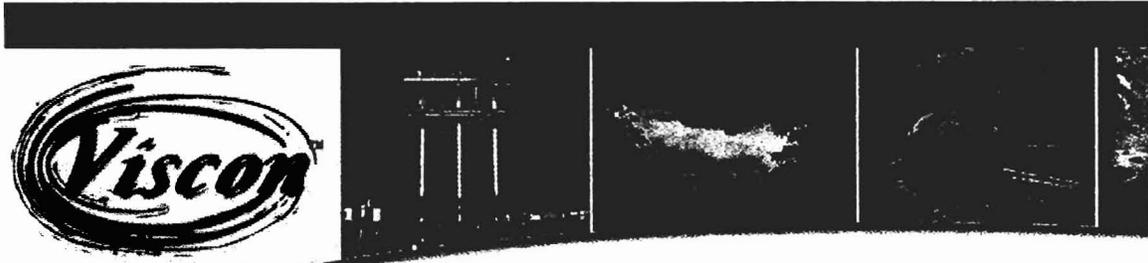
Toxicity is expressed as % of larvae with normal development. A control laboratory sample, with no added toxicant, typically can show 83.4% normally developed larvae. A reference toxicant containing 18 micrograms/liter of zinc ion could show 77.6% normally developed larvae, which is not statistically significant. On the other hand, at 32 micrograms of zinc ion/liter, only 6.6% of normally developed larvae were seen. The no effect concentration (NOEC) in this case is 18 micrograms/liter of zinc ion, while the low effect concentration (LOEC) concentration is 32.

All of the following Elastol extract concentrations showed fewer abnormally developed abalone larvae than either the control-lab or blank samples run at the same time: 0.1, 1.0, 3.2, 5.6, 10.0, 18.0, 32.0 and 56.0%. None of these Elastol samples differed significantly from the percent of larvae with normal development in the reference samples. Actually, however, all Elastol results were slightly less toxic than the "non-toxic" references. No other Elastol extract tests were run between 56.0% and 100.0%. Since the 100.0% test suggests that there was no "saline habitat" for the larvae, the number of abnormal larvae may be an artifact of the test.

### Conclusion

Elastol did not interfere with the normal development of abalone larvae. The test at 100.0% was forced beyond the characteristics of the medium to support the organisms. Therefore, the NOEC of 56.0% and the LOEC of 100.0% are conservative.

**Attachment 15**  
**2008 Power Test**



## *Cleaning Tomorrow's Air Today*

After review of the post durability test dated January 4, 2008, comments coming back from C.A.R.B. indicated questions about engine power. It appeared that there were reductions in the average H.P. specifically the first mode. Viscon California was instructed to have a Caterpillar field technician inspect the engine. After inspecting the engine the technician advised Viscon California that the turbo charger was leaking oil into the combustion chamber and due to leaking seals the engine would not make full power. The following report is a test performed that will show a return to full power with a rebuilt turbo charger installed

## **Attachment 16**

# **DTSC & OEHHA/ATEB QUESTIONS & ANSWERS**



Linda S. Adams  
Secretary for  
Environmental Protection



## Department of Toxic Substances Control

Maureen F. Gorsen, Director  
1001 "I" Street  
P.O. Box 806  
Sacramento, California 95812-0806



Arnold Schwarzenegger  
Governor

### MEMORANDUM

TO: Bob Okamoto  
Air Pollution Specialist  
Air Resources Board

FROM: Li Tang   
Multimedia Products Section  
Department of Toxic Substances Control

SUBJECT: Comments on Viscon Multi Media Evaluation (Tier II) Dated November 14, 2008.

DATE: December 23, 2008

Staff of the Department of Toxic Substances Control (DTSC) reviewed the November 14, 2008 Viscon Multi Media Evaluation (Tier II) prepared by Las Palmas Oil and Dehydration Company. According to the draft "Guidance Document and Recommendations on the Types of Scientific Information to Be Submitted by Applicants for California Fuels Environmental Multimedia Evaluations", a Tier I report must develop a work plan and a Tier II report must develop and review the risk assessment protocol for future actions and reports. In general, DTSC found this Tier II report acceptable. However, the response regarding the analysis of the spill release into soil of Viscon diesel in Attachment No. 16 did not adequately address the issue in DTSC's comment #3 dated May 21, 2007.

To further clarify DTSC's comments on the Tier I report, DTSC adds the following explanations and requests regarding the spill of diesel containing Viscon onto soil. DTSC understood that Viscon is an effective additive to reduce air emissions (page 3 of Tier II). It does not contain toxicity (page 4 of Tier II). However, if the Viscon diesel accidentally spilled onto soil, the contaminated soil could be hazardous because diesel contains hazardous components<sup>1</sup>. The Tier II report stated that the Viscon additive may prevent the diesel dispersion through underground soil after a spill due to its high

<sup>1</sup> Ultra Low Sulfur Diesel Material Safety Data Sheet  
<http://www.gwec.com/media/EDocs/UltraLowSulfurDiesel1.pdf>

viscosity property (page 4 of Tier II). Since there are no supporting references, please provide reference(s) or actual test data to support the above statement<sup>2</sup>.

Since Viscon is highly viscous (page 4 of Tier II), it is unknown how the existence of Viscon may impact cleanup procedures of the Viscon diesel-contaminated soil. The common cleanup processes for diesel-contaminated soil include soil excavation<sup>3</sup>, thermal treatment<sup>4</sup>, and bioremediation<sup>5,6</sup>. It is also unknown if the increase of viscosity of diesel affects the cleanup cost or technical process. Therefore, DTSC suggests that the company provide literature references if available, or conduct tests on the properties of diesel-contaminated soil with and without Viscon; such as soil density, soil-diesel absorption ratio, and heat removal rate and biodegradability of diesel in soil. Then, provide an analysis of the positive and negative effects on cleanup processes based on actual data.

DTSC has additional comments as follows:

1. The first page of the Tier II report Part I states, "Viscon is used at less than 5 part per millions (ppm) UHMWPIB to the end use CARB diesel." Part II-B states, "It is used at a dose level in diesel fuel of about 500 ppm." Please clarify the concentration level difference.
2. Some attachments in the report were published in the early or late 80's. For example, Attachments 6, 9, 10, and 11 indicate that UHMWPIB has been used as an oil spill agent. If these attachments are used to prove that Viscon is not toxic and has a long history of safe usage in the environment, please provide updated information. Is polyisobutylene still used as an oil collection agent currently? If not, are there any safety issues involved in the updated information?

---

<sup>2</sup> One example of experiment protocol is provided here for your reference, which determined mechanism affecting the infiltration and distribution of fuel in the vadose zone. (Cory J. McDowell and Susan E. Powers, "Mechanism Affecting the Infiltration and Distribution of Ethanol-Blended Gasoline in the Vadose Zone", *Environ. Sci. Technol.* 2003, 1803-1810)

<sup>3</sup> Procedures for the Remediation of Diesel & Gasoline Spills,  
<http://www.deq.state.ok.us/factsheets/land/Dieselspill.pdf>

<sup>4</sup> Remediation Technologies Screening Matrix and Reference Guide, Version 4.0,  
<http://www.frtr.gov/matrix2/section4/4-26.html>

<sup>5</sup> Principles and Practices for Diesel Contaminated Soils, Volume 4,  
<http://www.aehs.com/publications/catalog/contents/pvol4.htm>

<sup>6</sup> Bioremediation (Natural Attenuation and Biostimulation) of Diesel-Oil-Contaminated Soil in an Alpine Glacier Skiing Area  
R. MARGESIN\* AND F. SCHINNER

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Received 18 December 2000/Accepted 24 April 2001, <http://aem.asm.org/cgi/reprint/67/7/3127.pdf>

December 23, 2008

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3. Please clarify why the usage as an oil collection agent is relevant to Viscon's multimedia risk assessment, such as aquatic toxicity.
4. Attachments 7 and 12 are about viscosity tests. Please explain how the results of these tests support the fuel multimedia risk assessment.

If you have any questions, please contact me at (916) 322-2505, or [ltang@dtsc.ca.gov](mailto:ltang@dtsc.ca.gov).  
Thank you.

cc: Sonia Low, Supervisor  
Multimedia Products Section  
Office of Pollution Prevention and Green Technology  
Department of Toxic Substances Control

Sherri Lehman, Chief  
Toxics in Products Branch  
Office of Pollution Prevention and Green Technology  
Department of Toxic Substances Control

## OEHHA/ATEB COMMENTS ON VISCON TIER II REPORT (12/08)

1. TAB 8, Table 1: WTD AVG. Total PM for Test No. 8MLP49 = 0.210 and Averages (BL After Viscon) = 0.268. But, in Table 1A: 8MLP49 Total PM = 0.282 and 5-Test Average = 0.282. **That was a typo on table 1A**
2. Table 2, comparing VOC emissions, what does <RL mean for butadiene?  
**Reporting limit**
3. Regarding the measurements of PM, where's the PM sampling protocol (SOP)?
  - a. Was the PM separated by size? If so, what is the range of PM sizes captured on the filters, and what is the relative efficiency for each size range: PM 10, PM 2.5, ultrafine? **The test protocol called for PM 10 only.**
  - b. Is there any difference in the particle sizes produced with Viscon vs ULSD? A substantial increase in ultrafines would be source of concern even if the total PM decreases. **The protocol did not ask to define particulate size.**
4. OEHHA raised a concern with question 3, dated 2/2/07 (TAB 16), regarding the significant increases in some carbonyls and VOCs (e.g., 43% increase in benzene, 23% increase in formaldehyde, 26% increase in acetaldehyde, and 77% increase in acrolein). According to Viscon's response, "The decrease in PM and CO<sub>2</sub> more than offset these increases. Although the percentage change would appear large, the g/bhp-hr are a very small number compared to the tons of reductions in PM and CO<sub>2</sub>." However, in TAB 8, Summary of Data, the decrease of 24.72% in PM appears to be calculated from a decrease of 0.0655 g/bhp-hr in PM. By that reasoning, would the data suggest that the 0.003922 g/bhp-hr increase in benzene (43%), 0.00621 g/bhp-hr increase in formaldehyde (23%), and the 0.004246 g/bhp-hr increase in 1,3-butadiene (1682%; TAB 8, Table 3) also result in tons of these compounds being added to the atmosphere as a result of the addition of Viscon, since there is only a 10-17-fold difference in numbers used to calculate the offset? How was it determined that the decreases in PM and CO<sub>2</sub> would offset the increases in VOCs and carbonyls with respect to impacts on human health? **That was my error in not including the post durability results in the Tier 2 Report. The results are in the final report date January 11, 2008. As with the improvements of PM and NOX using Viscon over time improved the results of carbonyls and VOC's. See attachment.**
5. Attachment 12 is labeled Toxicity and Soil Penetration. The physical properties of Elastol and described, but no discussion of toxicity. Where are the toxicity data, Tab 13? **Attachment 12 should just read Soil Penetration.**

**Subject: Response to Memorandum DTSC Comments**

1. DTSC wants to know the basis of our claim that “Viscon additive may prevent the diesel dispersion through underground soil after a spill due to its high viscosity property.”

**The company stated that under conditions of shear stress CARB diesel treated with Viscon has an immediate and temporary increase in viscosity and that this increase in viscosity could restrict travel of the fuel in soil. This is based upon the commonly known phenomenon that increased viscosity of a liquid reduces its rate of penetration through a porous substance.**

2. DTSC suggests on page 1 that we should conduct laboratory tests on the properties of contaminated soil samples, with and without Viscon.

**The ultra high molecular weight PIB content in treated CARB diesel is about 5 PPM. The company believes that it is not reasonable to require laboratory tests to determine whether the addition of 5 PPM of a C<sub>4</sub>H<sub>8</sub> hydrocarbon to CARB diesel would have a significant effect on the density of soil contaminated with diesel, on diesel absorption ratio of the contaminated soil, on heat removal rate or biodegradability of the contaminated sample.**

3. **Question # 1 from Memorandum dated 12 2 08** DTSC wants clarification of the difference between 500 PPM dose rate of Viscon and 5 PPM of ultra high molecular weight PIB.

**Viscon is used at a dose rate in CARB diesel of about 500 PPM. Viscon itself is comprised of 1 part ultra high molecular weight PIB and 99 parts CARB diesel. The ultra high molecular weight PIB content of Viscon treated diesel is about .01 x 500 PPM = 5 PPM.**

4. **Question # 2 from Memorandum dated 12 2 08** DTSC assumes that attachments 6, 9, 10 and 11 are for the purpose of proving that Viscon is not toxic and asks for additional history on the use of ultra high molecular weight PIB as an oil spill agent.

**Attachment 6 was included as background information. It was considered relevant because it represented past evaluation of ultra high molecular weight PIB as a substance acceptable for use in California waters.**

**Attachments 9, 10 and 11 were included as background information showing history of the use of ultra high molecular weight PIB in environmental clean up operations.**

**The company which owns the Viscon technology was spun off about 10 years ago from GTA, the company responsible for oil spill applications of ultra high molecular weight PIB. We have no information on current use of their oil spill technology.**

5. **Question # 3 from Memorandum dated 12 2 08** DTSC asks for the relevance of the oil spill application to the multi media risk assessment.

**The oil collection product, Elastol, has the same active component, ultra high molecular weight PIB, as Viscon. We consider technical data relating to physical properties and toxicology of Elastol relevant to the risk assessment of Viscon.**

**6. Question # 4 from Memorandum dated 12 2 08** DTSC characterizes attachments 7 and 12 as viscosity tests and asks why these tests support the multi media risk assessment.

**Attachment 7 is a scientific description of the phenomenon upon which Viscon is based. It is intended to give the reviewer of the multi media risk assessment an understanding of how a minute amount of a simple polymer can produce a significant increase in the efficiency of internal combustion engines and in doing so reduce the production of harmful exhaust emissions.**

**Attachment 12 is a scientific report on the properties of hydrocarbon liquids treated with ultra high molecular weight PIB in water environments. This study was included as background information related to potential releases of Viscon treated CARB diesel into water.**

**Table 2**  
**Percent change of various emissions comparing ULSF to ULSF w/ Viscon - Caterpillar 3306 Diesel Engine (Units in g/bhp-hr)**  
 Testing Conducted by Olson-Ecologic Engine Testing Laboratories for Viscon California October 2006

	ULSF				ULSF w/ Viscon			Average	Change
	8MLP17	8MLP18	8MLP19	Average	8MLP39	8MLP40	8MLP41		
<b>VOC's</b>	(Analyzed via Gas Chromatography by Olson-EcoLogic Engine Testing Laboratories)								
1,3-butadiene	0.000245	0.000212	0.000300	<b>0.000252</b>	<RL	<RL	<RL	<RL	<b>-100%</b>
benzene	0.008692	0.008825	0.009560	<b>0.009026</b>	0.011935	0.014013	0.012897	<b>0.012948</b>	<b>43%</b>
toluene	0.004514	0.004058	0.004111	<b>0.004228</b>	0.005860	0.006042	0.006219	<b>0.006040</b>	<b>43%</b>
ethylbenzene	0.002038	0.001774	0.001621	<b>0.001811</b>	0.002025	0.002119	0.002183	<b>0.002109</b>	<b>16%</b>
m&p-xylene	0.004081	0.003440	0.004175	<b>0.003898</b>	0.002775	0.002875	0.003084	<b>0.002911</b>	<b>-25%</b>
o-xylene	0.002292	0.002320	0.002320	<b>0.002311</b>	0.001828	0.001766	0.001810	<b>0.001801</b>	<b>-22%</b>
<b>PAH's</b>	(Analyzed via Gas Chromatography/Mass Spectrometry by Desert Research Institute)								
naphthalene	0.0000125192	0.0000071081	0.0000119718	<b>0.0000105330</b>	0.0000051731	0.0000046456	0.0000039993	<b>0.0000046060</b>	<b>-56%</b>
2-methylnaphthalene	0.0000112961	0.0000084828	0.0000095142	<b>0.0000097644</b>	0.0000024317	0.0000027416	0.0000025569	<b>0.0000025767</b>	<b>-74%</b>
acenaphthylene	0.0000002048	0.0000001814	0.0000001930	<b>0.0000001930</b>	0.0000001683	0.0000001606	0.0000001573	<b>0.0000001621</b>	<b>-16%</b>
acenaphthene	0.0000005852	0.0000006578	0.0000007354	<b>0.0000006595</b>	0.0000000615	0.0000000706	0.0000000610	<b>0.0000000644</b>	<b>-90%</b>
fluorene	0.0000005526	0.0000005425	0.0000005102	<b>0.0000005351</b>	0.0000001376	0.0000001311	0.0000001238	<b>0.0000001308</b>	<b>-76%</b>
phenanthrene	0.0000013504	0.0000013821	0.0000013007	<b>0.0000013444</b>	0.0000004623	0.0000005479	0.0000004993	<b>0.0000005032</b>	<b>-63%</b>
anthracene	0.0000001898	0.0000001834	0.0000001699	<b>0.0000001810</b>	0.0000000647	0.0000000816	0.0000000780	<b>0.0000000748</b>	<b>-59%</b>
fluoranthene	0.0000000719	0.0000000720	0.0000000579	<b>0.0000000673</b>	0.0000000559	0.0000000599	0.0000000350	<b>0.0000000503</b>	<b>-25%</b>
pyrene	0.0000002207	0.0000002166	0.0000002438	<b>0.0000002270</b>	0.0000001584	0.0000001207	0.0000000982	<b>0.0000001258</b>	<b>-45%</b>
benz(a)anthracene	0.0000000074	0.0000000084	0.0000000070	<b>0.0000000076</b>	0.0000000025	0.0000000015	0.0000000006	<b>0.0000000015</b>	<b>-80%</b>
chrysene-triphenylene	0.0000000108	0.0000000105	0.0000000141	<b>0.0000000118</b>	0.0000000073	0.0000000045	0.0000000042	<b>0.0000000054</b>	<b>-55%</b>
benzo(b+j)fluoranthene	0.0000000016	0.0000000006	0.0000000006	<b>0.0000000010</b>	0.0000000000	0.0000000000	0.0000000010	<b>0.0000000003</b>	<b>-67%</b>
benzo(k)fluoranthene	0.0000000005	0.0000000002	0.0000000003	<b>0.0000000003</b>	0.0000000002	0.0000000000	0.0000000003	<b>0.0000000002</b>	<b>-50%</b>
BeP	0.0000000013	0.0000000006	0.0000000006	<b>0.0000000009</b>	0.0000000005	0.0000000005	0.0000000006	<b>0.0000000005</b>	<b>-37%</b>
BaP	0.0000000002	0.0000000000	0.0000000010	<b>0.0000000004</b>	0.0000000000	0.0000000000	0.0000000000	<b>0.0000000000</b>	<b>-100%</b>
perylene	0.0000000006	0.0000000000	0.0000000002	<b>0.0000000003</b>	0.0000000000	0.0000000000	0.0000000000	<b>0.0000000000</b>	<b>-100%</b>
indeno[123-cd]pyrene	0.0000000004	0.0000000000	0.0000000000	<b>0.0000000001</b>	0.0000000002	0.0000000000	0.0000000000	<b>0.0000000001</b>	<b>-50%</b>
dibenzo(ah+ac)anthracene	0.0000000002	0.0000000000	0.0000000000	<b>0.0000000001</b>	0.0000000000	0.0000000000	0.0000000007	<b>0.0000000002</b>	<b>299%</b>
benzo(ghi)perylene	0.0000000000	0.0000000000	0.0000000000	<b>0.0000000000</b>	0.0000000000	0.0000000000	0.0000000004	<b>0.0000000001</b>	

**Table 2 (cont.)**

	<i>ULSF</i>				<i>ULSF w/ Viscon</i>			<i>Average</i>	<i>Change</i>
	<i>8MLP17</i>	<i>8MLP18</i>	<i>8MLP19</i>	<i>Average</i>	<i>8MLP39</i>	<i>8MLP40</i>	<i>8MLP41</i>		
<b>Carbonyls</b>	(Analyzed via High Performance Liquid Chromatography by Desert Research Institute)								
formaldehyde	0.027876	0.026773	0.027121	<b>0.027257</b>	0.035915	0.032348	0.032137	<b>0.033467</b>	<b>23%</b>
acetaldehyde	0.007410	0.007211	0.007535	<b>0.007385</b>	0.009786	0.009169	0.008887	<b>0.009281</b>	<b>26%</b>
acetone	0.002020	0.001754	0.001827	<b>0.001867</b>	0.002895	0.002536	0.002490	<b>0.002640</b>	<b>41%</b>
acrolein	0.000813	0.001405	0.000857	<b>0.001025</b>	0.002133	0.001474	0.001846	<b>0.001817</b>	<b>77%</b>
propionaldehyde	0.001346	0.001304	0.001346	<b>0.001332</b>	0.001651	0.001444	0.001443	<b>0.001512</b>	<b>14%</b>
crotonaldehyde	0.000904	0.001060	0.001027	<b>0.000997</b>	0.001260	0.001122	0.001175	<b>0.001186</b>	<b>19%</b>
methyl ethyl ketone	0.000527	0.000410	0.000448	<b>0.000462</b>	0.000484	0.000437	0.000518	<b>0.000480</b>	<b>4%</b>
methacrolein	0.000474	0.000491	0.000377	<b>0.000447</b>	0.000635	0.000500	0.000552	<b>0.000562</b>	<b>26%</b>
butyraldehyde	0.004892	0.004370	0.005015	<b>0.004759</b>	0.004111	0.003893	0.004090	<b>0.004031</b>	<b>-15%</b>
benzaldehyde	0.000631	0.000617	0.000652	<b>0.000633</b>	0.001054	0.000536	0.000583	<b>0.000724</b>	<b>14%</b>
glyoxal	0.000404	0.000134	0.000429	<b>0.000322</b>	0.000644	0.000631	0.000731	<b>0.000669</b>	<b>108%</b>
valeraldehyde	0.000770	0.000515	0.000567	<b>0.000617</b>	0.000590	0.000424	0.000475	<b>0.000497</b>	<b>-20%</b>
m-tolualdehyde	0.000012	0.000025	0.000012	<b>0.000016</b>	0.000126	0.000101	0.000101	<b>0.000110</b>	<b>570%</b>
hexanaldehyde	0.000350	0.000292	0.000305	<b>0.000316</b>	0.000453	0.000382	0.000270	<b>0.000368</b>	<b>17%</b>

**Table 3**

**Percent change of various emissions comparing ULSF to ULSF w/ 10x Viscon - Caterpillar 3306 Diesel Engine (Units in g/bhp-hr)**

Testing Conducted by Olson-Ecologic Engine Testing Laboratories for Viscon California October 2006

	<i>ULSF</i>				<i>ULSF w/ 10x Viscon</i>		<i>Average</i>	<i>Change</i>
	<i>8MLP17</i>	<i>8MLP18</i>	<i>8MLP19</i>	<i>Average</i>	<i>8MLP46</i>	<i>8MLP47</i>		
<b>VOC's</b>	(Analyzed via Gas Chromatography by Olson-EcoLogic Engine Testing Laboratories)							
1,3-butadiene	0.000245	0.000212	0.000300	<b>0.000252</b>	<RL	<RL	<RL	<b>-100%</b>
benzene	0.008692	0.008825	0.009560	<b>0.009026</b>	0.011786	0.010998	<b>0.011392</b>	<b>26%</b>
toluene	0.004514	0.004058	0.004111	<b>0.004228</b>	0.005157	0.005194	<b>0.005176</b>	<b>22%</b>
ethylbenzene	0.002038	0.001774	0.001621	<b>0.001811</b>	0.001969	0.001515	<b>0.001742</b>	<b>-4%</b>
m&p-xylene	0.004081	0.003440	0.004175	<b>0.003898</b>	0.002573	0.002435	<b>0.002504</b>	<b>-36%</b>
o-xylene	0.002292	0.002320	0.002320	<b>0.002311</b>	0.001691	0.001706	<b>0.001699</b>	<b>-26%</b>

Percent change of various emissions comparing ULSF to ULSF w/ Viscon - Caterpillar 3306 Diesel Engine (Units in g/bhp-hr)

**VOC's**

(Analyzed via Gas Chromatography by Olson-EcoLogic Engine Testing Laboratories)

	<u>10/10/2006</u>	<u>12/21/2007</u>	<u>12/21/2007</u>	<u>12/21/2007</u>		
	<b>Baseline Avg.</b>	<b>8MLPV58</b>	<b>8MLPV59</b>	<b>8MLPV60</b>	<b>Avg.</b>	<b>Change</b>
1,3-butadiene	0.000252	0.004201	0.005098	0.003878	0.004392	1641%
benzene	0.009026	0.009855	0.009093	0.008764	0.009237	2%
toluene	0.004228	0.005123	0.005008	0.004917	0.005016	19%
ethylbenzene	0.001811	0.002436	0.002532	0.002426	0.002465	36%
m&p-xylene	0.003898	0.003837	0.004109	0.004064	0.004003	3%
o-xylene	0.002311	0.002282	0.002894	0.002525	0.002567	11%

**Carbonyls**

(Analyzed via High Performance Liquid Chromatography by Desert Research Institute)

	<u>10/10/2006</u>	<u>12/11/2007</u>	<u>12/11/2007</u>	<u>12/11/2007</u>		
	<b>Baseline Avg.</b>	<b>8MLPV52</b>	<b>8MLPV53</b>	<b>8MLPV54</b>	<b>Avg.</b>	<b>Change</b>
formaldehyde	0.027257	0.032759	0.026156	0.031159	0.030025	10%
acetaldehyde	0.007385	0.007214	0.006248	0.007383	0.006948	-6%
acetone	0.001867	0.000364	0.001660	0.000632	0.000886	-53%
acrolein	0.001025	0.000178	0.001332	0.000507	0.000672	-34%
propionaldehyde	0.001332	0.001030	0.001104	0.001136	0.001090	-18%
crotonaldehyde	0.000997	0.000000	0.000894	0.000101	0.000332	-67%
methyl ethyl ketone	0.000462	0.000028	0.000241	0.000047	0.000105	-77%
methacrolein	0.000447	0.000045	0.000000	0.000064	0.000037	-92%
butyraldehyde	0.004759	0.000519	0.000511	0.000645	0.000558	-88%
benzaldehyde	0.000633	0.000350	0.000751	0.000366	0.000489	-23%
glyoxal	0.000322	0.000000	0.000000	0.000000	0.000000	-100%
valeraldehyde	0.000617	0.000000	0.000000	0.000000	0.000000	-100%
m-tolualdehyde	0.000016	0.000170	0.000340	0.000158	0.000223	1260%
hexanaldehyde	0.000316	0.000420	0.000316	0.000448	0.000395	25%

**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**8-Mode Steady State Trace Toxics by Gas Chromatography Results**

Chemist: Nathan Imus

Date: 12/21/2007

**Sample Analysis**

Sample ID: **8MLPV58**  
 Sample Type: *Dilute Exhaust*  
 Sample Time: 12/20/2007 15:10  
 Dilution (DF): 8.5200  
 Collection (min): 20

Comments: w/ Viscon  
 Injection Time: 12/20/2007 15:18  
 Weighted Avg. bhp: 131.5400  
 Work (bhp-hr): 43.8467  
 Tunnel Volume (m<sup>3</sup>/min): 70  
 Total Volume (ft<sup>3</sup>): 49441

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	237.50
benzene	582.57
toluene	455.71
ethylbenzene	152.59
m&p-xylene	271.42
o-xylene	158.87

Sample ID: **Dilution Air 58**  
 Sample Type: *Dilution Air*  
 Sample Time: 12/20/2007 15:10

Injection Time: 12/20/2007 23:47

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	3.42
benzene	12.29
toluene	162.49
ethylbenzene	14.31
m&p-xylene	53.60
o-xylene	29.33

**Sample Result**

Analyte	Conc <sub>corrected</sub> (ppbC)	Conc (ppb)	Raw Conc (ppb)	Conc (g/bhp-hr)
1,3-butadiene	<b>234.08</b>	58.52	498.59	<b>0.004201</b>
benzene	<b>570.28</b>	95.05	809.80	<b>0.009855</b>
toluene	<b>293.22</b>	41.89	356.89	<b>0.005123</b>
ethylbenzene	<b>138.28</b>	17.29	147.27	<b>0.002436</b>
m&p-xylene	<b>217.82</b>	27.23	231.98	<b>0.003837</b>
o-xylene	<b>129.54</b>	16.19	137.96	<b>0.002282</b>

Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_03\_8mlpv58.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 03\_8MLPV58

Injection Date: 12/20/2007 3:18 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HUIE Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 13 rows of identified peaks and a Totals row.

Total Unidentified Counts : 463833 counts

Detected Peaks: 132 Rejected Peaks: 1 Identified Peaks: 13

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: -30 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 45 microVolts

Manual injection

Error Log:

300 GC:

3800 GC:

\*\*\*\*\*

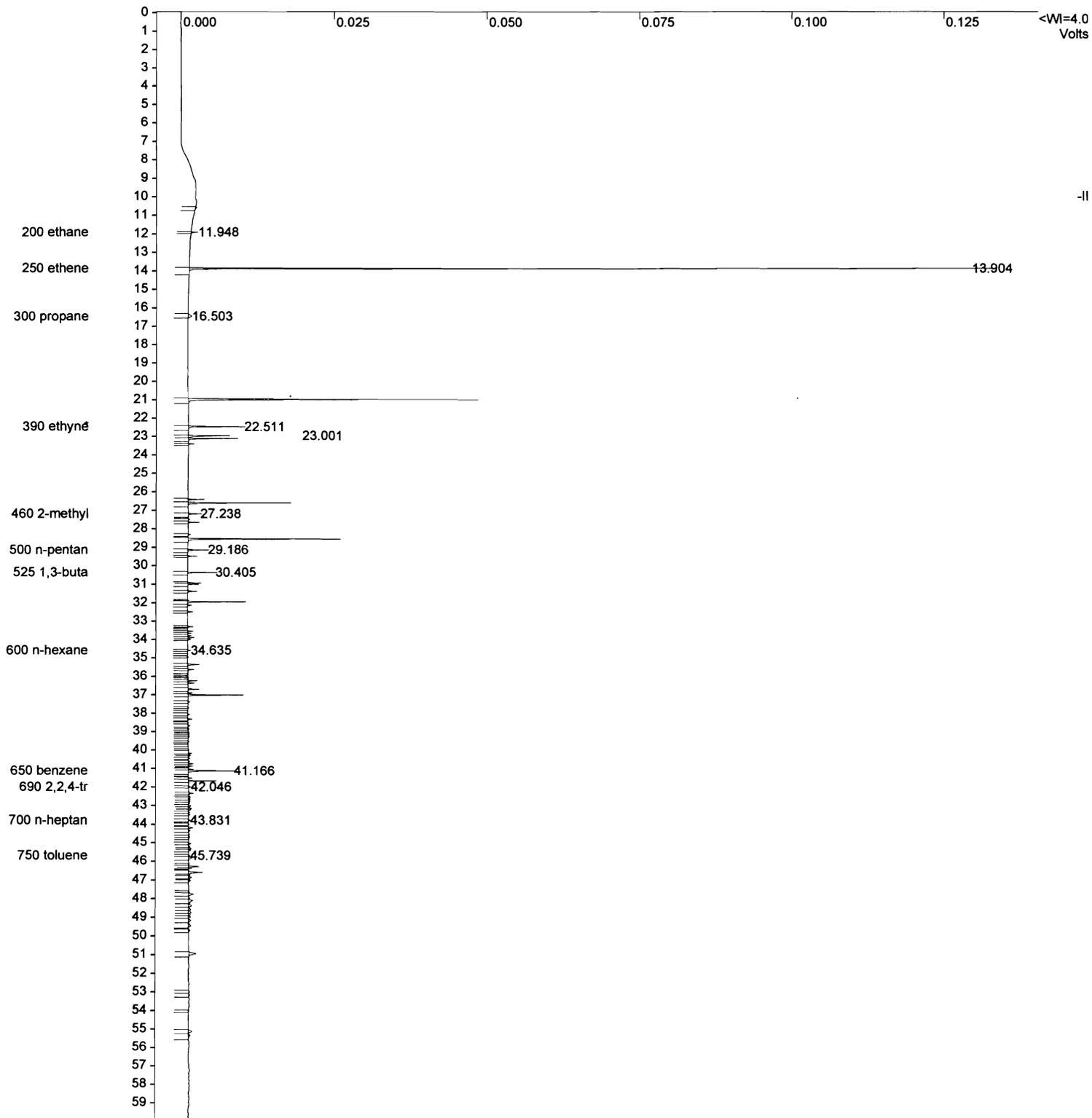
Title : GC# 1 - LE S/N08901  
In File : c:\star\data\122007\3800.45\_03\_8mlpv58.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 03\_8MLPV58

Injection Date: 12/20/2007 3:18 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 1%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_03\_8mlpv58.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 03\_8MLPV58

Injection Date: 12/20/2007 3:18 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Total Unidentified Counts : 4946851 counts

Detected Peaks: 203 Rejected Peaks: 2 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: 3 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 29 microVolts

Manual injection

Error Log:

3800 GC:

300 GC:

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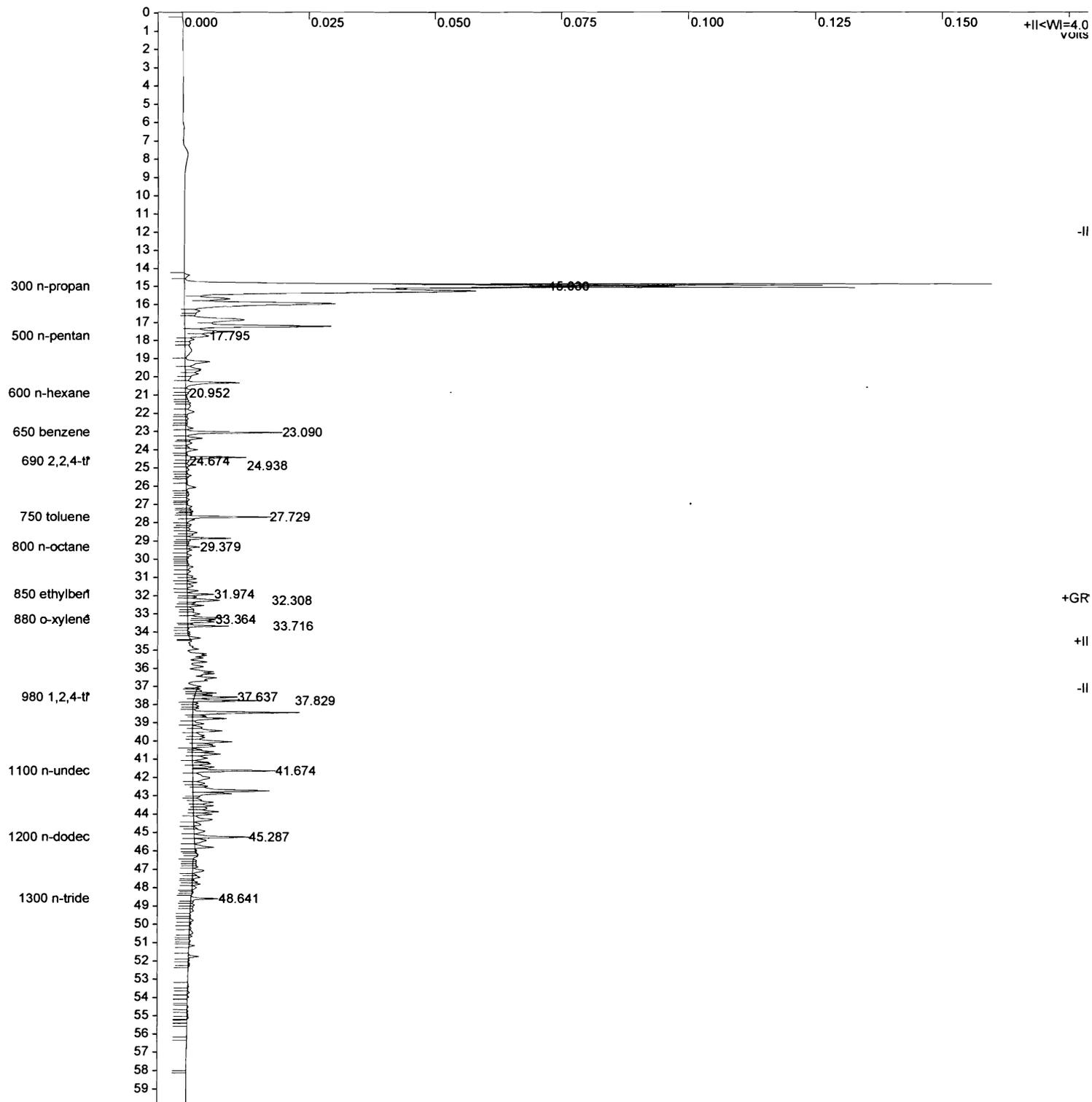
Title : GC# 1 - MR S/N08902  
Run File : c:\star\data\122007\3800.44\_03\_8mlpv58.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 03\_8MLPV58

Injection Date: 12/20/2007 3:18 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 44  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 1%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_08\_dilution air 58.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 08\_Dilution Air 58

Injection Date: 12/20/2007 11:47 PM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚlě Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 11 rows of peak data and a Totals row.

Total Unidentified Counts : 143393 counts

Detected Peaks: 49 Rejected Peaks: 0 Identified Peaks: 11

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: -54 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 35 microVolts

Manual injection

Error Log:

800 GC:

300 GC:

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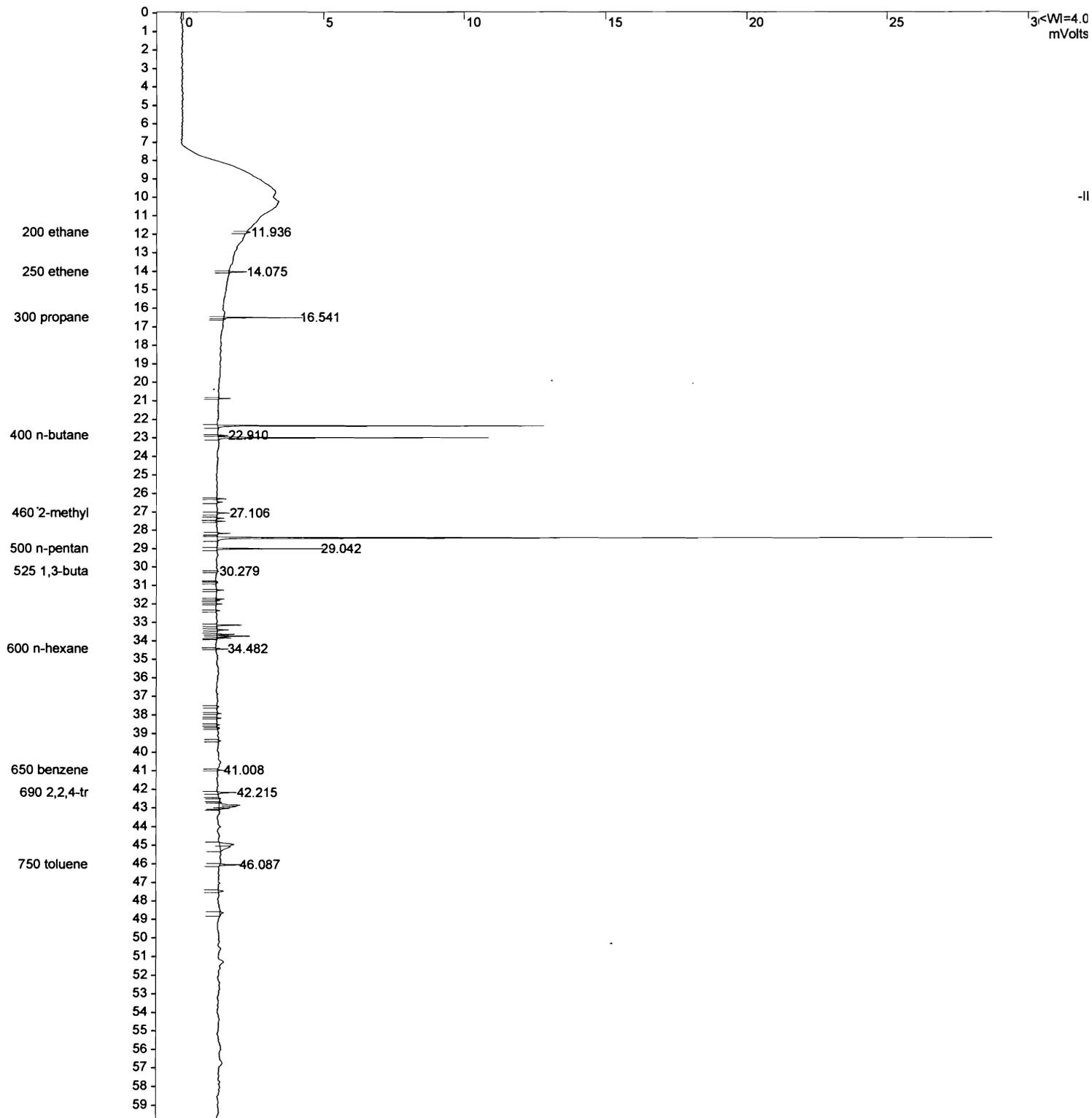
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_08\_dilution air 58.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 08\_Dilution Air 58

Injection Date: 12/20/2007 11:47 PM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 0%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_08\_dilution air 58.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 08\_Dilution Air 58

Injection Date: 12/20/2007 11:47 PM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Status Codes:

- User-defined peak endpoint(s)

Total Unidentified Counts : 26537574 counts

Detected Peaks: 156 Rejected Peaks: 3 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -18 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 27 microVolts

Manual injection

Error Log:

800 GC:

300 GC:

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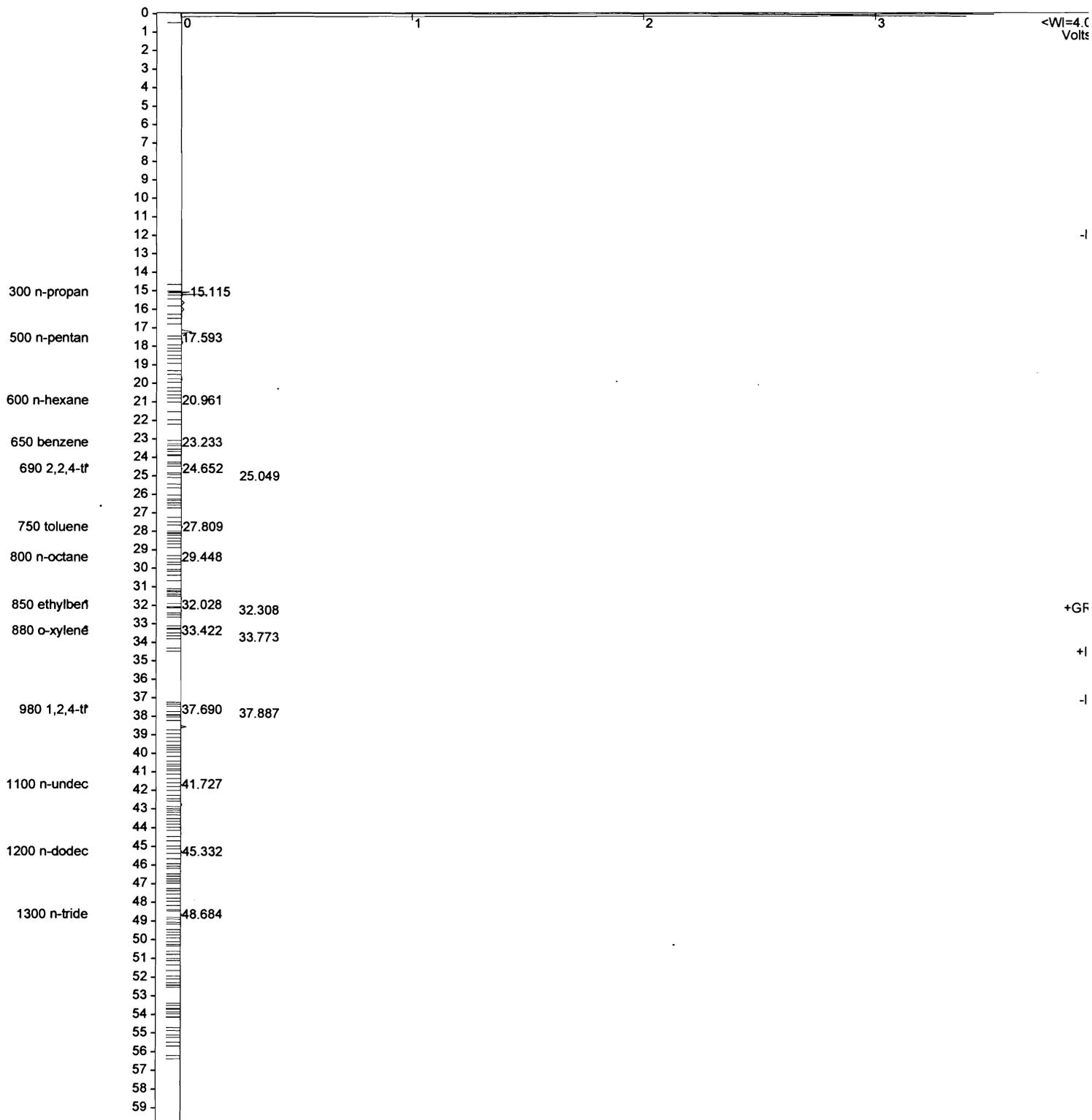
Title : GC# 1 - MR S/N08902  
Run File : c:\star\data\122007\3800.44\_08\_dilution air 58.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 08\_Dilution Air 58

Injection Date: 12/20/2007 11:47 PM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 44  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 16 Zero Offset = 2%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**8-Mode Steady State Trace Toxics by Gas Chromatography Results**

Chemist: Nathan Imus

Date: 12/21/2007

**Sample Analysis**

Sample ID: **8MLPV59**  
 Sample Type: *Dilute Exhaust*  
 Sample Time: 12/20/2007 16:40  
 Dilution (DF): 9.0000  
 Collection (min): 20

Comments: w/ Viscon  
 Injection Time: 12/20/2007 16:57  
 Weighted Avg. bhp: 131.7400  
 Work (bhp-hr): 43.9133  
 Tunnel Volume (m<sup>3</sup>/min): 70  
 Total Volume (ft<sup>3</sup>): 49441

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	288.01
benzene	537.38
toluene	431.24
ethylbenzene	151.93
m&p-xylene	264.04
o-xylene	185.46

Sample ID: **Dilution Air 59**  
 Sample Type: *Dilution Air*  
 Sample Time: 12/20/2007 16:40

Injection Time: 12/21/2007 0:52

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	3.56
benzene	10.35
toluene	144.17
ethylbenzene	8.00
m&p-xylene	30.43
o-xylene	20.92

**Sample Result**

Analyte	Conc <sub>corrected</sub> (ppbC)	Conc (ppb)	Raw Conc (ppb)	Conc (g/bhp-hr)
1,3-butadiene	<b>284.45</b>	71.11	640.01	<b>0.005098</b>
benzene	<b>527.03</b>	87.84	790.55	<b>0.009093</b>
toluene	<b>287.07</b>	41.01	369.09	<b>0.005008</b>
ethylbenzene	<b>143.93</b>	17.99	161.92	<b>0.002532</b>
m&p-xylene	<b>233.61</b>	29.20	262.81	<b>0.004109</b>
o-xylene	<b>164.54</b>	20.57	185.11	<b>0.002894</b>

Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_04\_8mlpv59.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 04\_8MLPV59

Injection Date: 12/20/2007 4:57 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 12 rows of peak data and a summary row for Group 0.

Total Unidentified Counts : 438304 counts

Detected Peaks: 136 Rejected Peaks: 0 Identified Peaks: 12

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: -27 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 37 microVolts

Manual injection

Error Log:

3800 GC:

300 GC:

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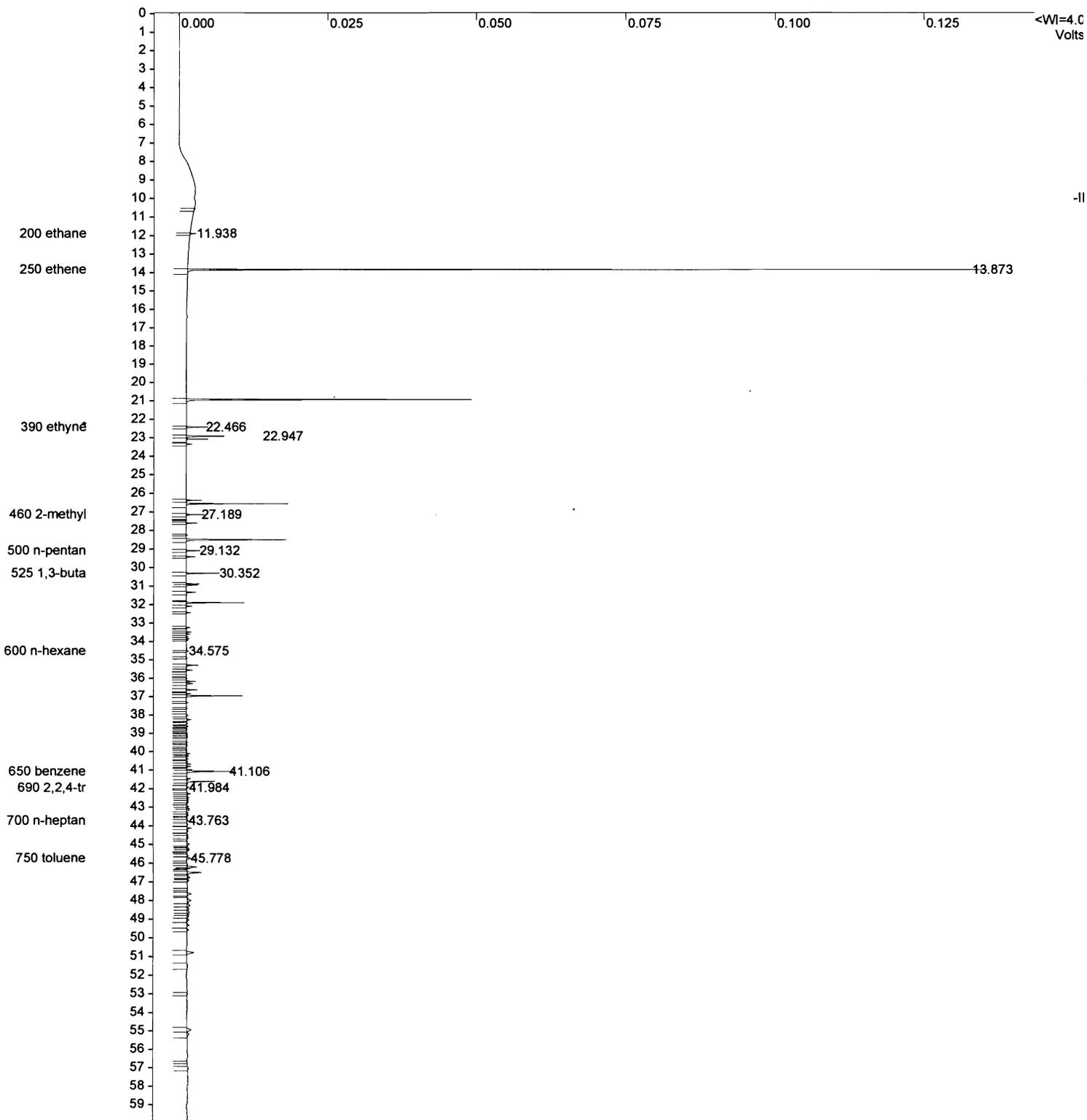
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_04\_8mlpv59.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 04\_8MLPV59

Injection Date: 12/20/2007 4:57 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 1%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_04\_8mlpv59.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 04\_8MLPV59

Injection Date: 12/20/2007 4:57 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚÍŠ Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 11 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Total Unidentified Counts : 5320684 counts

Detected Peaks: 204 Rejected Peaks: 1 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: 26 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 40 microVolts

Manual injection

Error Log:

800 GC:

800 GC:

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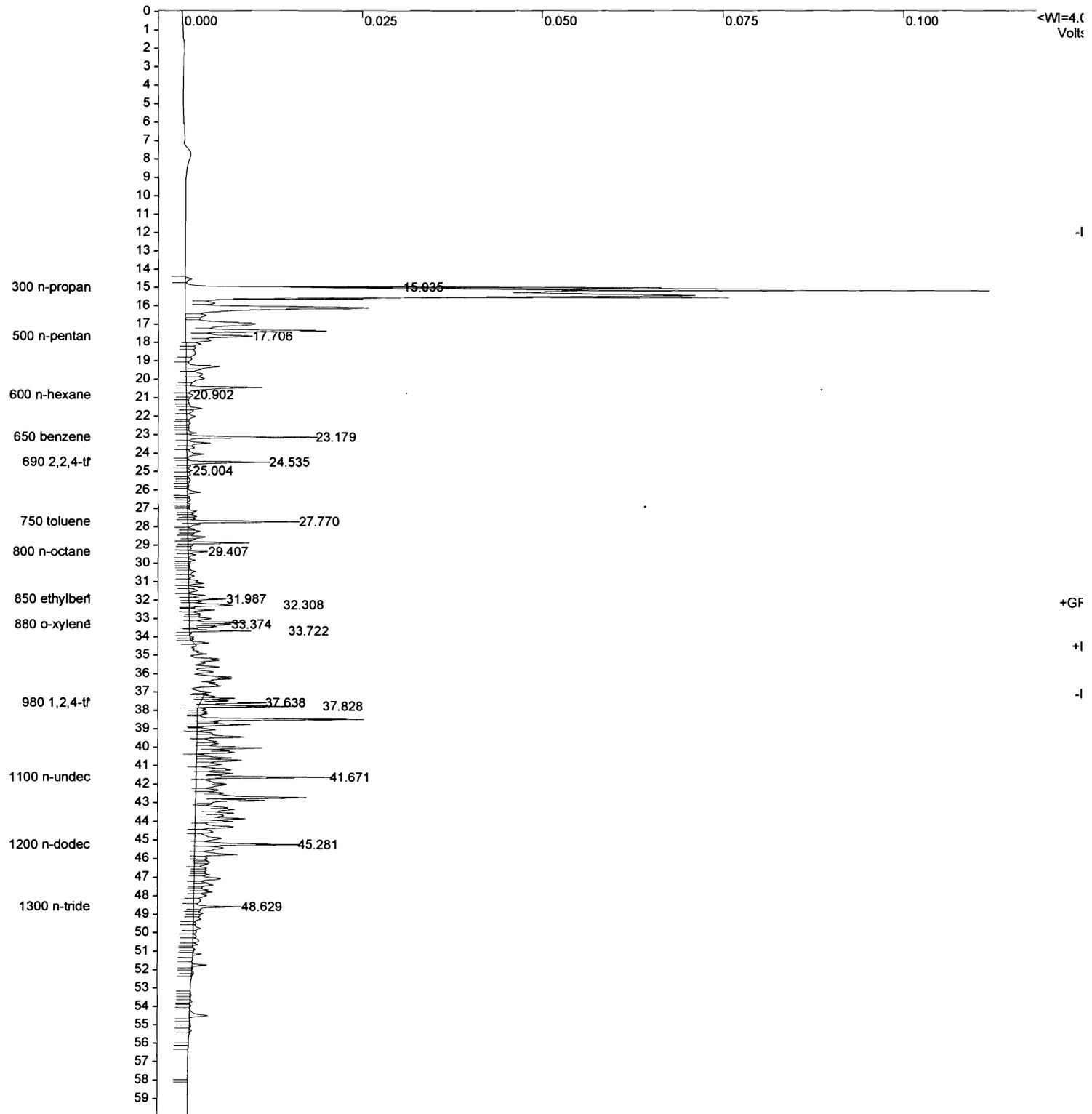
Title : GC# 1 - MR S/N08902  
 Run File : c:\star\data\122007\3800.44\_04\_8mlpv59.run  
 Method File : c:\star\methods\methods\121707.mth  
 Sample ID : 04\_8MLPV59

Injection Date: 12/20/2007 4:57 PM      Calculation Date: 12/21/2007 12:01 PM

Operator : NI      Detector Type: 3800 (1000 Volts)  
 Workstation: STARWORKSTATION HÚië      Bus Address : 44  
 Instrument : Lotus THS      Sample Rate : 10.00 Hz  
 Channel : Front = FID      Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min      Attenuation = 1      Zero Offset = 1%  
 Start Time = 0.000 min      End Time = 59.940 min      Min / Tick = 1.00



Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_09\_dilution air 59.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 09\_Dilution Air 59

Injection Date: 12/21/2007 12:52 AM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 10 identified peaks and a Totals row.

Total Unidentified Counts : 74125 counts

Detected Peaks: 39 Rejected Peaks: 0 Identified Peaks: 10

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: 9 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 36 microVolts

Manual injection

Error Log:

3800 GC:

7800 GC:

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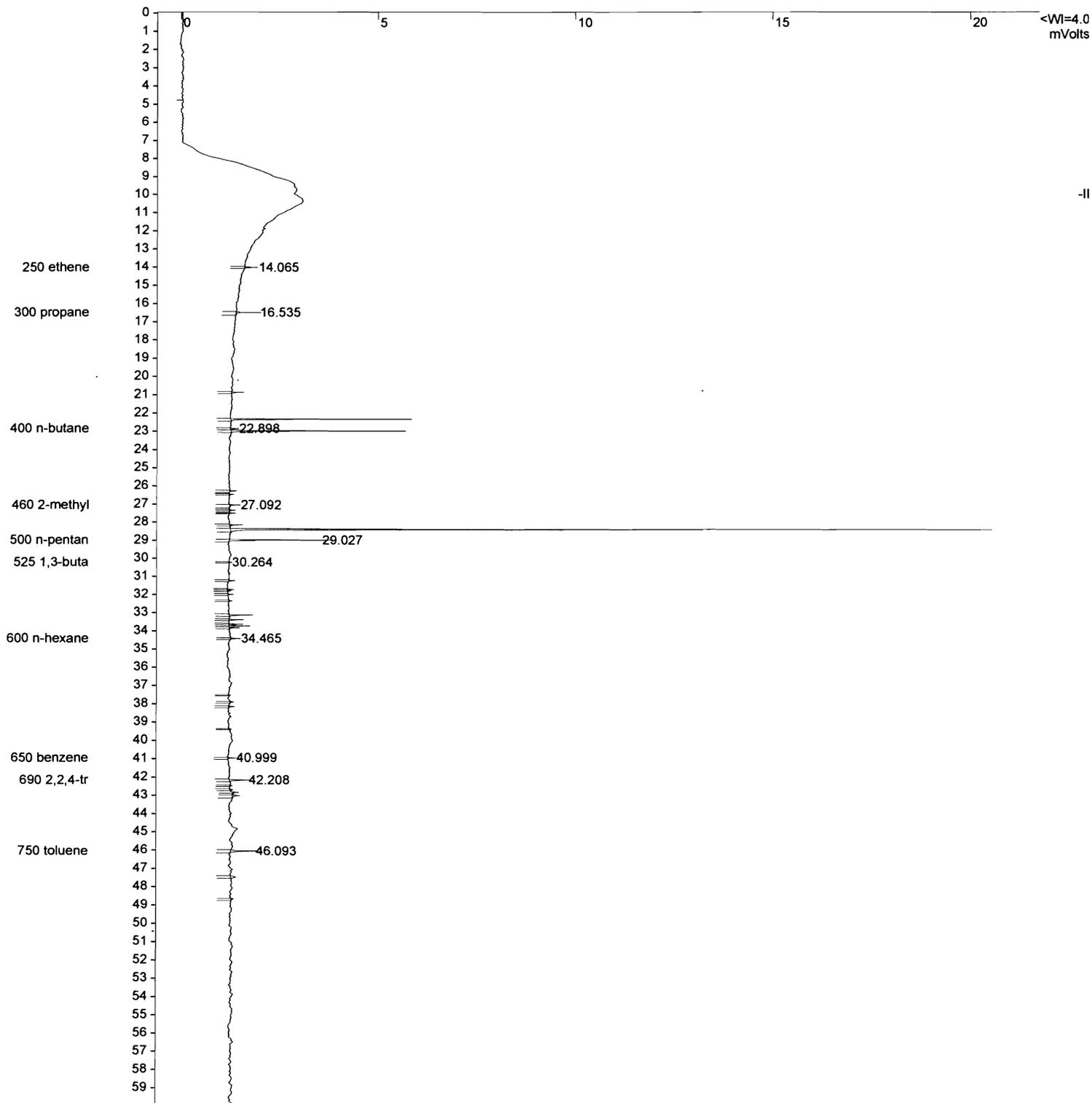
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_09\_dilution.air 59.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 09\_Dilution Air 59

Injection Date: 12/21/2007 12:52 AM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 0%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_09\_dilution air 59.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 09\_Dilution Air 59

Injection Date: 12/21/2007 12:52 AM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚie Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a summary row for Group 0.

Status Codes:

\* - User-defined peak endpoint(s)

Total Unidentified Counts : 1076818 counts

Detected Peaks: 162 Rejected Peaks: 4 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -10 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 12 microVolts

Manual injection

Error Log:

3800 GC:

3800 GC:

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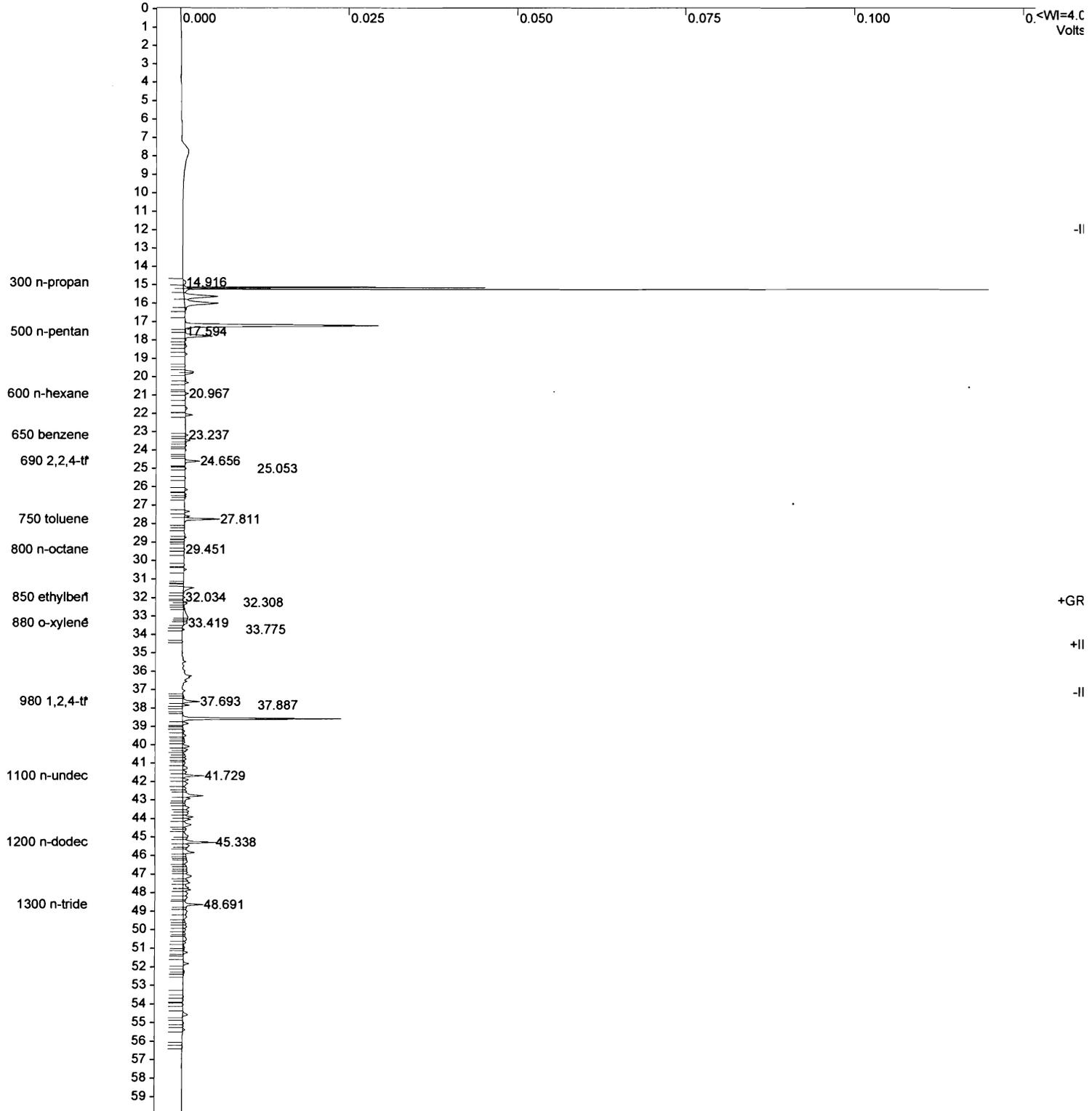
Title : GC# 1 - MR S/N08902  
Run File : c:\star\data\122007\3800.44\_09\_dilution air 59.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 09\_Dilution Air 59

Injection Date: 12/21/2007 12:52 AM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚÍë Bus Address : 44  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 1%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**8-Mode Steady State Trace Toxics by Gas Chromatography Results**

Chemist: Nathan Imus

Date: 12/21/2007

**Sample Analysis**

Sample ID: **8MLPV60**  
 Sample Type: *Dilute Exhaust*  
 Sample Time: 12/20/2007 19:30  
 Dilution (DF): 9.5000  
 Collection (min): 20

Comments: *w/ Viscon*  
 Injection Time: 12/20/2007 19:38  
 Weighted Avg. bhp: 131.2900  
 Work (bhp-hr): 43.7633  
 Tunnel Volume (m<sup>3</sup>/min): 70  
 Total Volume (ft<sup>3</sup>): 49441

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	218.10
benzene	513.14
toluene	356.86
ethylbenzene	145.38
m&p-xylene	258.95
o-xylene	162.30

Sample ID: **Dilution Air 60**  
 Sample Type: *Dilution Air*  
 Sample Time: 12/20/2007 19:30

Injection Time: 12/21/2007 1:57

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	2.47
benzene	6.92
toluene	75.95
ethylbenzene	7.91
m&p-xylene	28.69
o-xylene	19.21

**Sample Result**

Analyte	Conc <sub>corrected</sub> (ppbC)	Conc (ppb)	Raw Conc (ppb)	Conc (g/bhp-hr)
1,3-butadiene	<b>215.63</b>	53.91	512.12	<b>0.003878</b>
benzene	<b>506.22</b>	84.37	801.52	<b>0.008764</b>
toluene	<b>280.91</b>	40.13	381.24	<b>0.004917</b>
ethylbenzene	<b>137.47</b>	17.18	163.25	<b>0.002426</b>
m&p-xylene	<b>230.26</b>	28.78	273.43	<b>0.004064</b>
o-xylene	<b>143.09</b>	17.89	169.92	<b>0.002525</b>

Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_05\_8mlpv60.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 05\_8MLPV60

Injection Date: 12/20/2007 7:38 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\*\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 12 rows of peak data and a Totals row.

Total Unidentified Counts : 415770 counts

Detected Peaks: 129 Rejected Peaks: 2 Identified Peaks: 12

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: 23 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 27 microVolts

Manual injection

Error Log:

3800 GC:

1800 GC:

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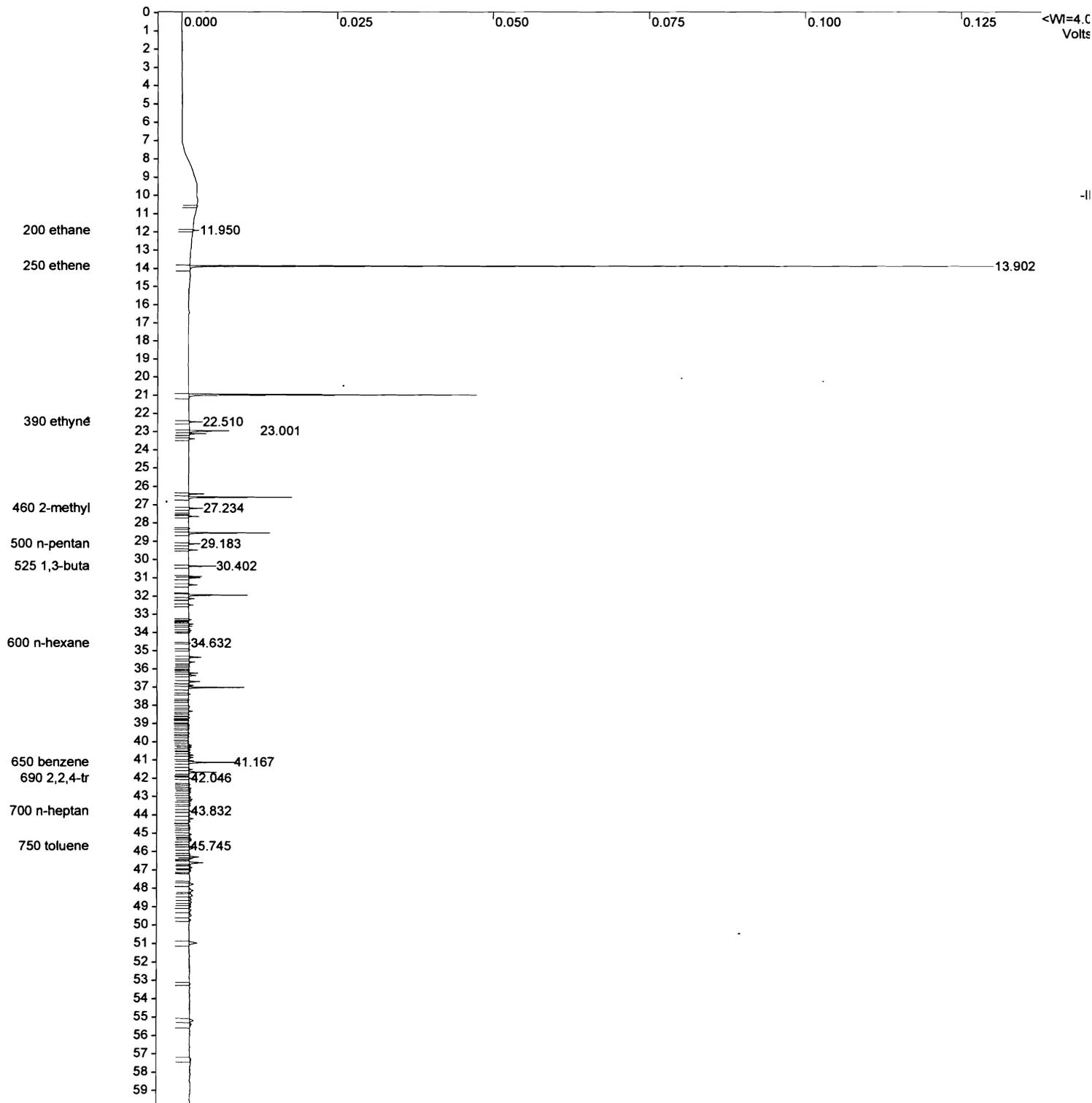
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_05\_8mlpv60.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 05\_8MLPV60

Injection Date: 12/20/2007 7:38 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 1%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_05\_8mlpv60.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 05\_8MLPV60

Injection Date: 12/20/2007 7:38 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Total Unidentified Counts : 33403384 counts

Detected Peaks: 202 Rejected Peaks: 0 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -5 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 20 microVolts

Manual injection

Error Log:

3800 GC:

300 GC:

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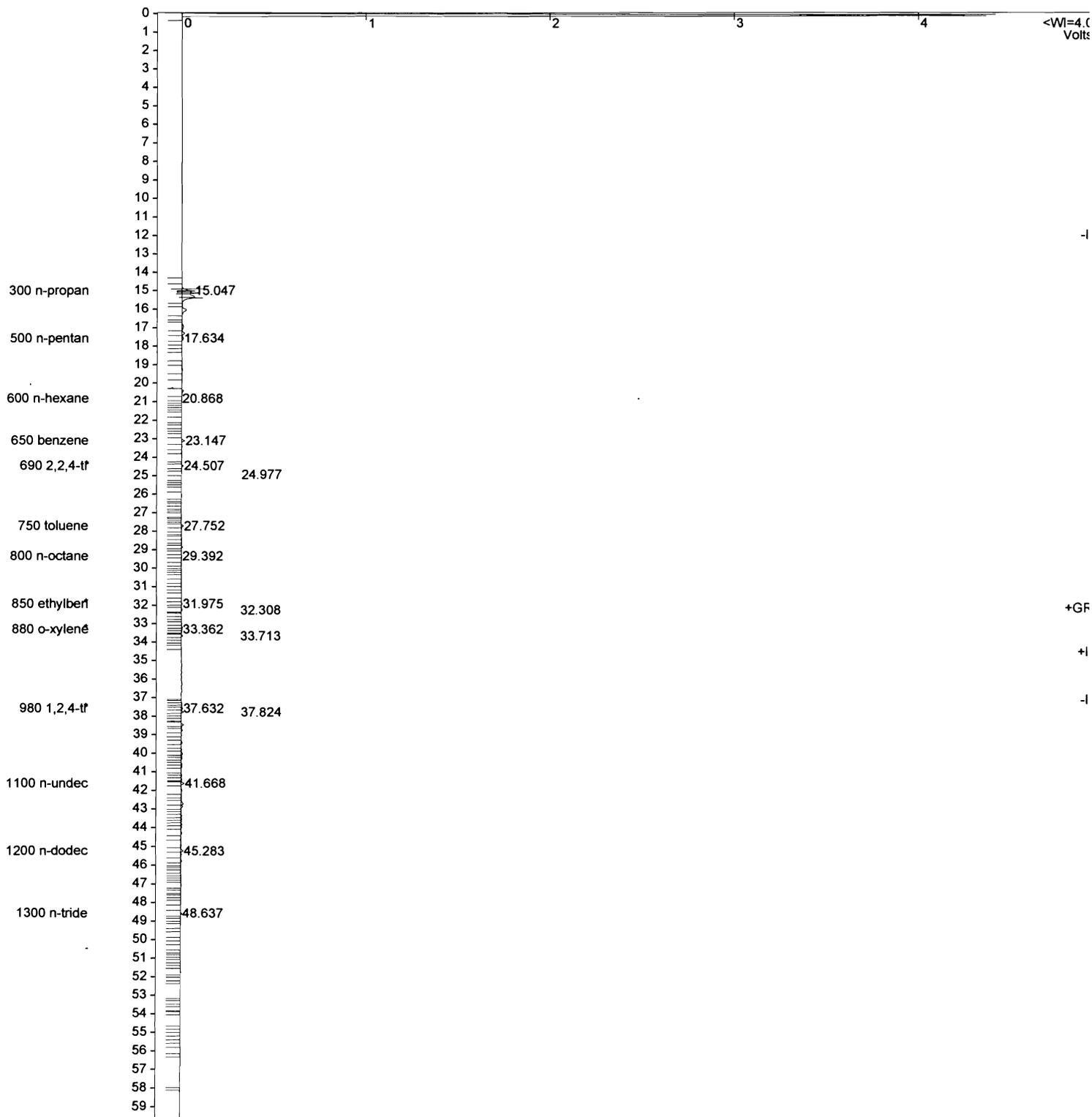
Title : GC# 1 - MR S/N08902  
Run File : c:\star\data\122007\3800.44\_05\_8mlpv60.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 05\_MLpv60

Injection Date: 12/20/2007 7:38 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 44  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\*\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 20 Zero Offset = 2%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_10\_dilution air 60.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 10\_Dilution Air 60

Injection Date: 12/21/2007 1:57 AM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\*\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 10 rows of peak data and a Totals row.

Total Unidentified Counts : 55458 counts

Detected Peaks: 33 Rejected Peaks: 0 Identified Peaks: 10

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: 0 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 76 microVolts

Manual injection

Error Log:

800 GC:

3800 GC:

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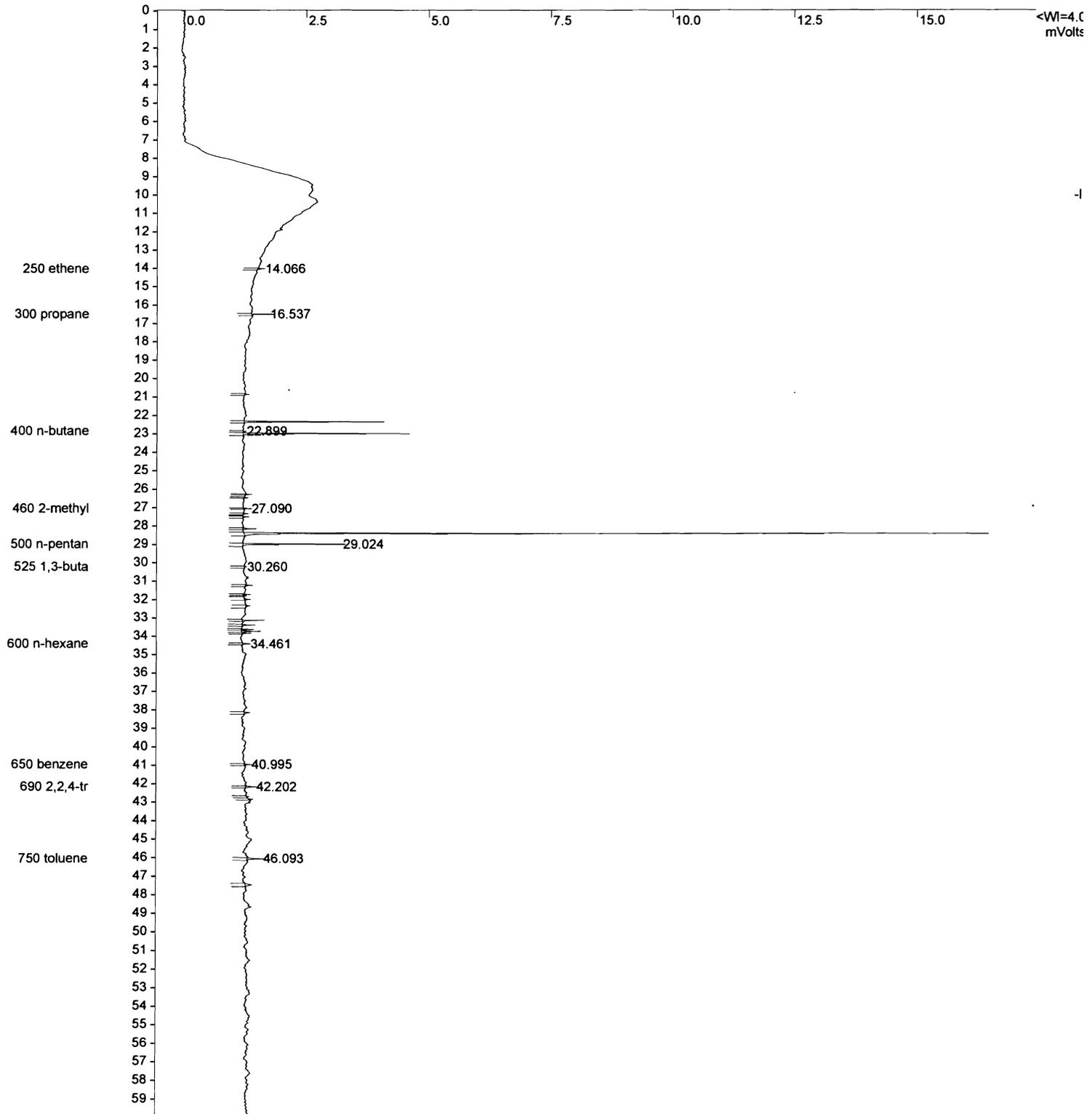
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_10\_dilution air 60.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 10\_Dilution Air 60

Injection Date: 12/21/2007 1:57 AM      Calculation Date: 12/21/2007 2:02 PM

Operator : NI      Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië      Bus Address : 45  
Instrument : Lotus THS      Sample Rate : 10.00 Hz  
Channel : Front = FID      Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min      Attenuation = 1      Zero Offset = 0%  
Start Time = 0.000 min      End Time = 59.940 min      Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_10\_dilution air 60.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 10\_Dilution Air 60

Injection Date: 12/21/2007 1:57 AM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\*\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Total Unidentified Counts : 835804 counts

Detected Peaks: 156 Rejected Peaks: 6 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -6 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 12 microVolts

Manual injection

Error Log:

3800 GC:

800 GC:

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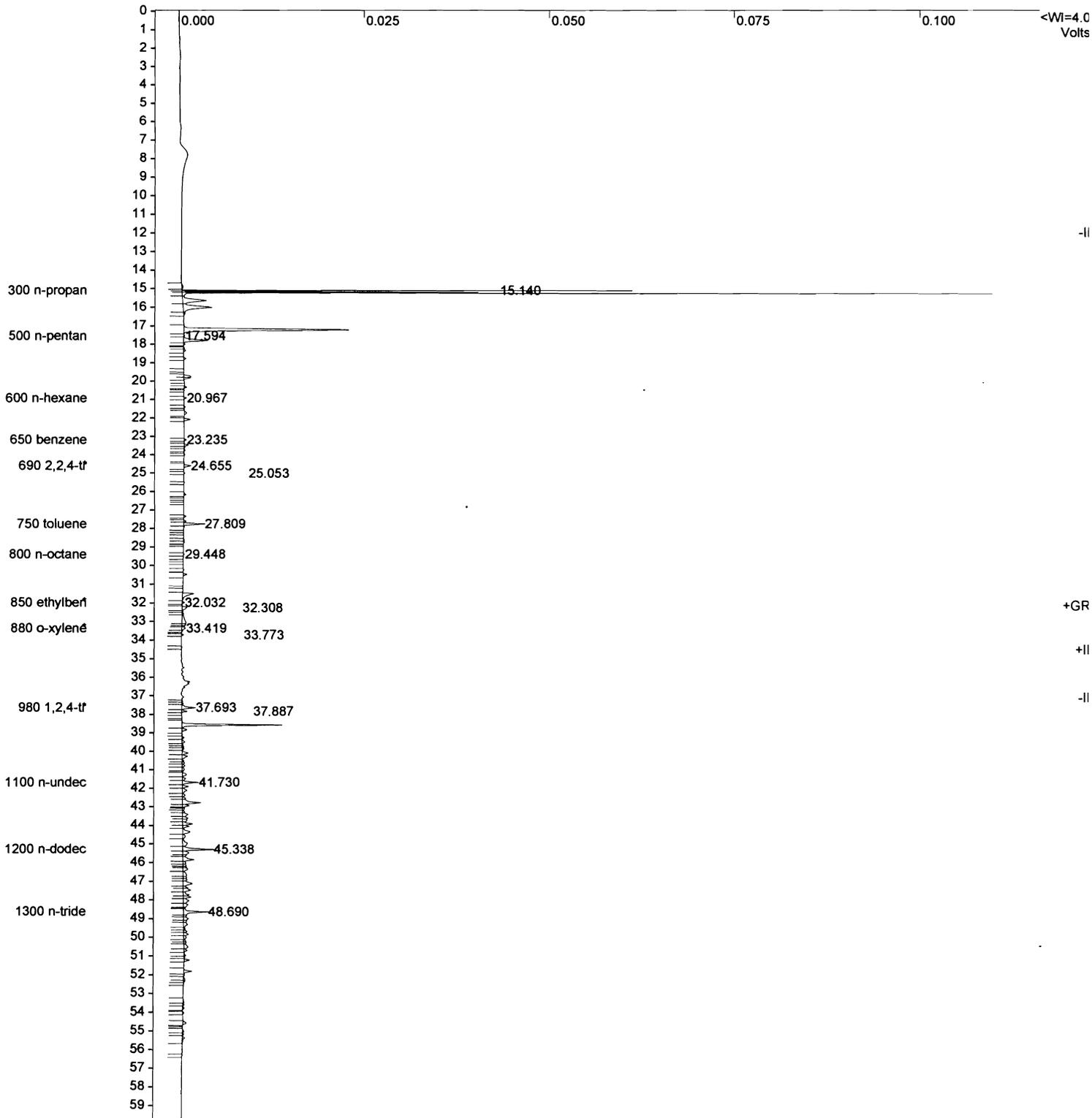
Title : GC# 1 - MR S/N08902  
 Run File : c:\star\data\122007\3800.44\_10\_dilution air 60.run  
 Method File : c:\star\methods\methods\121707.mth  
 Sample ID : 10\_Dilution Air 60

Injection Date: 12/21/2007 1:57 AM      Calculation Date: 12/21/2007 12:11 PM

Operator : NI      Detector Type: 3800 (1000 Volts)  
 Workstation: STARWORKSTATION HÚIĚ      Bus Address : 44  
 Instrument : Lotus THS      Sample Rate : 10.00 Hz  
 Channel : Front = FID      Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min      Attenuation = 1      Zero Offset = 1%  
 Start Time = 0.000 min      End Time = 59.940 min      Min / Tick = 1.00



**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**8-Mode Steady State Trace Toxics by Gas Chromatography Results**

Chemist: Nathan Imus

Date: 12/21/2007

**Sample Analysis**

Sample ID: **8MLPV61**  
 Sample Type: *Dilute Exhaust*  
 Sample Time: 12/20/2007 21:26  
 Dilution (DF): 9.4400  
 Collection (min): 20

Comments: *w/ Viscon*  
 Injection Time: 12/20/2007 21:36  
 Weighted Avg. bhp: 130.5200  
 Work (bhp-hr): 43.5067  
 Tunnel Volume (m<sup>3</sup>/min): 70  
 Total Volume (ft<sup>3</sup>): 49441

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	266.22
benzene	519.32
toluene	315.93
ethylbenzene	137.70
m&p-xylene	252.22
o-xylene	159.89

Sample ID: **Dilution Air 61**  
 Sample Type: *Dilution Air*  
 Sample Time: 12/20/2007 21:26

Injection Time: 12/21/2007 3:03

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	0.00
benzene	7.15
toluene	36.09
ethylbenzene	64.44
m&p-xylene	28.50
o-xylene	17.75

**Sample Result**

Analyte	Conc <sub>corrected</sub> (ppbC)	Conc (ppb)	Raw Conc (ppb)	Conc (g/bhp-hr)
1,3-butadiene	<b>266.22</b>	66.56	628.28	<b>0.004816</b>
benzene	<b>512.17</b>	85.36	805.81	<b>0.008920</b>
toluene	<b>279.84</b>	39.98	377.38	<b>0.004927</b>
ethylbenzene	<b>73.26</b>	9.16	86.45	<b>0.001301</b>
m&p-xylene	<b>223.72</b>	27.97	263.99	<b>0.003972</b>
o-xylene	<b>142.14</b>	17.77	167.73	<b>0.002523</b>

Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_06\_8mlpv61.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 06\_8MLPV61

Injection Date: 12/20/2007 9:36 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\*\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 12 rows of peak data and a summary row for Group 0.

Total Unidentified Counts : 400211 counts

Selected Peaks: 132 Rejected Peaks: 1 Identified Peaks: 12

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: -14 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 43 microVolts

Manual injection

Error Log:

3800 GC:

800 GC:

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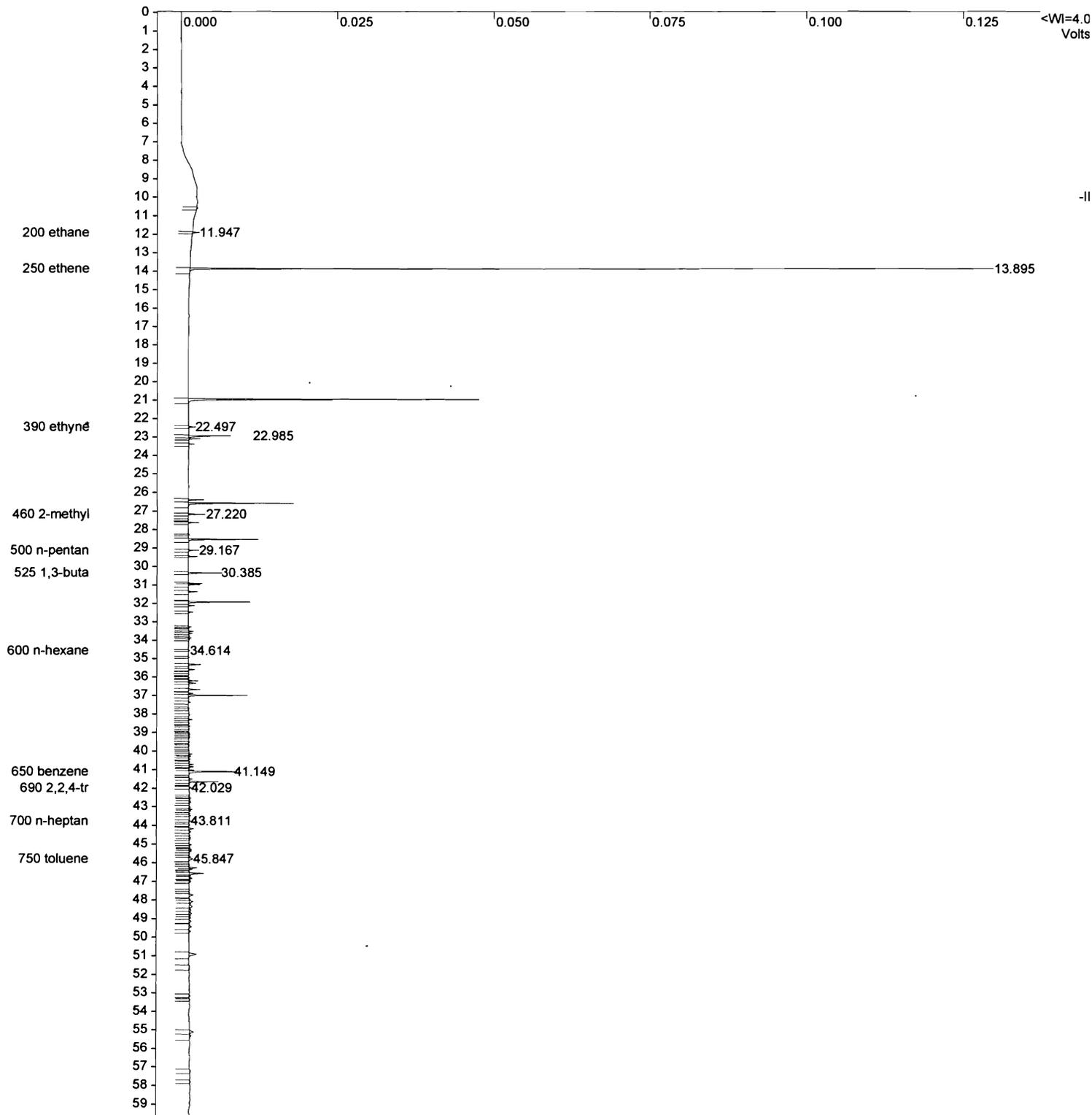
Title : GC# 1 - LE S/N08901  
In File : c:\star\data\122007\3800.45\_06\_8mlpv61.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 06\_8MLPV61

Injection Date: 12/20/2007 9:36 PM Calculation Date: 12/21/2007 1:59 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚiě Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 1%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



itle : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_06\_8mlpv61.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 06\_8MLPV61

Injection Date: 12/20/2007 9:36 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Status Codes:

- User-defined peak endpoint(s)

Total Unidentified Counts : 25219426 counts

Detected Peaks: 199 Rejected Peaks: 2 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -81 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 17 microVolts

Manual injection

Error Log:

3800 GC:

800 GC:

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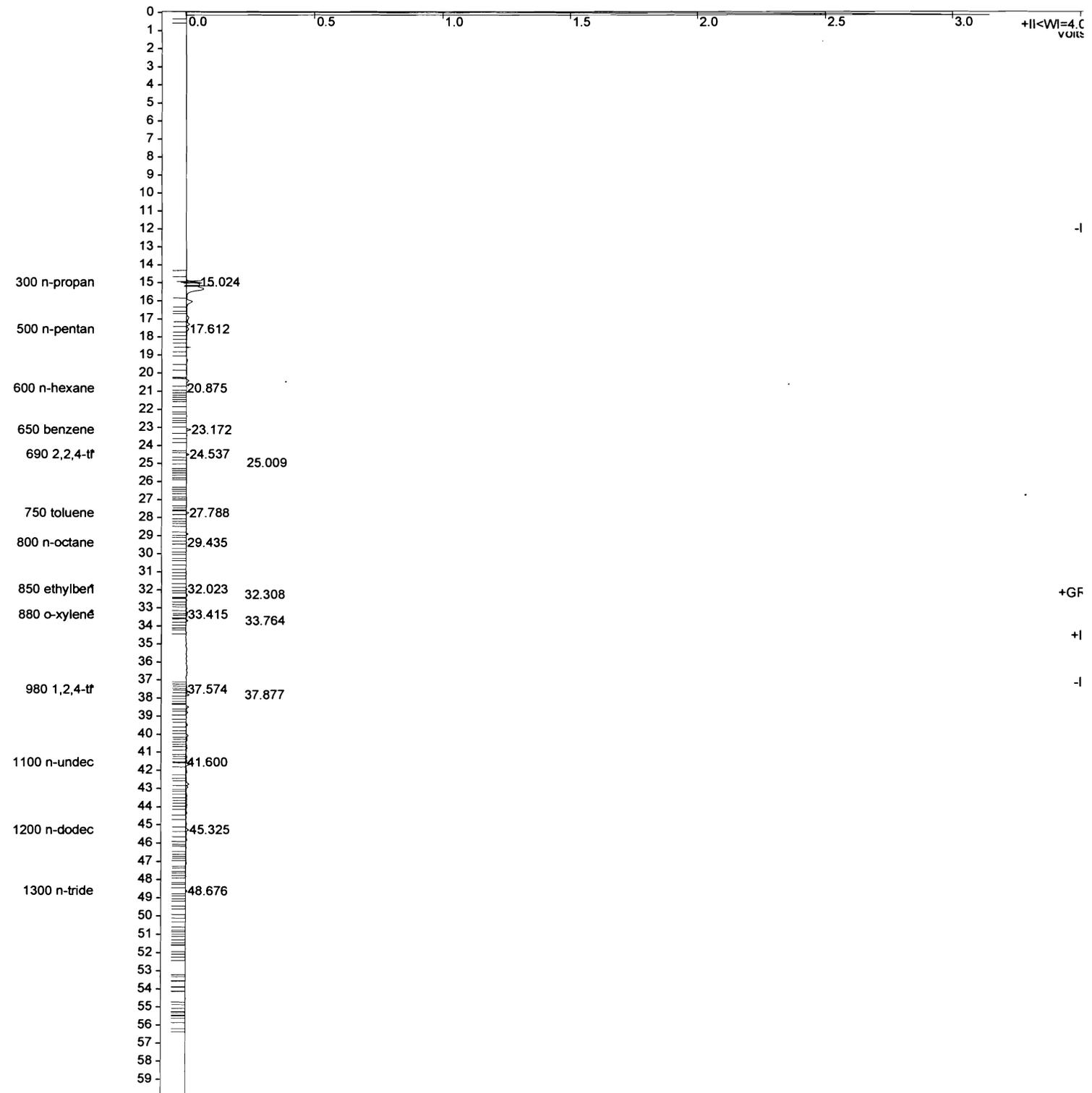
Title : GC# 1 - MR S/N08902  
 Run File : c:\star\data\122007\3800.44\_06\_8mlpv61.run  
 Method File : c:\star\methods\methods\121707.mth  
 Sample ID : 06\_8MLPV61

Injection Date: 12/20/2007 9:36 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
 Workstation: STARWORKSTATION HÚië Bus Address : 44  
 Instrument : Lotus THS Sample Rate : 10.00 Hz  
 Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 14 Zero Offset = 2%  
 Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_07\_8mlpv61 dup.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 07\_8MLPV61 Dup

Injection Date: 12/20/2007 10:41 PM Calculation Date: 12/21/2007 2:15 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HUIE Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 12 rows of peak data and a Totals row.

Status Codes:
U - User-defined peak endpoint(s)

Total Unidentified Counts : 417901 counts

Detected Peaks: 131 Rejected Peaks: 0 Identified Peaks: 12

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: 7 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 39 microVolts

Manual injection

Error Log:

0800 GC:

0800 GC:

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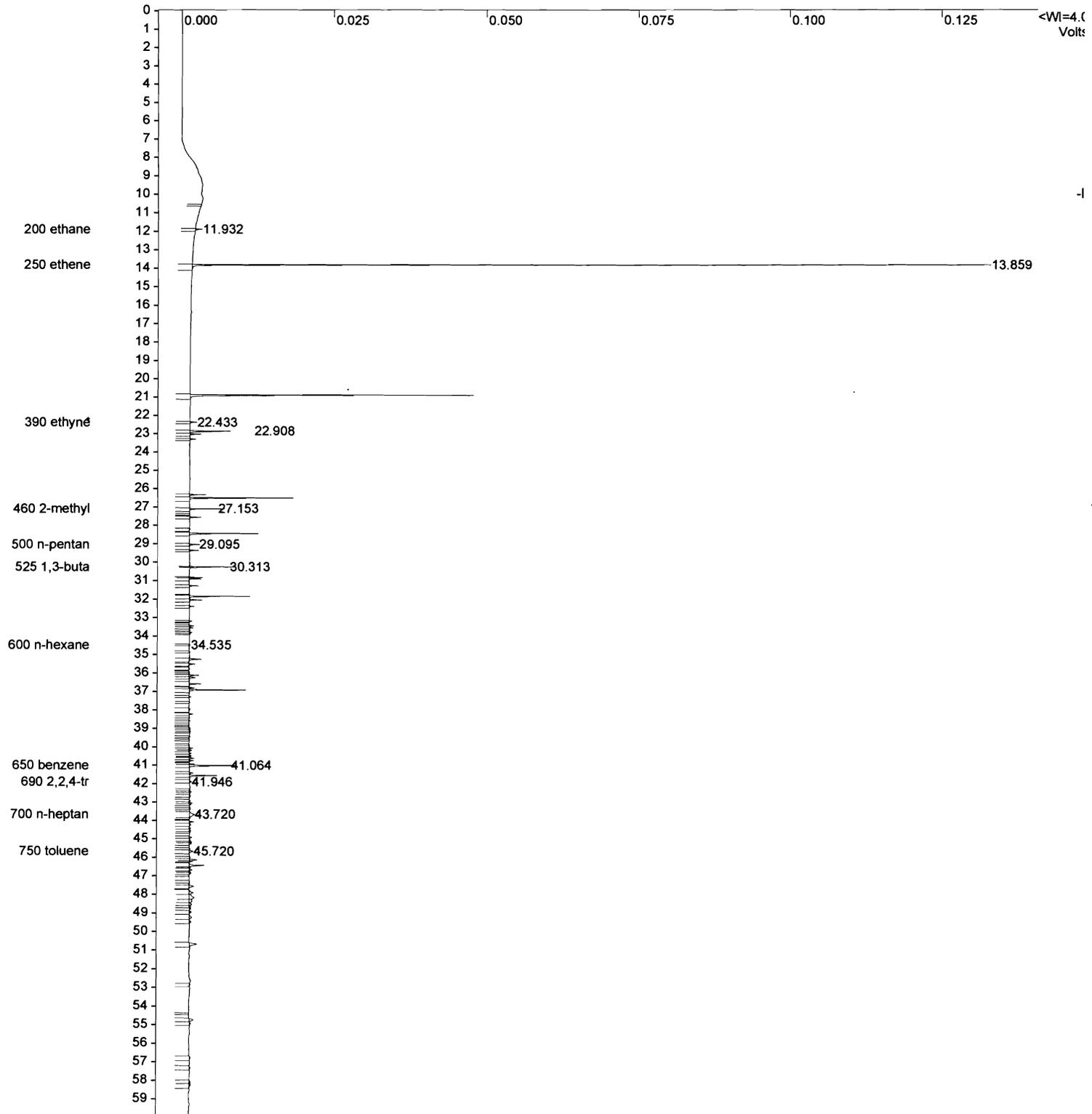
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_07\_8mlpv61 dup.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 07\_8MLPV61 Dup

Injection Date: 12/20/2007 10:41 PM Calculation Date: 12/21/2007 2:15 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 1%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_07\_8mlpv61 dup.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 07\_8MLPV61 Dup

Injection Date: 12/20/2007 10:41 PM Calculation Date: 12/21/2007 12:01 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and summary rows for Group 0 and Totals.

Total Unidentified Counts : 4820572 counts

Detected Peaks: 196 Rejected Peaks: 3 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -10 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 31 microVolts

Manual injection

Error Log:

3800 GC:

800 GC:

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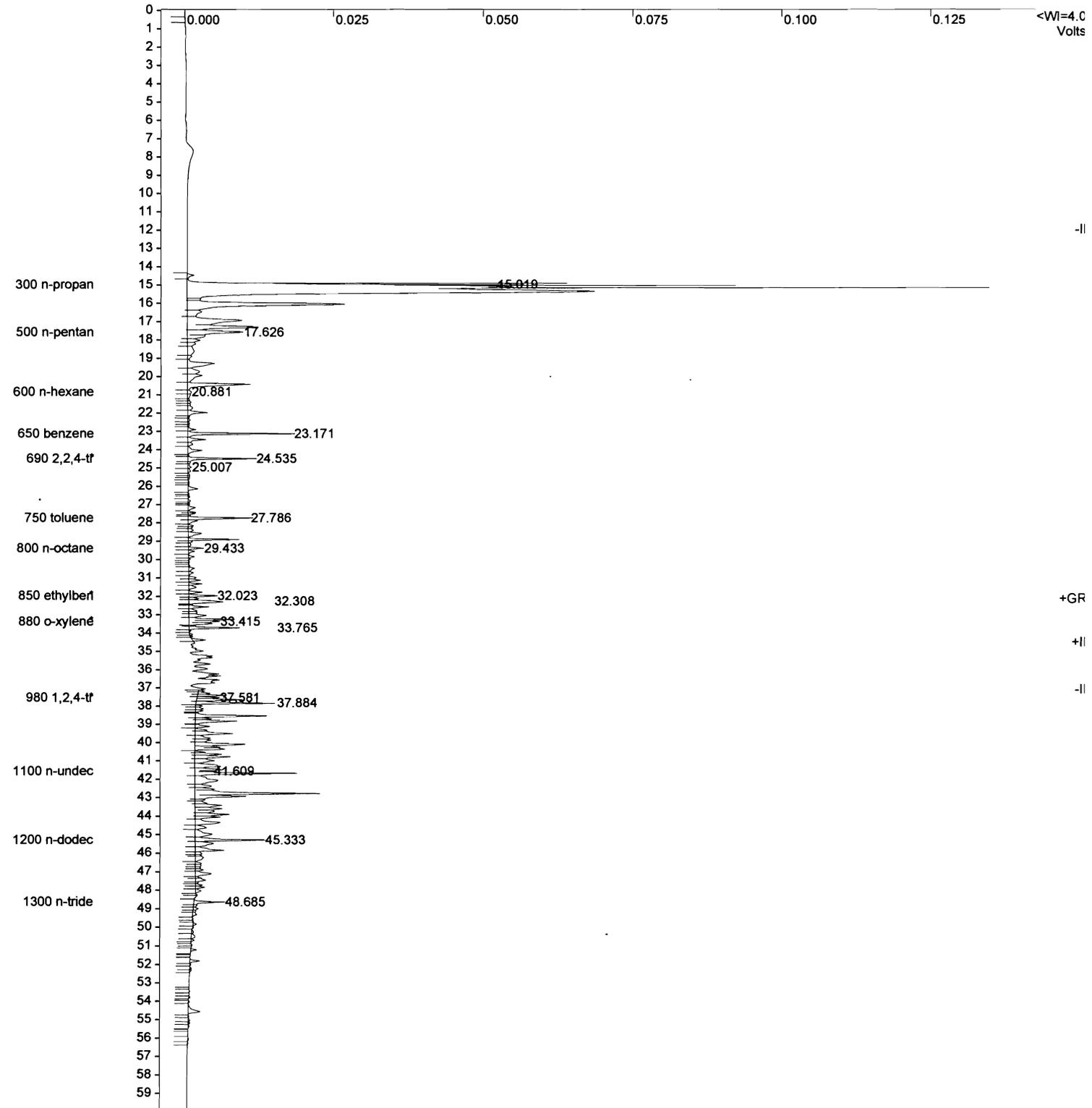
Title : GC# 1 - MR S/N08902  
Run File : c:\star\data\122007\3800.44\_07\_8mlpv61 dup.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 07\_8MLPV61 Dup

Injection Date: 12/20/2007 10:41 PM      Calculation Date: 12/21/2007 12:01 PM

Operator : NI      Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië      Bus Address : 44  
Instrument : Lotus THS      Sample Rate : 10.00 Hz  
Channel : Front = FID      Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min      Attenuation = 1      Zero Offset = 1%  
Start Time = 0.000 min      End Time = 59.940 min      Min / Tick = 1.00



Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_11\_dilution air 61.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 11\_Dilution Air 61

Injection Date: 12/21/2007 3:03 AM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.938 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 10 rows of peak data and a Totals row.

Total Unidentified Counts : 38094 counts

Detected Peaks: 28 Rejected Peaks: 0 Identified Peaks: 10

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: -24 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 36 microVolts

Manual injection

Error Log:

3800 GC:

3800 GC:

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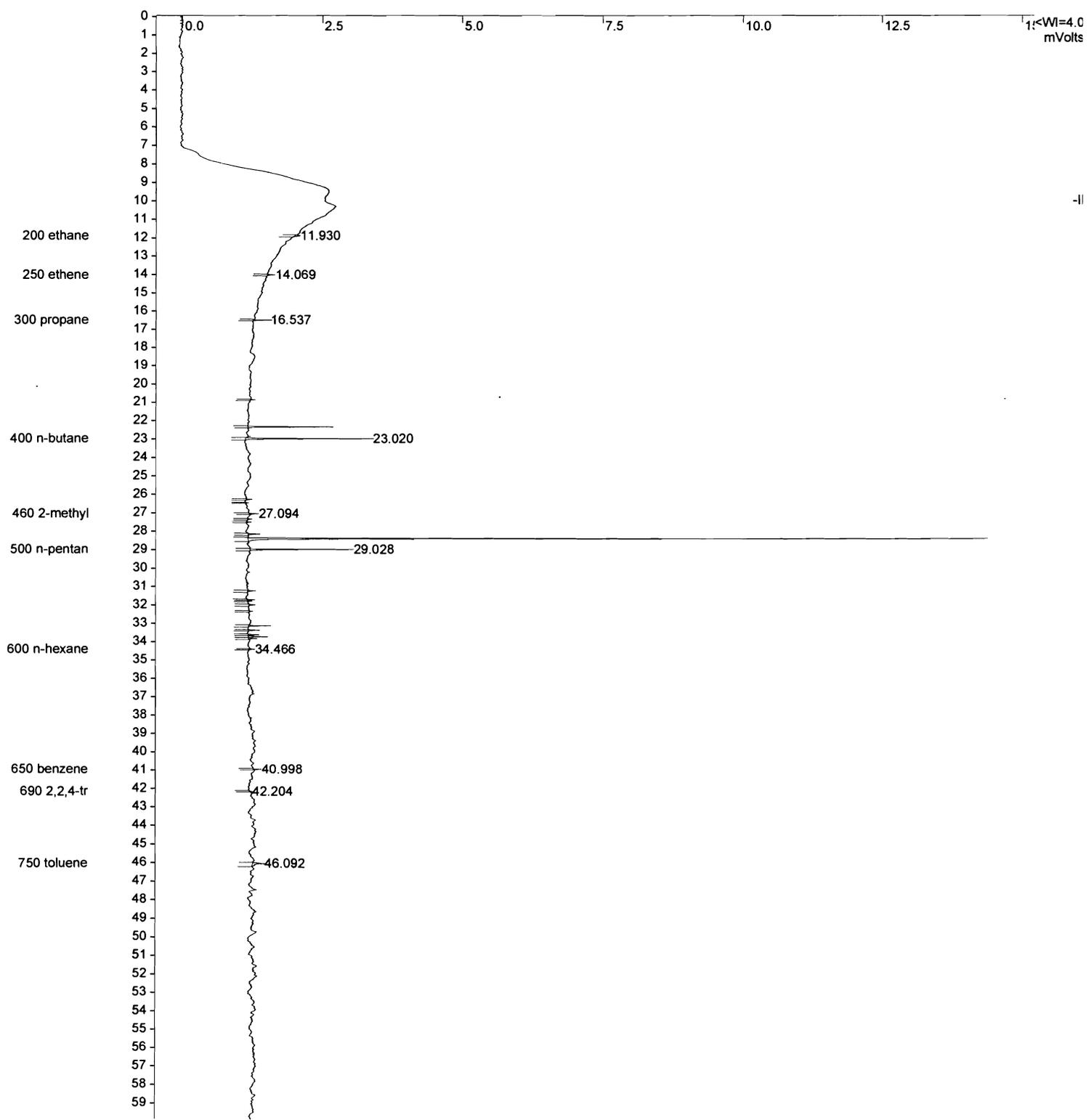
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_11\_dilution air 61.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 11\_Dilution Air 61

Injection Date: 12/21/2007 3:03 AM      Calculation Date: 12/21/2007 2:02 PM

Operator : NI      Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië      Bus Address : 45  
Instrument : Lotus THS      Sample Rate : 10.00 Hz  
Channel : Front = FID      Run Time : 59.938 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min      Attenuation = 1      Zero Offset = 0%  
Start Time = 0.000 min      End Time = 59.938 min      Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_11\_dilution air 61.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 11\_Dilution Air 61

Injection Date: 12/21/2007 3:03 AM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Total Unidentified Counts : 698050 counts

Detected Peaks: 147 Rejected Peaks: 4 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: 24 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 26 microVolts

Manual injection

Error Log:

3800 GC:

800 GC:

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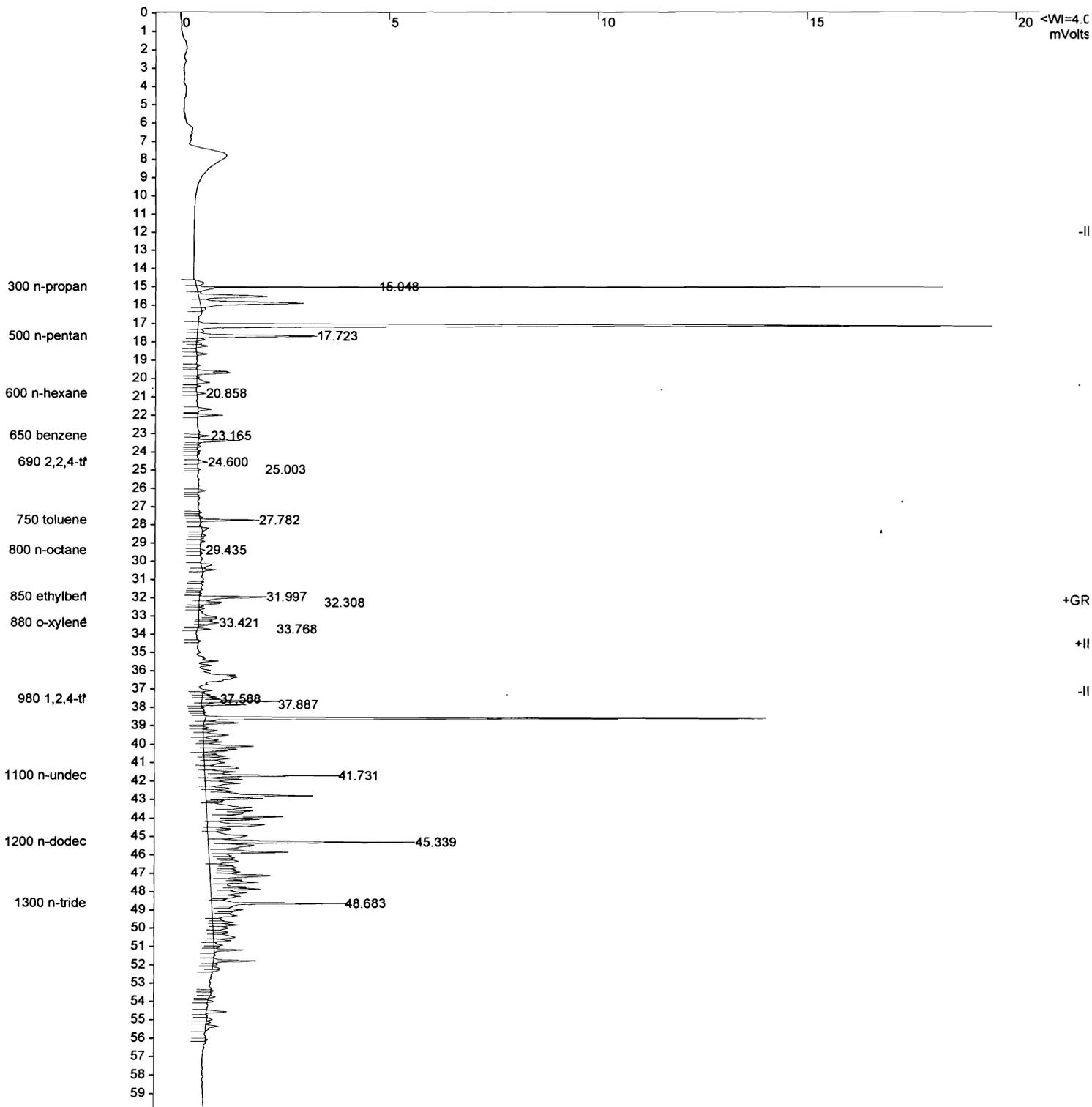
Title : GC# 1 - MR S/N08902  
Run File : c:\star\data\122007\3800.44\_11\_dilution air 61.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 11\_Dilution Air 61

Injection Date: 12/21/2007 3:03 AM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 44  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 0%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**8-Mode Steady State Trace Toxics by Gas Chromatography Results**

Chemist: Nathan Imus

Date: 12/21/2007

**QA/QC**

**Tunnel Blank**

Sample ID: **1220 Tun Blank**  
 Sample Type: *Tunnel Air*  
 Sample Time: 12/20/2007 10:48

Comments:  
 Injection Time: 12/20/2007 10:57

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	0.00
benzene	10.24
toluene	93.16
ethylbenzene	36.87
m&p-xylene	27.79
o-xylene	35.07

Sample ID: **Tun Dilution Air**  
 Sample Type: *Dilution Air*  
 Sample Time: 12/20/2007 10:48

Injection Time: 12/20/2007 12:05

Analyte	Conc <sub>uncorrected</sub> (ppbC)
1,3-butadiene	0.00
benzene	10.02
toluene	96.77
ethylbenzene	10.03
m&p-xylene	38.66
o-xylene	22.25

**Sample Result**

Analyte	Conc <sub>corrected</sub> (ppbC)	Conc (ppb)
1,3-butadiene	<RL	<RL
benzene	0.22	<b>0.04</b>
toluene	<RL	<RL
ethylbenzene	26.84	<b>3.36</b>
m&p-xylene	<RL	<RL
o-xylene	12.82	<b>1.60</b>

## QA/QC (cont.)

## Duplicate Analysis (uncorrected)

Analyte	<u>Sample ID</u>	<u>Sample ID</u>	RPD (%)
	<b>8MLPV61</b>	<b>8MLPV61 Dup</b>	
	Conc (ppbC)	Conc (ppbC)	
1,3-butadiene	266.22	307.20	14.29
benzene	519.32	526.13	1.30
toluene	315.93	313.39	0.81
ethylbenzene	137.70	139.45	1.26
m&p-xylene	252.22	253.15	0.37
o-xylene	159.89	158.84	0.66

## Crossover Check (uncorrected)

Sample ID: **8MLPV61**

Analyte	Conc <sub>Low Range</sub> (ppbC)	Conc <sub>Mid Range</sub> (ppbC)	RPD (%)
benzene	502.97	519.32	3.20

Title :
Run File : c:\star\data\122007\3800.45\_01\_1220 tun blank.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 01\_1220 Tun Blank

Injection Date: 12/20/2007 10:57 AM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.938 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 9 columns: Peak No., Peak Name, Result, Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Status Codes. Rows include ethane, ethene, propane, ethyne, n-butane, 2-methyl, n-pentan, n-hexane, benzene, and a Totals row.

Total Unidentified Counts : 70806 counts

Detected Peaks: 37 Rejected Peaks: 0 Identified Peaks: 9

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: -28 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 18 microVolts

Manual injection

Revision Log:

Stream: 2, Advance Time: 10:57:04 AM

\*\*\*\*\*

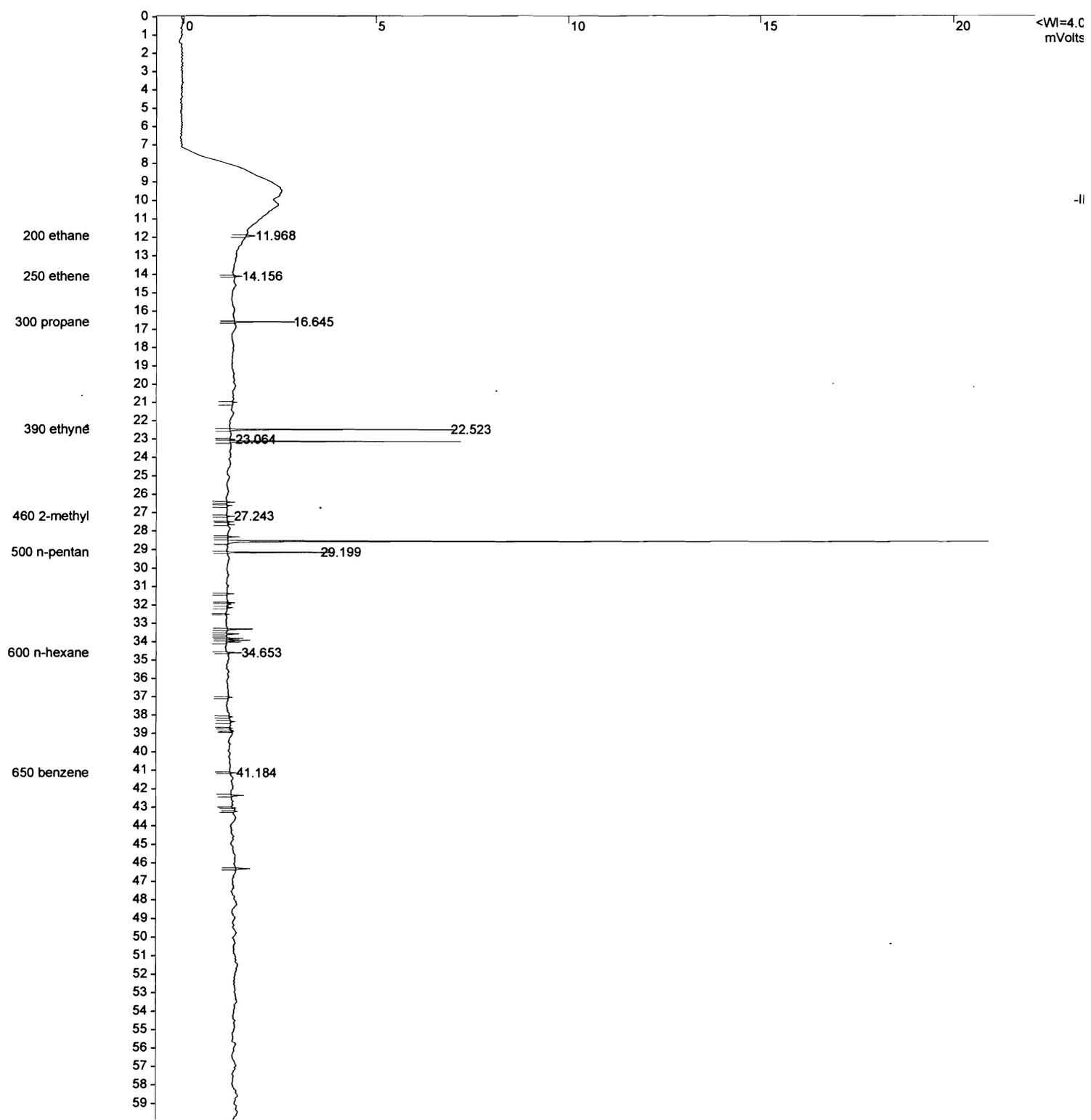
Title :  
Run File : c:\star\data\122007\3800.45\_01\_1220 tun blank.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 01\_1220 Tun Blank

Injection Date: 12/20/2007 10:57 AM      Calculation Date: 12/21/2007 2:02 PM

Operator : NI      Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië      Bus Address : 45  
Instrument : Lotus THS      Sample Rate : 10.00 Hz  
Channel : Front = FID      Run Time : 59.938 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min      Attenuation = 1      Zero Offset = 0%  
Start Time = 0.000 min      End Time = 59.938 min      Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_01\_1220 tun blank.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 01\_1220 Tun Blank

Injection Date: 12/20/2007 10:57 AM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚÍë Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Status Codes:

- User-defined peak endpoint(s)

Total Unidentified Counts : 20534416 counts

Detected Peaks: 156 Rejected Peaks: 5 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -43 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 26 microVolts

Manual injection

Error Log:

1800 GC:

800 GC:

\*\*\*\*\*

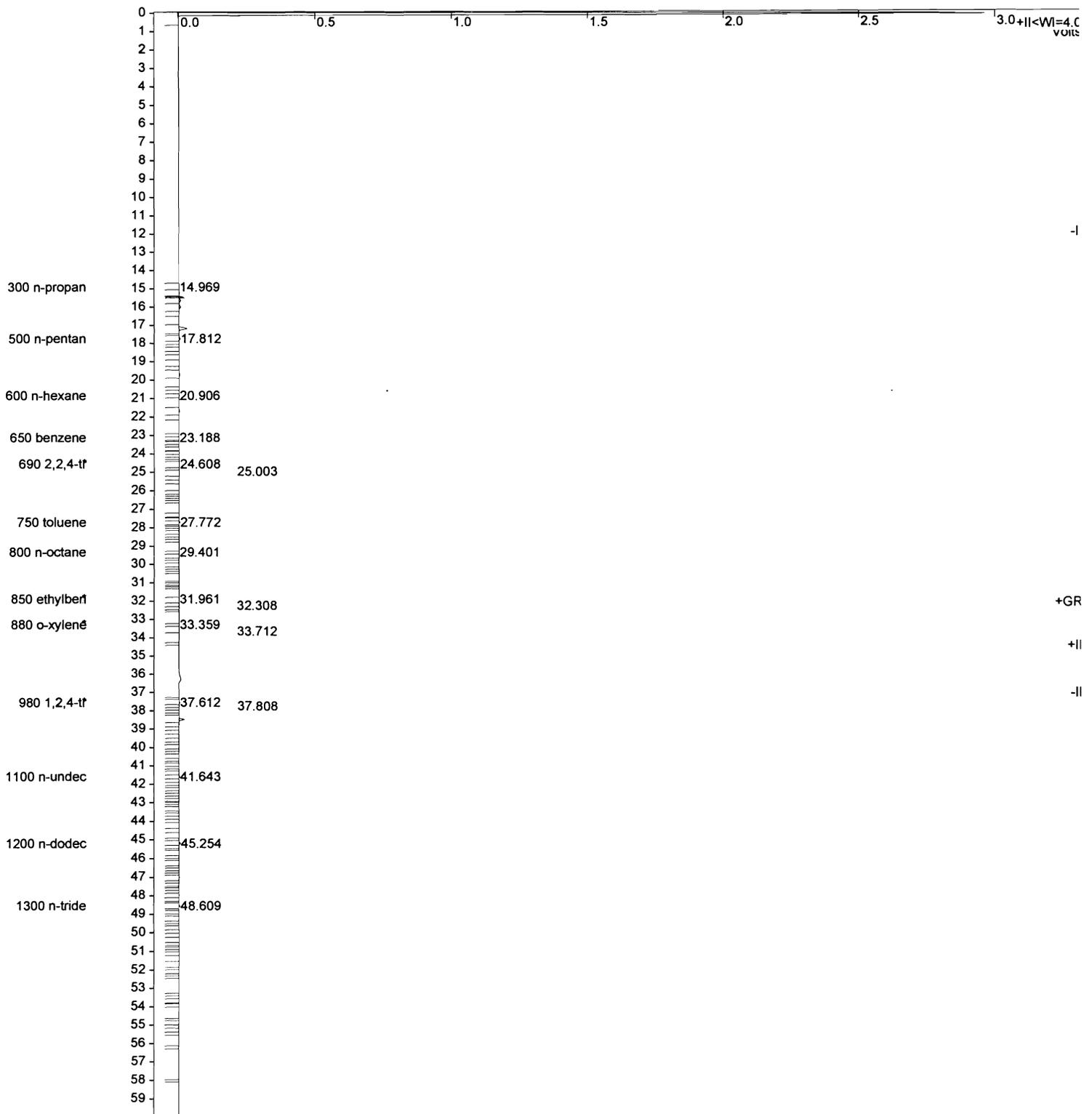
Title : GC# 1 - MR S/N08902  
 un File : c:\star\data\122007\3800.44\_01\_1220 tun blank.run  
 ethod File : c:\star\methods\methods\121707.mth  
 Sample ID : 01\_1220 Tun Blank

njection Date: 12/20/2007 10:57 AM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
 orkstation: STARWORKSTATIOy HÚië Bus Address : 44  
 nstrument : Lotus THS Sample Rate : 10.00 Hz  
 Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 14 Zero Offset = 2%  
 Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - LE S/N08901
Run File : c:\star\data\122007\3800.45\_02\_tun dilution air.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 02\_Tun Dilution Air

Injection Date: 12/20/2007 12:05 PM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HÚië Bus Address : 45
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\*\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 10 rows of peak data and a Totals row.

Total Unidentified Counts : 81053 counts

Detected Peaks: 39 Rejected Peaks: 0 Identified Peaks: 10

Multiplier: 1 Divisor: 41.73 Unidentified Peak Factor: 1

Baseline Offset: -22 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 31 microVolts

Manual injection

Error Log:

800 GC:

3800 GC:

\*\*\*\*\*

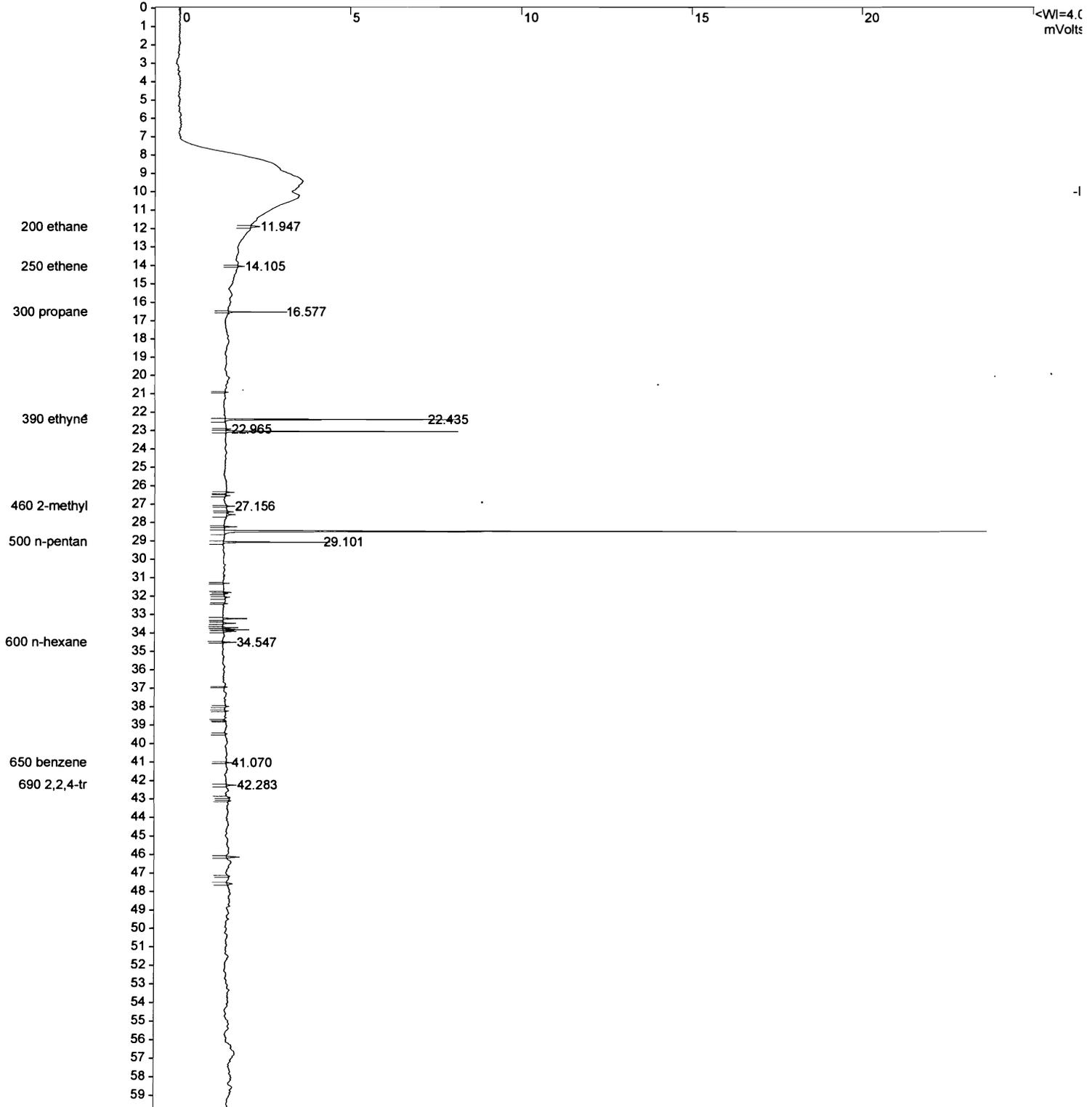
Title : GC# 1 - LE S/N08901  
Run File : c:\star\data\122007\3800.45\_02\_tun dilution air.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 02\_Tun Dilution Air

Injection Date: 12/20/2007 12:05 PM Calculation Date: 12/21/2007 2:02 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION Bus Address : 45  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 0%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



Title : GC# 1 - MR S/N08902
Run File : c:\star\data\122007\3800.44\_02\_tun dilution air.run
Method File : c:\star\methods\methods\121707.mth
Sample ID : 02\_Tun Dilution Air

Injection Date: 12/20/2007 12:05 PM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)
Workstation: STARWORKSTATION HUIE Bus Address : 44
Instrument : Lotus THS Sample Rate : 10.00 Hz
Channel : Front = FID Run Time : 59.940 min

\*\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: External Standard

Table with 10 columns: Peak No., Peak Name, Result (ppbC), Ret. Time (min), Time Offset (min), Area (counts), Sep. Code, Width 1/2 (sec), Group, Status Codes. Contains 17 rows of peak data and a Totals row.

Status Codes:

U - User-defined peak endpoint(s)

Total Unidentified Counts : 1222659 counts

Detected Peaks: 157 Rejected Peaks: 4 Identified Peaks: 17

Multiplier: 1 Divisor: 178.12 Unidentified Peak Factor: 1

Baseline Offset: -25 microVolts LSB: 1 microVolts

Noise (used): 25 microVolts - fixed value
Noise (monitored before this run): 31 microVolts

Manual injection

Error Log:

800 GC:

800 GC:

\*\*\*\*\*

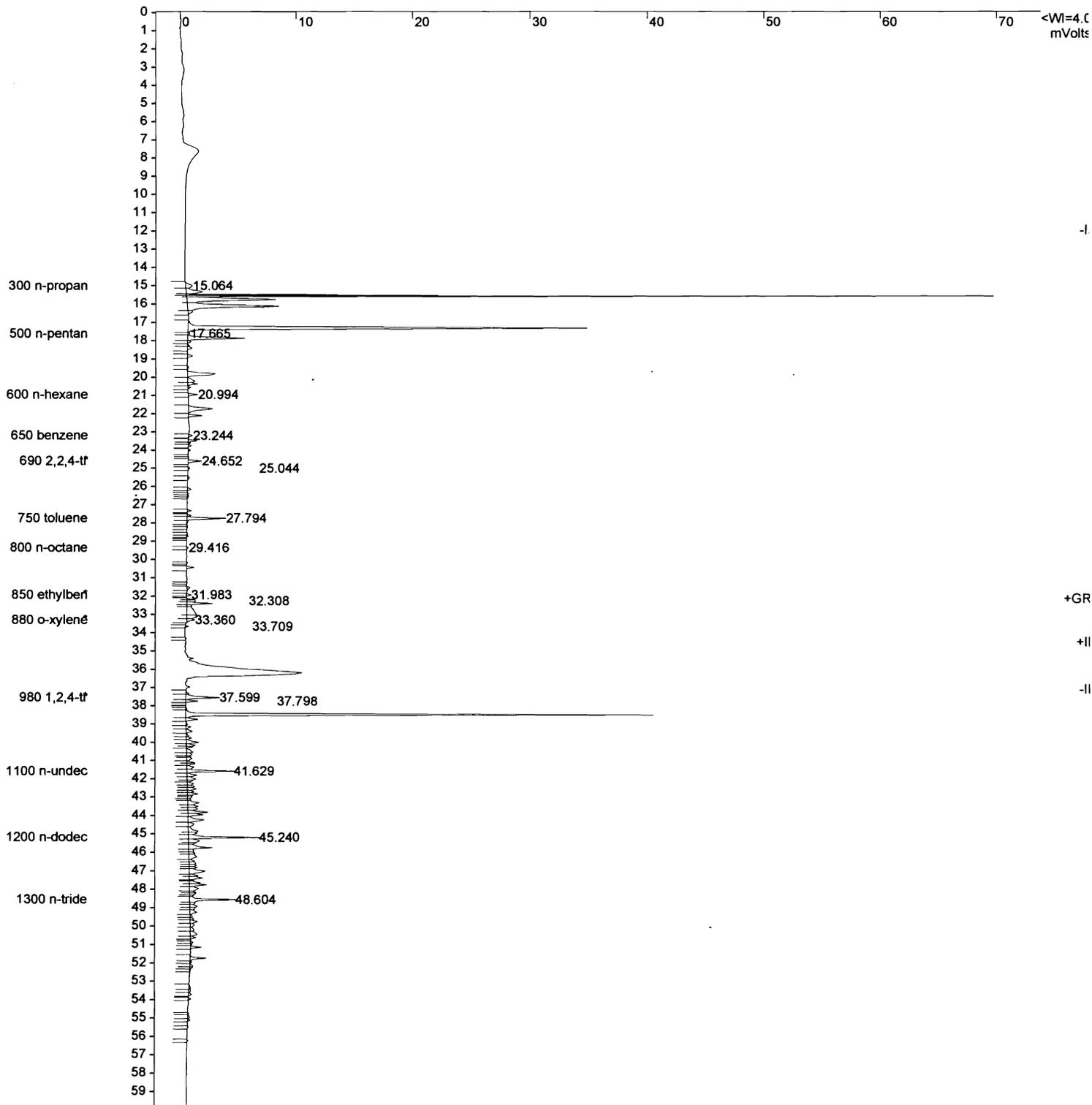
Title : GC# 1 - MR S/N08902  
Run File : c:\star\data\122007\3800.44\_02\_tun dilution air.run  
Method File : c:\star\methods\methods\121707.mth  
Sample ID : 02\_Tun Dilution Air

Injection Date: 12/20/2007 12:05 PM Calculation Date: 12/21/2007 12:11 PM

Operator : NI Detector Type: 3800 (1000 Volts)  
Workstation: STARWORKSTATION HÚië Bus Address : 44  
Instrument : Lotus THS Sample Rate : 10.00 Hz  
Channel : Front = FID Run Time : 59.940 min

\* GC Workstation Version 6.41 \*\* 04469-2780-826-1335 \*\*

Chart Speed = 0.34 cm/min Attenuation = 1 Zero Offset = 0%  
Start Time = 0.000 min End Time = 59.940 min Min / Tick = 1.00



**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**Steady State Carbonyls by High Performance Liquid Chromatography Results**

Chemist: *Nathan Imus*

Date: *1/8/2008*

**Sample Analysis**

Sample ID: **8MLPV52**

Comments: *w/ Viscon*

Sample Type: *Dilute Exhaust*

Sample Date: *12/11/07*

Injection Date: *12/19/07*

Collection (min): *20*

Weighted Avg. bhp: *131.8900*

Work (bhp-hr): *43.9633*

Tunnel Volume (m<sup>3</sup>/min): *70*

Total Volume (ft<sup>3</sup>): *49441*

Analyte	Conc (ppbv)	Uncertainty (ppbv)	Conc (g/bhp-hr)	Uncertainty (g/bhp-hr)
formaldehyde	824.03	56.31	<b>0.032759</b>	0.002238
acetaldehyde	123.72	18.35	<b>0.007214</b>	0.001070
acetone	4.74	1.19	<b>0.000364</b>	0.000092
acrolein	2.39	0.83	<b>0.000178</b>	0.000062
propionaldehyde	13.39	1.80	<b>0.001030</b>	0.000139
crotonaldehyde	0.00	0.69	<b>0.000000</b>	0.000064
methyl ethyl ketone	0.29	0.79	<b>0.000028</b>	0.000075
methacrolein	0.49	0.69	<b>0.000045</b>	0.000064
butyraldehyde	5.43	0.86	<b>0.000519</b>	0.000083
benzaldehyde	2.49	0.69	<b>0.000350</b>	0.000098
glyoxal	0.00	0.41	<b>0.000000</b>	0.000032
valeraldehyde	0.00	0.65	<b>0.000000</b>	0.000074
m-tolualdehyde	1.07	0.61	<b>0.000170</b>	0.000098
hexanaldehyde	3.17	0.79	<b>0.000420</b>	0.000105

**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**Steady State Carbonyls by High Performance Liquid Chromatography Results**

Chemist: *Nathan Imus*

Date: *1/8/2008*

**Sample Analysis**

Sample ID: **8MLPV53**

Comments: *w/ Viscon*

Sample Type: *Dilute Exhaust*

Sample Date: *12/11/07*

Injection Date: *12/19/07*

Collection (min): *20*

Weighted Avg. bhp: *131.4800*

Work (bhp-hr): *43.8267*

Tunnel Volume (m<sup>3</sup>/min): *70*

Total Volume (ft<sup>3</sup>): *49441*

Analyte	Conc (ppbv)	Uncertainty (ppbv)	Conc (g/bhp-hr)	Uncertainty (g/bhp-hr)
formaldehyde	655.90	46.57	<b>0.026156</b>	0.001857
acetaldehyde	106.82	16.33	<b>0.006248</b>	0.000955
acetone	21.53	1.91	<b>0.001660</b>	0.000148
acrolein	17.89	2.41	<b>0.001332</b>	0.000180
propionaldehyde	14.32	1.91	<b>0.001104</b>	0.000148
crotonaldehyde	9.60	1.47	<b>0.000894</b>	0.000136
methyl ethyl ketone	2.52	0.91	<b>0.000241</b>	0.000087
methacrolein	0.00	0.69	<b>0.000000</b>	0.000065
butyraldehyde	5.34	0.82	<b>0.000511</b>	0.000078
benzaldehyde	5.33	0.96	<b>0.000751</b>	0.000135
glyoxal	0.00	0.41	<b>0.000000</b>	0.000032
valeraldehyde	0.00	0.65	<b>0.000000</b>	0.000074
m-tolualdehyde	2.13	0.66	<b>0.000340</b>	0.000105
hexanaldehyde	2.38	0.71	<b>0.000316</b>	0.000094

**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**Steady State Carbonyls by High Performance Liquid Chromatography Results**

Chemist: *Nathan Imus*

Date: *1/8/2008*

**Sample Analysis**

Sample ID: **8MLPV54**

Comments: *w/ Viscon*

Sample Type: *Dilute Exhaust*

Sample Date: *12/11/07*

Injection Date: *12/20/07*

Collection (min): *20*

Weighted Avg. bhp: *130.5700*

Work (bhp-hr): *43.5233*

Tunnel Volume (m<sup>3</sup>/min): *70*

Total Volume (ft<sup>3</sup>): *49441*

Analyte	Conc (ppbv)	Uncertainty (ppbv)	Conc (g/bhp-hr)	Uncertainty (g/bhp-hr)
formaldehyde	775.94	53.51	<b>0.031159</b>	0.002149
acetaldehyde	125.35	18.67	<b>0.007383</b>	0.001100
acetone	8.14	1.29	<b>0.000632</b>	0.000100
acrolein	6.76	1.21	<b>0.000507</b>	0.000091
propionaldehyde	14.63	1.97	<b>0.001136</b>	0.000153
crotonaldehyde	1.08	0.70	<b>0.000101</b>	0.000065
methyl ethyl ketone	0.49	0.79	<b>0.000047</b>	0.000076
methacrolein	0.69	0.69	<b>0.000064</b>	0.000065
butyraldehyde	6.69	0.98	<b>0.000645</b>	0.000094
benzaldehyde	2.58	0.73	<b>0.000366</b>	0.000104
glyoxal	0.00	0.41	<b>0.000000</b>	0.000032
valeraldehyde	0.00	0.65	<b>0.000000</b>	0.000075
m-tolualdehyde	0.98	0.61	<b>0.000158</b>	0.000099
hexanaldehyde	3.34	0.79	<b>0.000448</b>	0.000106

**Olson-EcoLogic Engine Testing Laboratories, LLC**  
**Carbonyls by High Performance Liquid Chromatography Results**

Chemist: *Nathan Imus*

Date: *1/8/2008*

**QA/QC**

**Tunnel Blank**

Sample ID: **1211 Tun Blank**

Comments:

Sample Type: *Tunnel Air*

Sample Date: *12/11/07*

Injection Date: *12/19/07*

Analyte	Conc (ppbv)	Uncertainty (ppbv)
formaldehyde	0.06	7.81
acetaldehyde	0.60	3.03
acetone	6.70	1.24
acrolein	0.00	0.74
propionaldehyde	0.00	0.58
crotonaldehyde	0.00	0.69
methyl ethyl ketone	0.87	0.82
methacrolein	0.00	0.69
butyraldehyde	0.00	0.41
benzaldehyde	0.00	0.61
glyoxal	0.00	0.41
valeraldehyde	0.00	0.65
m-tolualdehyde	0.00	0.58
hexanaldehyde	0.00	0.62

**Field Blank**

Sample ID: **Field Blank**

Comments:

Sample Type: *Field Air*

Sample Date: *12/11/07*

Injection Date: *12/21/07*

Analyte	Conc (ppbv)	Uncertainty (ppbv)
formaldehyde	5.50	0.89
acetaldehyde	3.16	0.84
acetone	7.73	0.64
acrolein	0.00	0.52
propionaldehyde	0.00	0.41
crotonaldehyde	0.00	0.49
methyl ethyl ketone	2.33	0.59
methacrolein	0.00	0.49
butyraldehyde	0.00	0.29
benzaldehyde	0.00	0.43
glyoxal	0.00	0.29
valeraldehyde	0.00	0.46
m-tolualdehyde	0.00	0.41
hexanaldehyde	0.00	0.44



## Department of Toxic Substances Control



Linda S. Adams  
Secretary for  
Environmental Protection

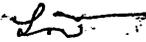
Maureen F. Gorsen, Director  
1001 "I" Street  
P.O. Box 806  
Sacramento, California 95812-0806



Arnold Schwarzenegger  
Governor

### MEMORANDUM

**TO:** Bob Okamoto  
Air Pollution Specialist  
Air Resources Board

**FROM:** Li Tang   
Multimedia Products Section  
Department of Toxic Substances Control

**SUBJECT:** Reply to Viscon's "Response to Memorandum DTSC Comments"  
Regarding Multi Media Evaluation (Tier II)

**DATE:** February 24, 2009

Viscon has responded to Department of Toxic Substances Control's (DTSC) December 23, 2008 comments on Viscon's Multi Media Evaluation (Tier II) report. Viscon's response:

- 1) Explains that a 500 ppm of Viscon contains 5 ppm of PIB;
- 2) Provides a copy of a renewed license for Elastol as an oil spill cleanup agent;
- 3) Emphasizes that attachments 6, 9, 10, and 11 in the Tier II report were provided as historical background information; and
- 4) Includes a copy of a letter prepared by HCI Environmental & Engineering Service (HCIEES) Stating that a recent jet fuel spill that the company had contracted to clean up would have been better contained because Viscon had been added to the fuel.

DTSC appreciates the explanation in item 1 and the copy of the renewed Elastol license in item 2, but has questions about the other two points that Viscon makes. Specifically:

Item 3 above: The historical information provided by Viscon does not address our concerns. Descriptions and explanations that connect the Viscon properties to the historical information are necessary in the Tier II report.

Item 4 above: Viscon's response appears contain conflicting information. In the first paragraph, Viscon states that the diesel viscosity has "immediate and temporary increase" due to trace Viscon addition and later, Viscon argues that it would not be

February 24, 2009

Page 2

reasonable to require laboratory testing of the properties of soil contaminated with Viscon fuel. They base this argument on the very low PIB concentration. The HCIEES letter did not support its claim that jet fuel spills with Viscon have been better contained than spills of regular fuel. If this is a fact, DTSC would like to review the supporting data. DTSC does not find the assertion by HCIEES that Viscon is "just more of the same that is already being dealt with (hydrocarbons), regarding these widely used and popular technologies" to be persuasive. For example, it is common for a long chain hydrocarbon to be non-biodegradable, or less biodegradable than a short chain hydrocarbon. As mentioned in the Air Resources Board's comments, PIB "may be highly persistent in environmental media." It is unknown if the persistence affects the diesel cleanup from soil. This is the reason that DTSC requests laboratory tests to avoid the future environmental impact.

If you have any questions, please contact me at (916)323- 9757, or my staff Li Tang at (916).322-2505, or [ltang@dtsc.ca.gov](mailto:ltang@dtsc.ca.gov). Thank you.

cc: Sherri Lehman, Chief  
Toxics in Products Branch  
Office of Pollution Prevention and Green Technology  
Department of Toxic Substances Control

Andre Algazi, Chief  
Consumer Products Section  
Office of Pollution Prevention and Green Technology  
Department of Toxic Substances Control

Aubrey Sideco  
Air Resources Section, SSD  
California Air Resources Board

**Question:** Comments on “Viscon Multi Media Evaluation.”

*The information on health effects of PIB is not adequate for an evaluation of potential adverse impacts. Such information may be unavailable because this substance has not been tested for health effects. The PIB used in Viscon has a very large molecular weight so that it is unlikely to pass through the wall of the mammalian gastrointestinal tract. The PIB does not appear to be susceptible to biological oxidation, reduction or other type of metabolic reaction. Therefore, it may be highly persistent in environmental media.*

*The document is inconsistent in the comparison of Viscon emissions with emissions from combustion of the control fuel (CARB diesel). I recommend that all comparisons be calculated as (Viscon emission minus CARB diesel emission) divided by CARB diesel emission where both the Viscon emission term and the CARB diesel emission term are calculated as the amount of the substance emitted per horsepower-hour of work performed by the engine used in the test.*

**Response:**

Section 2708 of the Verification Procedure, Warranty and In-Use Compliance Requirements for In-Use Strategies to Control Emissions from Diesel Engines (Verification Procedure) provides specific guidance regarding the determination of emissions reductions from diesel emission control strategies. Applicants are required to calculate emissions reductions for a specific diesel emission control strategy based on the average of all valid test results before and after implementation of the strategy. The Verification Procedure characterizes the test results before any changes are made to the test engine or test fuel as “baseline”, and those tests performed after changes as “control”. The percent reduction is the difference between the average baseline and average control emissions divided by the average baseline emissions, multiplied by 100 percent.

Unfortunately, the use of the term “control” as required by the Verification Procedure has recently led to some confusion. In the scientific community, the term control is generally used to refer to a sample in which a factor whose effect is being tested is absent or is held constant. Therefore, it is easy to understand how some may inadvertently view the results of the “control” emissions tests as if they were baseline results. It may help the reader to think of the “control” fuel as the “modified” fuel when comparing to the baseline fuel.

The convention used throughout both the Tier I and Tier II reports from Viscon is that all test results before any changes are made are referred to as “baseline” and all test results after changes have been made are referred to as “control”.



May 22, 2009

In response to the DTSC Question concerning Dosage levels dated December 23, 2008. To calculate the dosage rate, 1 oz. of Viscon goes into 20 gallons of diesel. This would be 1 divided by 2560 = .00039. The amount of PIB in the 1 oz of Viscon is 1% of the total. 1% PIB / 99% diesel. 390 PPM divided by 100 = 3.9 PPM. 3.9 PPM is less than 5 parts per million.



February 05, 2009

Mr. Michael Porter  
Viscon USA  
3121 Standard Street  
Bakersfield, CA 93308  
(661) 327-7061 / Office  
(661) 327-7743 / Facsimile

**Re: The Effects of Viscon (Polyisobutylene) on Current Disposal/Recycling Options for Petroleum Contaminated Soils**

Dear Mike:

Thank you for contacting our firm, regarding the above referenced product and its effects on current technologies available for the proper disposal and/or recycling of petroleum contaminated soils.

First, let us confirm that the individual principals of this firm, to include myself, have been actively involved in the proper remediation of petroleum contaminated soils for twenty years. We have performed soil remediation through the following technologies, over those twenty years:

- a) Vapor Extraction
- b) Bio-Remediation
- c) Thermal Desorption
- d) Landfill and/or Land Cover

We have steered our clients over the years towards those recycling options listed above. While twenty years ago, recycling was not an option for everyone, mainly due to cost, our industry has matured. Recycling is now available as well as affordable to most, which have the unpleasant fortune of contaminated soil to contend with.

As we write this letter to you, we have been contracted by AIG Insurance to perform a clean-up of jet fuel at the Mojave Airport. Our Emergency Response Crews have been dispatched and we will begin (under the direction of Kern County Environmental Health) the arduous task of remediation, transportation and recycling of petroleum contaminated soil. This client would have been better served, had Viscon been in the jet fuel that spilled. The spill would have been better contained (to the clients liking as well as our environments), due to Viscon's ability to retard the petroleum products from permeating as quickly.



S. California  
Los Angeles Metro  
(Corporate Office)  
114 Business Center Drive  
Corona, Ca 92880  
800.988.4424  
951.280.0118 Fax

N. California  
Sacramento Metro  
915 L. Street, Ste. C  
Sacramento, Ca 95814  
800.988.4424  
916.444.3204 Fax

Nevada  
Las Vegas Metro  
101 S. Rainbow Blvd  
Las Vegas, NV 89128  
800.988.4424  
702.221.2711 Fax



Mr. Michael Porter

**Viscon USA**

Page 2 of 2

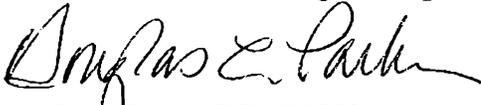
Additionally and in order to answer your question directly, Viscon being a long hydrocarbon chain would have posed no issues, as it relates to the recycling options available to us today.

While most of our clients choose to either Bio-Remediate or Thermal Desorption, these technologies either “eat,” hydrocarbon chains by utilizing bugs or “thermally destroy” hydrocarbon chains, by burning/incineration. Viscon, simply said, is just more of the same that is already being dealt with (hydrocarbons), regarding these widely used and popular technologies.

Please do not hesitate to contact me directly, if you have any additional questions or if we can be of further assistance.

Sincerely,

**HCI Environmental & Engineering Service**

A handwritten signature in black ink, appearing to read "Douglas L. Parker". The signature is fluid and cursive, with a long horizontal stroke at the end.

Douglas L. Parker, REA I-08091  
Registered Environmental Assessor I

Sk:DLP



DEPARTMENT OF FISH AND GAME

<http://www.dfg.ca.gov>

Office of Spill Prevention and Response

1700 K Street, Suite 250

Sacramento, CA 95814

Telephone: (916) 327-9960



July 24, 2007

Mr. Terry Politi  
Technical Representative  
General Technologies Application Inc.  
7400 Gallerher Rd.  
Gainesville, VA 20155

Dear Mr. Politi:

Thank you for your interest in renewing the license for the product "Elastol". We received and reviewed the renewal information submitted and it meets the regulatory requirements. Enclosed is your license for the Oil Spill Cleanup Agent "Elastol." Unless revoked for good cause, this license is valid until August 10, 2012.

Please be aware that licensing of a product as an oil spill clean up agent by the Office of Spill Prevention and Response in no way constitutes an endorsement by the State of California nor necessitates that the product will be used at the time of a spill. Please be advised that the appropriateness of product use will be assessed on a case-by-case basis at the time oil is released into the waters of the State and is contingent upon approval by the Administrator of the Office of Spill Prevention and Response and the Federal Regional Response Team, Region IX. Finally, the Office of Spill Prevention and Response restricts the use of loose particulate materials for purposes of on-water spill response. As such, loose particulate materials must be contained, either in pillows, socks, or within the confines of booms or booming operations, for use in cleanup for all waters of the State. These restrictions are necessary to ensure that large quantities of foreign materials are not lost to the environment where they can become available to wildlife.

Should you have any questions or need any additional information regarding the licensing of your product, please contact Ms. Yvonne Najah Addassi of my staff at telephone number (916) 324-7626.

Sincerely,

Ms. Lisa Curtis  
Acting Administrator  
Office of Spill Prevention and Response

Enclosure

*Conserving California's Wildlife Since 1870*

**State of California  
Department of Fish and Game  
Office of Spill Prevention and Response**

**LICENSE FOR OIL SPILL CLEANUP AGENT**

**ISSUED TO: General Technologies Application Inc.**

<b>PRODUCT NAME:</b>	Elastol
<b>PRODUCT CLASSIFICATION:</b>	Elasticity Modifier
<b>DATE ISSUED:</b>	August 10, 2007
<b>EXPIRATION DATE:</b>	August 10, 2012

The applicant, having followed the procedures as outlined in Government Code Sections 8670.13.1 & 8670.13.2., and having submitted a proper application which was reviewed and found in order, is hereby issued this license.

Use of the product must be in strict compliance with referenced Government Code Sections and subsequent regulations, which requires approval of the Administrator of the Office of Spill Prevention and Response and the Federal Region IX Regional Response Team prior to use.

The term of this license shall be for a period of five (5) years unless revoked prior to that date for good cause.

This license does not denote endorsement of the product by the Department of Fish and Game or any other State Agency.

Lisa Curtis  
Administrator  
Office of Spill Prevention and Response

**From:** Sideco, Aubrey@ARB  
**Sent:** Tuesday, February 24, 2009 1:13 PM  
**To:** Sideco, Aubrey@ARB  
**Subject:** FW: VISCON addition comments prompted by DTSC

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**From:** Robert Hodam [mailto:RHodam@waterboards.ca.gov]  
**Sent:** Wednesday, February 18, 2009 5:34 PM  
**To:** Peterson, Jim@ARB; Sideco, Aubrey@ARB; Simeroth, Dean@ARB; Vergara, Floyd@ARB; Guthrie, Jim@ARB; Lee, John@ARB; Rosenkranz, Kirk@ARB; Lee, Linda@ARB; Schuy, Mark@ARB; Hill, Rodney@ARB; Okamoto, Bob@ARB; Daley, Shawn@ARB; Li Tang; Sherri Lehman; Sonia Low; Xiaoying Zhou; Andy Salmon; Bruce Winder; Elaine Khan; Hristo Hristov; Melanie Marty; Page Painter; James Giannopoulos  
**Cc:** Bloudoff, Dean@ARB; Benjamin Heningburg; Laura Fisher  
**Subject:** RE: VISCON addition comments prompted by DTSC

To: Bob Okamoto — the comments below are based on my recent conversations with Li Tang, DTSC. Please add these comments to my previous comments.

**Documentation of the effect of PIB on soil characteristics and biodegradability**

DTSC has requested documentation of the effects of a release of Viscon-diesel blend into soil compared to a diesel release into soil. The applicant Viscon California LLC has not provided any scientific data to confirm their 'common sense' conclusion that the mobility of diesel in soil is decreased, or to document the change in biodegradability of Viscon-diesel blends.

Viscon is basically polyisobutylene (PIB). This relatively non-toxic chemical has been used for some years to clean up marine petroleum product spills in water and on beaches, including formal authorization to do so by the DWQ, State Water Resources Control Board. A brief internet search for data on oil spill cleanup reveals that PIB, which has an affinity for organic material, works by increasing the viscosity of petroleum products and thus decreasing the penetration of the petroleum into beach sand and by creating an adhesive elastic effect on petroleum floating on surface water so that the oil can be skimmed off.

The applicant's Tier I document indicates that PIB is to be blended < 1% in diesel to achieve the projected benefits.

One could speculate from the oil spill cleanup reports and the concentration of PIB in the proposed Viscon-diesel blend that such a blend would be more viscous and less mobile in soil, especially high organic content soil, than diesel without Viscon. One could also speculate, based on physical property data indicating that PIB has a relatively low biodegradability, that a Viscon-diesel blend would have a lower biodegradability than diesel without Viscon. However, Viscon LLC has not provided published scientific references to confirm either soil property changes or biodegradability.

Viscon LLC has, however, provided a letter dated February 5, 2009 from HCI Environmental and Engineering Service, which speculates on the hypothetical benefits of Viscon in the cleanup of a recent jet fuel spill had the fuel contained Viscon. This anecdotal opinion does not constitute evidence of purported benefits, nor does it address DTSC's concerns about changes in soil properties.

The letter also implies that bioremediation is a viable option for the cleanup of Viscon-diesel releases. That is contrary to the conclusion one might reach based on available PIB physical property data, which indicates PIB is not relatively biodegradable.

Consequently, **Viscon LLC should at least provide published scientific data documenting both the biodegradability of Viscon-diesel and its fate and transport in soil compared to diesel without Viscon. If supporting data are not available, Viscon LLC should conduct laboratory tests which compare diesel to Viscon-diesel for biodegradability and fate and transport for the most likely worst cases. They may want to consider a high organic content soil as a candidate soil for one of the fate and transport tests.**

Bob

Robert Hodam, MS Eng. MBA.  
Alternative Fuels Lead  
Division of Water Quality  
State Water Resources Control Board  
California Environmental Protection Agency  
<http://www.waterboards.ca.gov/>

1001 I St. 15th Floor  
Sacramento, CA 95814, USA

rhodam@waterboards.ca.gov  
Cell Phone (916) 705-4234  
Fax: (916) 341-5808

**From:** Sideco, Aubrey@ARB  
**Sent:** Tuesday, February 24, 2009 1:14 PM  
**To:** Sideco, Aubrey@ARB  
**Subject:** FW: VISCON TAB16\_correction to 1st email

---

**From:** Robert Hodam [mailto:RHodam@waterboards.ca.gov]  
**Sent:** Tuesday, February 17, 2009 4:56 PM  
**To:** Peterson, Jim@ARB; Sideco, Aubrey@ARB; Simeroth, Dean@ARB; Vergara, Floyd@ARB; Guthrie, Jim@ARB; Lee, John@ARB; Rosenkranz, Kirk@ARB; Lee, Linda@ARB; Schuy, Mark@ARB; Hill, Rodney@ARB; Okamoto, Bob@ARB; Daley, Shawn@ARB; Li Tang; Sherri Lehman; Sonia Low; Dr. Xiao Ying Zhou; Andy Salmon; Bruce Winder; Elaine Khan; Hristo Hristov; Melanie Marty; Page Painter; James Giannopoulos  
**Cc:** Bloudoff, Dean@ARB; Benjamin Heningburg; Laura Fisher  
**Subject:** RE: VISCON TAB16\_correction to 1st email

Aubrey,

I have reviewed Viscon's responses to the MMWG comments on their Tier I submission, and re-reviewed their Tier I documents; I have no additional comments or questions.

In fact, upon re-review of the physical characteristics of polyisobutylene, this product seems idea in terms of restricting the mobility of diesel leaked from a UST and thus reducing the cost of cleanup. Given its low solubility in water one may even be able to wash the product from all but the most organic soils.

Consultation with Water Board toxicologists confirms that polyisobutylene appears to pose no significant toxicology issues (mammalian LD50 = 2000 mg/kg).

regards, Bob

Robert Hodam, MS Eng. MBA.  
Alternative Fuels Lead  
Division of Water Quality  
State Water Resources Control Board  
California Environmental Protection Agency  
<http://www.waterboards.ca.gov/>

1001 I St. 15th Floor  
Sacramento, CA 95814, USA

rhodam@waterboards.ca.gov  
Cell Phone (916) 705-4234  
Fax: (916) 341-5808



Mr. Michael Porter, President  
Viscon USA  
3121 Standard Street  
Bakersfield, Ca 93308  
661.327.7061 / Office  
661.327.7743 / Facsimile  
[Lpodmikeporter@aol.com](mailto:Lpodmikeporter@aol.com)

Dear Mr. Porter

In regards to the recent letter from the DTSC regarding a long chain hydrocarbon to be non-biodegradable, this is simply false. At Western Environmental Inc., we have extensive knowledge of the bio-remediation process. A long chain hydrocarbon is biodegradable; however the time to break the chain is the concerning factor in the process. We have had success with reducing the hydrocarbon chains of C18 to C43 in a 90 day period. Included is test data labeled CLNFL 006- Screened, this is our base line data at the start of the treatment process. Also included is the test data labeled CLNFL 006-7, this is the seventh sample taken of the base line material after 90 days. As you can see, the total hydrocarbon concentration of the soil went from 813 ppm to 311 ppm. The concentration of the microbes was 0.3 gallons of brew per ton at a rate of 52.5 gallons per hour. The soil was watered daily and aerated based on temperature increases and plateaus. The time is the main factor in the degradation of the hydrocarbon molecules into their constituent parts, carbon dioxide and water. If an additive of < 1% polyisobutylene (PIB) was added to the hydrocarbons this would prolong the process of degradation, but it certainly would not stop the process.

Thermal treatment on the other hand is also another treatment option. The thermal desorption process involves the heating the petroleum contaminated soil to a temperature of 500 to 700 degrees Fahrenheit to volatilize the entrained petroleum contaminants. These process gasses then enter the bag house for particulate removal then the air stream is duct into the thermal oxidizer where they are heated to a

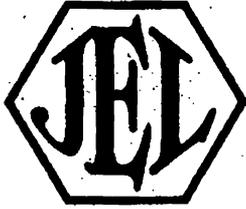
**62-150 Gene Welmas Dr. Mecca, CA. 92254 - Phone (760)396-0222 Fax (760)396-4300**  
**Website: [wei-mecca.com](http://wei-mecca.com)**



minimum temperature of 1400 degrees. At this high temperature the volatilized hydrocarbons are oxidized and destroyed.

Both processes achieve the same result, but the obvious concern is the time it takes to get the desired result. With the proper resources and time, the bioremediation of long chain hydrocarbons is a viable method of treatment.

Matthew Mullen  
QA/QC, Compliance Manager



# Jones Environmental, Inc.

Testing Laboratories

P.O. Box 5387 • Fullerton, CA 92838  
(714) 449-9937 • FAX (714) 449-9685

JONES ENVIRONMENTAL

## LABORATORY REPORT

Client: Western Environmental, Inc.  
Client Address: P.O. Box 248  
Mecca, CA 92254

Report Date: 02/15/08  
JEL Ref. No.: ST-4084

Project: WINDROWS-006  
Project Address: 62-150 Gene Welmas, Mecca, CA

Date Sampled: 02/08/08  
Date Received: 02/11/08  
Date Analyzed: 02/14/08  
Physical State: Soil

### ANALYSES REQUESTED

1. Mod 8015 Diesel - Simulated Distillation Extended Range (EPA 2887)

Approval:

Steve Jones, Ph.D.  
Laboratory Manager



# Jones Environmental, Inc.

## Testing Laboratories

P.O. Box 5387 • Fullerton, CA 92838  
(714) 449-9937 • FAX (714) 4499685

### JONES ENVIRONMENTAL

### QUALITY CONTROL INFORMATION

<b>Client:</b>	Western Environmental, Inc.	<b>Report Date:</b>	02/15/08
<b>Client Address:</b>	P.O. Box 248 Mecca, CA 92254	<b>JEL Ref. No.:</b>	ST-4084
<b>Project:</b>	WINDROWS-006	<b>Date Sampled:</b>	02/08/08
<b>Project Address:</b>	62-150 Gene Welmas, Mecca, CA	<b>Date Received:</b>	02/11/08
		<b>Date Analyzed:</b>	02/14/08
		<b>Physical State:</b>	Soil

### Modified 8015 Diesel (Simulated Distillation Extended Range)

Sample Spiked: CLEAN SOIL

<u>Parameter</u>	<u>MS Recovery (%)</u>	<u>MSD Recovery (%)</u>	<u>RPD</u>	<u>Acceptability Range (%)</u>
Diesel	106%	99%	7.4%	65 - 125

Method Blank = Not Detected

- MS = Matrix Spike
- MSD = Matrix Spike Duplicate
- RPD = Relative Percent Difference



# Jones Environmental, Inc.

## Testing Laboratories

P.O. Box 5387 • Fullerton, CA 92838  
(714) 449-9937 • FAX (714) 4499685

### JONES ENVIRONMENTAL

### LABORATORY RESULTS

<b>Client:</b>	Western Environmental, Inc.	<b>Report Date:</b>	02/15/08
<b>Client Address:</b>	P.O. Box 248 Mecca, CA 92254	<b>JEL Ref. No.:</b>	ST-4084
<b>Project:</b>	WINDROWS-006	<b>Date Sampled:</b>	02/08/08
<b>Project Address:</b>	62-150 Gene Welmas, Mecca, CA	<b>Date Received:</b>	02/11/08
		<b>Date Analyzed:</b>	02/14/08
		<b>Physical State:</b>	Soil

### Modified 8015 Diesel (Simulated Distillation Extended Range)

<u>Carbon Chain Range</u>	Sample ID Concentration (mg/Kg)	
	<u>CLNFL006- B</u>	<u>CLNFL006- SCREENED</u>
C6-C7	ND	ND
C8-C9	3.0	29
C10-C11	7.2	7.0
C12-C13	23	24
C14-C15	41	41
C16-C17	54	56
C18-C19	48	60
C20-C23	102	147
C24-C27	103	158
C28-C31	106	159
C32-C35	44	91
C36-C39	17	31
C40-C43	5.8	10
C44+	ND	ND
<b>Total</b>	<b>554</b>	<b>813</b>
<b>Surrogate Recovery Hexacosane %</b>		
<b>Acceptance Range: 65% - 125%</b>	--♦	--♦
<b>Dilution Factor</b>	1	1
<b>Practical Quantitation limits</b>	10	10

ND = Not Detected

# Chain-of-Custody Record

Client <b>WESTERN ENVIRONMENTAL</b>	Date <b>02-08-2008</b>
Project Name <b>WINDROWS-006</b>	Client Project #
Project Address <b>62-150 GENE WELLS DR. MECCA, CA. 92254</b>	Turn Around Requested: <input type="checkbox"/> Immediate Attention <input type="checkbox"/> Rush 24-48 Hours <input checked="" type="checkbox"/> Rush 72-96 Hours <input type="checkbox"/> Normal <input type="checkbox"/> Mobile Lab
Project Contact <b>MATTHEW MULLEN</b>	

JEL Project #  
**ST4084**

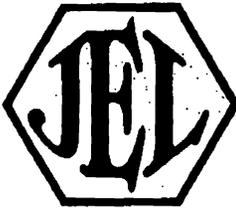
Page **1** of **1**

Lab Use Only

Sample Condition as Received:  
 Chilled  yes  no  
 Sealed  yes  no

Sample ID	Discussion	Date	Time	Laboratory Sample Number	Sample Matrix: Soil (S), Sludge (SL), Aqueous (A), Soil Gas (SG)	Analysis Requested	Number of Containers	Remarks/Special Instructions
<b>CLNF006-B</b>		<b>02-08-08</b>	<b>2PM</b>		<b>S X</b>		<b>1</b>	<b>E-MAIL TO:</b>
<b>CLNF006-SCREENED</b>		<b>02-08-08</b>	<b>2PM</b>		<b>S X</b>		<b>1</b>	<b>MATTHEW@WEST-MECCA.COM</b>

1 Relinquished by (signature) 	Date <b>02-08-08</b>	2 Received by (signature) 	Date <b>2/11/08</b>	2	Total Number of Containers
Company <b>WESTERN ENVIRONMENTAL</b>	Time <b>2:30 PM</b>	Company <b>JEI</b>	Time <b>15:00</b>		
3 Relinquished by (signature)	Date	4 Received by Laboratory (signature)	Date	The delivery of samples and the signature on this Chain of Custody form constitutes authorization to perform the analyses specified above under the Terms and Conditions set forth on the back hereof. 	
Company	Time	Company	Time		



# Jones Environmental, Inc.

Testing Laboratories

P.O. Box 5387 • Fullerton, CA 92838  
(714) 449-9937 • FAX (714) 449-9685

JONES ENVIRONMENTAL

## LABORATORY REPORT

**Client:** Western Environmental, Inc.  
**Client Address:** P.O. Box 248  
Mecca, CA 92254

**Report Date:** 05/20/08  
**JEL Ref. No.:** ST-4231

**Project:** FILL SOIL  
**Project Address:** 62-150 Gene Welmas, Mecca, CA

**Date Sampled:** 05/09/08  
**Date Received:** 05/12/08  
**Date Analyzed:** 05/15/08  
**Physical State:** Soil

### ANALYSES REQUESTED

1. Mod 8015 Diesel - Simulated Distillation Extended Range (EPA 2887)

Approval:



Steve Jones, Ph.D.  
Laboratory Manager



# Jones Environmental, Inc.

## Testing Laboratories

P.O. Box 5387 • Fullerton, CA 92838  
(714) 449-9937 • FAX (714) 4499685

### JONES ENVIRONMENTAL

### LABORATORY RESULTS

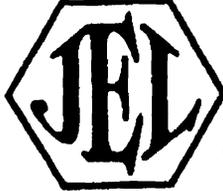
<b>Client:</b>	Western Environmental, Inc.	<b>Report Date:</b>	05/20/08
<b>Client Address:</b>	P.O. Box 248 Mecca, CA 92254	<b>JEL Ref. No.:</b>	ST-4231
<b>Project:</b>	FILL SOIL	<b>Date Sampled:</b>	05/09/08
<b>Project Address:</b>	62-150 Gene Welmas, Mecca, CA	<b>Date Received:</b>	05/12/08
		<b>Date Analyzed:</b>	05/15/08
		<b>Physical State:</b>	Soil

#### Modified 8015 Diesel (Simulated Distillation Extended Range)

	Sample ID Concentration (mg/Kg)
<u>Carbon Chain Range</u>	<u>CLNFL-</u>
	<u>006-7</u>
C6-C7	ND
C8-C9	ND
C10-C11	ND
C12-C13	ND
C14-C15	29.4
C16-C17	41.8
C18-C19	37.0
C20-C23	49.3
C24-C27	62.4
C28-C31	62.6
C32-C35	28.5
C36-C39	ND
C40-C43	ND
C44+	ND
Total	311
<b>Surrogate Recovery Hexacosane %</b>	
<u>Acceptance Range: 65% - 125%</u>	--♦
<b>Dilution Factor</b>	1
<b>Practical Quantitation limits</b>	10

ND = Not Detected

♦ = High Hydrocarbon concentration in this sample prevented adequate surrogate recovery



# Jones Environmental, Inc.

Testing Laboratories

P.O. Box 5387 • Fullerton, CA 92838  
(714) 449-9937 • FAX (714) 4499685

## JONES ENVIRONMENTAL

### QUALITY CONTROL INFORMATION

<b>Client:</b>	Western Environmental, Inc.	<b>Report Date:</b>	05/20/08
<b>Client Address:</b>	P.O. Box 248 Mecca, CA 92254	<b>JEL Ref. No.:</b>	ST-4231
<b>Project:</b>	FILL SOIL	<b>Date Sampled:</b>	05/09/08
<b>Project Address:</b>	62-150 Gene Welmas, Mecca, CA	<b>Date Received:</b>	05/12/08
		<b>Date Analyzed:</b>	05/15/08
		<b>Physical State:</b>	Soil

### Modified 8015 Diesel (Simulated Distillation Extended Range)

Sample Spiked: Clean Soil

<u>Parameter</u>	<u>MS Recovery (%)</u>	<u>MSD Recovery (%)</u>	<u>RPD</u>	<u>Acceptability Range (%)</u>
Diesel	100%	100%	0.1%	65 - 125

Method Blank = Not Detected

MS = Matrix Spike  
MSD = Matrix Spike Duplicate  
RPD = Relative Percent Difference

# Chain-of-Custody Record

Client <i>Western Environmental</i>		Date <i>5-9-08</i>		Analysis Requested <i>Soil (S), Sludge (SL), Aqueous (A), Soil Gas (SG)</i> <i>DOES NOT</i>				JEL Project # <i>ST4231</i>
Project Name <i>Fill Soil</i>		Client Project #						Page <i>1 of 1</i>
Project Address <i>6750 Evans Holmes Dr - Woodbridge, CA 97254</i>		Turn Around Requested: <input type="checkbox"/> Immediate Attention <input type="checkbox"/> Rush 24-48 Hours <input type="checkbox"/> Rush 72-96 Hours <input checked="" type="checkbox"/> Normal <input type="checkbox"/> Mobile Lab						Lab Use Only Sample Condition as Received: Chilled <input type="checkbox"/> yes <input type="checkbox"/> no Sealed <input type="checkbox"/> yes <input type="checkbox"/> no
Project Contact <i>Matthew Mullen</i>								

Sample ID	Discussion	Date	Time	Laboratory Sample Number	Sample Matrix: Soil (S), Sludge (SL), Aqueous (A), Soil Gas (SG)	Number of Containers	Remarks/Special Instructions
<i>PLNPL006-7</i>		<i>5-9-08</i>	<i>930</i>		<i>DOES NOT</i>	<i>1</i>	

① Relinquished by (signature) <i>Matthew Mullen</i>	Date <i>5-9-08</i>	② Received by (signature) <i>Karen Prance</i>	Date <i>5/12/08</i>	Total Number of Containers <i>1</i>
Company <i>Western Environmental</i>	Time <i>1015</i>	Company <i>JEL</i>	Time <i>1400</i>	
③ Relinquished by (signature)	Date	④ Received by Laboratory (signature)	Date	The delivery of samples and the signature on this Chain of Custody form constitutes authorization to perform the analyses specified above under the Terms and Conditions set forth on the back hereof. <i>Matthew Mullen</i>
Company	Time	Company	Time	



March 13, 2009

Dear Dr. Tang

Our response to questions and comments are set forth below. However, since there appears to be some misunderstanding about the content of our Tier II report, we would like to summarize certain aspects of Viscon as they appear in the Introduction to the Multi Media Evaluation.

First, the Tier II report covers both the Viscon product and the Viscon/diesel blend. The Viscon product is comprised of 99% CARB diesel and 1% high molecular weight PIB. The dosage rate for Viscon in diesel fuel is about 500 PPM with a PIB content of about 5 PPM.

Second, the PIB used in Viscon is a food grade material. In a lower molecular weight it is used in most of the chewing gum on the market in the United States.

#### Answers to Questions and Comments

1. Attachments 6, 9, 10 and 11 of the Tier II report do not address the concerns of her office. She asks that Viscon connect the Viscon properties to use of historical information in these papers.

Each of the attachments was offered as evidence that the Viscon product does not pose an environmental hazard.

Attachment 6 is a license granted by the California Water Resources Board for the use of Elastol as an oil spill cleanup agent. Elastol is comprised of finely ground high molecular weight PIB like the PIB in Viscon. Both come from the same supplier, BASF, and differ only in that Elastol has a molecular weight of about 6.3 million while the PIB in Viscon has a molecular weight of about 7 million. The license for Elastol was granted after tests for toxicity required by the California Water Resources Board, were carried out and reported to the Board. A summary of this test is attachment 14 of the Tier II report.

Viscon believes that the work carried out for the California Water Resources Board, and the Board's subsequent granting of a license support its conclusion that the active component of Viscon is not toxic in the water environment.

Attachment 9 is a report of work by Environment Canada evaluating the Elastol product described above as an oil spill recovery agent for spills on water. Elastol in these tests was evaluated in the laboratory at concentrations of 600 PPM to 6000 PPM in a series of crude oils, diesel fuel and Bunker C, and in the field in various oils at concentration of Elastol of 1000 to 9000 PPM. A conclusion of this report was that Elastol improves skimmer recovery of spilled oil on water and retards oil slick spreading.

Since the Viscon product is a solution of about 10,000 PPM in diesel fuel, the company believes that the Environment Canada test report is relevant to an evaluation of the effects of a release of Viscon to a water environment.

Attachment 10 is a report of tests by Florida International University evaluating the toxicity of weathered oils, with and without Elastol, to corals and sea grasses. A treatment concentration of 8000 PPM of Elastol to oil was included in the test. One of the conclusions of the test was that Elastol reduced the toxicity of the oil being tested.

The Elastol product, in solution in oil at 8000 PPM is similar to the 10,000 PPM PIB solution in Viscon. Viscon believes that this test data is relevant to evaluation of the effects of a release of the Viscon product into a water environment.

Attachment 11 is a report of an oil spill experiment carried out by the United States Mineral Management Service and Environment Canada. The program was to demonstrate the effectiveness of state of the art oil spill containment and recovery equipment in an open ocean environment under relatively high sea state conditions. Elastol was included in the test. One conclusion in the report is that Elastol significantly enhanced the recovery of the spilled oil.

Viscon believes this report is relevant to a release of the Viscon product to water in adverse weather and water conditions.

2. In referring to item 4 of her memorandum Dr. Tang states that Viscon is providing conflicting information, where it states that the Viscon/diesel blend has an "immediate and temporary increase" in viscosity and yet argues that it would not be reasonable to require laboratory testing of soil contaminated with Viscon/diesel fuel. We don't agree.

The phenomenon upon which the Viscon technology is based is extensional viscosity. Attachment 7 to the Tier II report is a scientific explanation of elongational viscosity. Section 2 of this paper explains that a small concentration of high molecular weight polyisobutylene (PIB) has little effect on the resting viscosity of the solution. Section 3 of the paper explains that high molecular weight PIB has a very large effect on the viscosity of a solution under strain even at very small concentrations of PIB. It is explained further that the rate of strain determines the time for a solution to increase in

viscosity. The higher the rate of strain, the more rapid the increase in viscosity. Later in the paper in Section 4 at page 4 it explains that when the strain is released elongational viscosity decreases.

The science of elongational viscosity establishes that a solution of 5 PPM of 7 million molecular weight PIB will have little or no effect on the transport of a Viscon/diesel solution in soil.

Dr. Tang also raises the question of the effect of 5 PPM of PIB in biodegradation treatment of Viscon/diesel contaminated soil. She correctly points out that high molecular weight hydrocarbons, like PIB, biodegrade more slowly than short chain hydrocarbons such as those in diesel fuel and therefore the PIB will be more persistent in the environment. It is important, however, to consider the properties of high molecular weight PIB in evaluating the environmental hazard posed by the differential in biodegradation rates of PIB and diesel fuel. The high molecular weight PIB in the Viscon product is a non-toxic, colorless, tasteless, odorless, food grade polymer which is insoluble in water. One of the uses of high molecular weight PIB produced by BASF is in food packaging. It is also a component in most of the chewing gum sold in the United States. In biodegradation treatment of contaminated soil from a 10,000 gallon spill of Viscon/diesel, 6 ounces of PIB would remain equivalent to 6 sticks of Double mint gum.

In summary Viscon believes that this is adequate support data provided by professionals in the bio-remediation industry. Further delays to the verification process of Viscon after six years of testing is unwarranted.

Respectfully,

Michael J. Porter



The Chemical Company

Helping Make  
Products Better™

April 25, 2008

Re: Oppanol® B Grades are in compliance with FDA Regulations

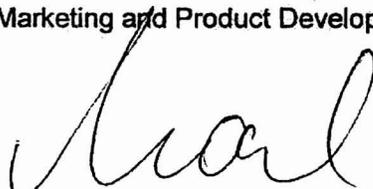
Dear Valued Oppanol® Customer:

We herewith confirm that all Oppanol® B grades fulfill the following FDA paragraphs:

21 CFR 172.615	"Chewing Gum Base"
21 CFR 175.105	"Adhesives"
21 CFR 175.125	"Pressure-Sensitive Adhesives"
21 CFR 175.300	"Resinous and Polymeric Coatings"
21 CFR 175.380	"Xylene-foraldehyde Resins condensed with 4,4-isopropylidene dephe nolepichlorohydrin Epoxy Resins"
21 CFR 175.390	"Zinc-silicon dioxide Matrix Coatings"
21 CFR 176.170	"Components of Paper and Paperboard in Contact with Aqueous and Fatty Foods"
21 CFR 176.180	"Components of Paper and Paperboard in Contact with Dry Food"
21 CFR 177.1210	"Closures with Sealing Gaskets for Food Containers"
21 CFR 178.2600	"Rubber Articles Intended for Repeated Use"
21 CFR 178.3570	"Lubricants with Incidental Food Contact"
21 CFR 178.3910	"Surface Lubricants used in the Manufacture of Metallic Articles"

Yours truly,

Performance Chemicals Automotive and Refinery Chemicals  
Global Marketing and Product Development



Dr. Helmut Mach  
Technical Marketing Manager  
BASF SE