

California Environmental Protection Agency



**Air Resources Board**

**SOP MLD 066**

**STANDARD OPERATING PROCEDURE FOR THE  
DETERMINATION OF OXYGENATES AND NITRILES IN  
AMBIENT AIR  
BY CAPILLARY COLUMN GAS  
CHROMATOGRAPHY/MASS SPECTROMETRY**

Northern Laboratory Branch  
Monitoring and Laboratory Division

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## STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF OXYGENATED AND NITRILE HYDROCARBONS IN AMBIENT AIR USING SUMMA CANISTER SAMPLING AND GAS CHROMATOGRAPHIC/MASS SPECTROMETRIC ANALYSIS

### 1.0 SCOPE

This document describes the procedures followed by Monitoring and Laboratory Division (MLD) staff to analyze oxygenated and nitrile hydrocarbons by Gas Chromatography with Mass Spectrometry detection, (GC/MS), in ambient air samples collected from the California Toxic Monitoring Network. Staff of the Northern Laboratory Branch (NLB), Organic Laboratory Section (OLS), developed the method. This Standard Operating Procedure (SOP) is based on the U.S. Environmental Protection Agency (EPA) Toxic Organic Compounds in Ambient Air Method TO-15, "Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)", EPA/625/R-96/010b, January 1999. [Table 1](#), page [21](#), lists the Target Compounds and their Chemical Abstract Service (CAS) numbers.

### 2.0 SUMMARY OF METHOD

Ambient air is collected in a SUMMA polished stainless steel canister using a Xontech 910A sampler. The sampling procedure for Toxic samples is detailed in the Air Resources Board Quality Assurance Manual, Volume II, Appendix Q. All the operational procedures and sampling conditions for each sample are documented in the field. A record of this information is sent back to the OLS along with the sample. Upon receipt, the sample canister pressure is measured with a calibrated external pressure gauge. This information and particulars of the collection are documented in the laboratory. The sample is then analyzed according to the SOP in the laboratory.

An ambient air sample is introduced into the analytical system from a pressurized canister through stainless steel or Teflon tubing with the aid of a mass flow controller (MFC) and a vacuum system. A digital readout attached to the MFC provides a visual indication of the proper sample flow during sampling. Automated sampling of up to 16 canisters can be accomplished using the system's multi-position stream selector valve.

The sample is trapped on a sorbent trap at 50°C. The sorbent trap consists of carbopack B, carbopack C and carboxen 1000. Moisture in the sample is removed from the sorbent trap by dry purging it with an inert gas. After purging, the

sorbent trap is rapidly heated to 300°C to transfer or desorb the contents onto a cryofocuser which is cooled to -130°. The cryofocuser is rapidly heated to 300°C to inject the sample onto a DB-624 capillary column.

The sample mixture is separated into individual components by their interaction with the capillary column's stationary phase, using temperature programmed gas chromatography. A Mass Selective Detector (MSD) detects the components eluting from the column. The target analytes, as shown in [Table 1](#), page 21, are subsequently identified and quantified. Identification of a component in a sample is based upon both the retention time and mass spectral matching. The response of one mass fragment, the Primary Quantitation Ion, is used for quantitation.

### 3.0 INTERFERENCES AND LIMITATIONS

- 3.1 Although studies have shown that the target compounds can be considered stable in stainless steel canisters, every effort must be made to analyze the sample within 30 days of the sampling date. Extreme care must be taken to prevent contamination during sample collection, transportation and subsequent analysis.
- 3.2 The MSD should be setup and tuned according to the manufacturer's specifications prior to sample analysis. The instrument tuning may be verified with 1-bromo-4-fluorobenzene (BFB). This is not a requirement of this SOP.
- 3.3 Although the retention time of an analyte is not the only parameter used in identifying a component in GC/MS, the retention times of the GC portion of the system must be reproducible.
- 3.4 All target compounds are identified by their mass spectrum and retention times. Compounds having similar GC retention times may co-elute. This can lead to misidentification or inaccurate quantitation. The use of a proper compound specific Primary Quantitation Ion, as well as secondary ions, may allow accurate quantitation and identification even under these circumstances. There is no substitute, however, for good chromatographic separation.
- 3.5 Very low target and non-target analyte concentrations may not produce a good quality spectrum. This may result in either low match quality or misidentification.
- 3.6 No more than 10 samples should be run consecutively without system recalibration. This is an internal OLS/SOP specific requirement, not a Laboratory Quality Control Manual requirement.

- 3.7 The analytical system may be contaminated when samples containing high compound concentrations are analyzed. A blank should be analyzed after a high concentration sample to check for possible carryover.
- 3.8 High boiling compounds being trapped on the column may cause daily baseline shifting, or the appearance of broad, extraneous “ghost” peaks. The column should be baked out prior to each set of analytical runs to remove these contaminants. The bake out temperature should not exceed the column’s maximum operating temperature of 260 °C.

3.8.1 Reference:

“1996/1997 Catalog and Technical Reference”, J & W Scientific, Inc.

- 3.9 The analytical system is capable of detecting compounds other than the target analytes. [Table 1](#), page 21, lists the compounds addressed by this procedure. Benzene and hexane are being used as stability markers.

#### 4.0 APPARATUS

- 4.1 A Lotus Consulting/Varian Model 3800 gas chromatograph, configured as a stand-alone Cryogenic Concentration System, with:

- 4.1.1 An automated sampler, consisting of a multi-position Stream Selector Valve (SSV) and a Mass Flow Controller (MFC) with a Control/Digital Readout module.

4.1.1.1 The MFC is mounted downstream of the SSV, sorbent trap, and cryofocuser to eliminate any contamination and to reduce dead volume in lines from sample trap.

4.1.1.2 The MFC is typically rated at 100 cm<sup>3</sup>/min at 100% full scale. The flow rate is set as a percentage of full scale. For example, a flow rate of 50 cm<sup>3</sup>/min corresponds to a setting of 50% full scale.

4.1.1.3 The Control/Digital Readout module is set to the side or on top of the GC.

4.1.1.4 A rotometer is mounted on the GC, between the MFC and the vacuum source, to allow visual confirmation of flow.

- 4.1.1.5 Reference:

"Stream Selector Valve Control Software For Varian Star Workstation Operator's Manual", by Randall Bramston-

## Cook of Lotus Consulting

- 4.1.2 A sorbent/cryogenic Concentrator system, containing:
  - 4.1.2.1 A 700  $\mu$ l,  $1/8$ -inch nickel tubing is packed with 60/80-mesh of carbopack B, carbopack C and carboxen 1000.
  - 4.1.2.2 A 100  $\mu$ l,  $1/16$  inch cryofocuser constructed of 0.04 inch internal diameter (i.d.) nickel tubing, without packing.
- 4.1.3 One Electronic Flow Controller (EFC) for automatic control of the cryofocuser/column carrier He flow.
- 4.1.4 Two manual, digital flow controllers, and two manual pressure regulators for setting He and N<sub>2</sub> purge/sweep flows. Three analog pressure gauges for use in gas monitoring and diagnosing problems with the flow system.
  - 4.1.4.1 The digital flow controllers are calibrated to deliver gas flows from zero to 100 cm<sup>3</sup>/min,  $\pm$  3%, with an inlet pressure of 80 psi.
- 4.1.5 A canister sampling manifold for connecting canisters to the automated sampler, using appropriate tubing and fittings.
  - 4.1.5.1 Examples of tubing size and material are  $1/8$ -inch teflon tubing,  $1/16$  inch stainless steel tubing,  $1/16$  inch nickel tubing, or  $1/16$  inch glass lined stainless steel tubing.
  - 4.1.5.2 A low-pressure regulator (LPR) with a teflon lined diaphragm.
  - 4.1.5.3 Canisters are connected to the manifold; the manifold is connected to the LPR, and then to the automated sampler's SSV.
- 4.1.6 Information and instruction on the proper operation of the Varian Model 3800 Gas Chromatograph can be found in the associated Varian manuals.
- 4.2 A Hewlett-Packard Model 6890 gas chromatograph, with:
  - 4.2.1 Electronic Pneumatic Controllers (EPC) for control of carrier gas, make-up gas, and detector gases.

- 4.2.1.1 In the current configuration, the Hewlett-Packard carrier gas EPC is not used. Carrier gas control is performed by the Lotus/Varian Cryogenic Pre-Concentrator (Section 4.1.3, page 4).
- 4.2.1.2 The make-up and detector gases EPCs are not used to perform this analysis. They can be used to control optional GC detectors.
- 4.2.2 A Hewlett-Packard Model 5973 Mass Selective Detector (MSD) interfaced to the HP 6890 GC. It is a quadrupole mass spectrometer design, capable of scanning from 33 to 550 amu. It is operated in the electron impact mode at 70 electron volts.
- 4.2.3 Information and instruction on the proper operation of the Hewlett-Packard Model 6890 Gas Chromatograph and the Hewlett-Packard Model 5973 Mass Selective Detector can be found in the associated manuals.
- 4.3 A J&W DB-624 30 m by 0.32 mm i.d., with 1.80  $\mu\text{m}$  film thickness, fused silica capillary column.
  - 4.3.1 Reference:

“1996/1997 Catalog and Technical Reference”, J & W Scientific, Inc.
- 4.4 A Varian GC Star Workstation that includes an Intel compatible PC, an Ethernet network adapter, Microsoft 9.X or NT 4.0 operating system, and Varian Star Chromatography software.
  - 4.4.1 The Workstation is used for GC system configuration, sample file lists, sequence lists, and method building.
  - 4.4.2 The Ethernet network adapter card provides digital communication with the GC.
  - 4.4.3 Reference:

Manuals, on CD-ROM, “Varian Star Chromatography Workstation”, Version 5.5, by Varian, Inc. (P/N 03-910818-01.4)

Manuals, on CD-ROM, “Varian Saturn GC/MS Workstation – System Software”, Version 5.51, by Varian, Inc. (P/N 03-910876-01)

“Varian GC Star Workstation Manual”, by Randall Bramston-Cook of

## Lotus Consulting

- 4.5 A Hewlett-Packard GC/MS ChemStation that includes an Intel compatible PC, an Ethernet network adapter, a GPIB interface card, Microsoft 9.X or NT 4.0 operating system, and Hewlett-Packard Analytical MSD Productivity ChemStation Software.
- 4.5.1 The ChemStation is used for storage of raw data files and the subsequent processing of the raw data to produce qualitative/quantitative data.
- 4.5.2 The Ethernet network adapter card provides digital communication with the GC.
- 4.5.3 The GPIB interface card provides digital data communication with the MSD.
- 4.5.4 Reference:
- Manuals, on CD-ROM, "HP 5973 MSD Reference Collection", Revision C.00.00, by Hewlett-Packard
- 4.6 The Star Chromatography Workstation and the Hewlett-Packard Analytical MSD Productivity ChemStation software can be operated from the same Intel compatible PC.
- 4.7 Stainless steel SUMMA passivated canisters for sample collection and standard preparation.

## 5.0 REAGENTS

- 5.1 A system blank/canister blank, consisting of zero air, ultrapure air, Grade 5 N<sub>2</sub>, or ultrapure N<sub>2</sub>, in a SUMMA canister that has been humidified with 150 µl of HPLC grade water. Alternatively, Ultrapure or Grade 5 N<sub>2</sub>, sampled directly from a gas cylinder, or headspace N<sub>2</sub>, sampled directly from a Liquid Nitrogen (LN<sub>2</sub>) Dewar can be substituted as the system blank.
- 5.2 A certified National Institute of Standards (NIST) standard calibration mixture, or mixtures, containing all analytes of interest is being ordered. This standard, or standards, should be slightly higher in concentration than the typical sample and must be within the dynamic range of the GC/MS system. [Appendix V](#), page 91, lists the concentrations of the current calibration standards associated with this SOP.
- 5.3 A control standard mixture, or mixtures, containing all analytes of interest at concentrations within the calibration range of the GC System. [Appendix V](#),

page 91, lists the concentrations of the Control Standards associated with this SOP.

- 5.4 One high pressure gas cylinder of Grade 5 or better Helium (He) for use as the GC column carrier gas.
- 5.5 One high pressure gas cylinder of Grade 5 or better Nitrogen (N<sub>2</sub>) for use in sample line purging, sorbent trap purging, sample loop purging, and leak testing.
- 5.6 One Liquid Nitrogen (LN<sub>2</sub>) Dewar for cooling the cryofocuser, and the GC column oven.
- 5.7 Perfluorotributylamine (FC43) for use in MS tuning.
- 5.8 A 2 part per million (ppm) solution of 1-bromo-4-fluorobenzene (BFB) for MS tuning verification. This optional procedure is not a requirement of this SOP.

## 6.0 INSTRUMENT CONFIGURATION AND PARAMETERS

- 6.1 Two separate instruments are used to perform this method. A Lotus Consulting/Varian Model 3800 gas chromatograph, configured as a stand-alone Sorbent Concentration System, handles the concentration of the sample, the introduction of the concentrated sample onto the gas chromatographic column, and the column carrier gas flow (Section 4.1, page 3). A Hewlett-Packard Model 6890 gas chromatograph, equipped with a Hewlett-Packard Model 5973 Mass Selective Detector (MSD), controls the column oven temperature, the interface between the detector and the column, and, through software, the acquisition and processing of data (Section 4.2, page 4).
- 6.2 Varian 3800 Concentrator
  - 6.2.1 The Varian 3800 Concentrator's gas flow and automation configurations are shown in Figure 1, page 32, through Figure 8, page 39. The nomenclature and function of the Concentrator's thermal zones are shown in Table 2, page 22. A complete listing of the current Varian Star Workstation method, which includes all of the setpoints controlled by the Workstation, is given in Appendix III, page 53. Each major item in the method is described below.

### 6.2.1.1 Front Valve Oven

This setting controls the isothermal temperature of the in-line Nafion™ sample dryer which is disconnected in this SOP (Section 4.1.6, page 4).

#### 6.2.1.2 Middle Valve Oven

This setting controls the isothermal temperature of the oven in which the SSV (Section 4.1.1, page 3), the Sample Valve (Valve 1), the first Sample Preconcentration Trap (sorbent trap) Valve (Valve 2), and Valve M are installed.

#### 6.2.1.3 Rear Valve Oven

This setting controls the isothermal temperature of the sample lines extending from the Sampling Manifold to the SSV (see 4.1.5, page 4).

#### 6.2.1.4 Valve Table

These settings control the action of the seven (7) time programmable valves/events of the Varian 3800 GC. The valve/relay number, the valve/relay name, the relay state, and the function at each state, are given in **Error! Reference source not found.**, page **Error! Bookmark not defined.**

#### 6.2.1.5 Front Injector Type 1079

This setting controls the programmed temperature of the multisorbent /Front Trap (Section 4.1.2.1, page 4).

#### 6.2.1.6 Middle Injector Type 1079

This setting controls the programmed temperature of the Cryofocuser/Middle Cold Trap (Section 4.1.2.2, page 4).

#### 6.2.1.7 Rear Injector Type 1041

This setting controls the programmed temperature of the oven in which the Sample Preconcentration Trap Valve (Valve 3) and the Series Bypass Valve (Valve 4) are installed. Under normal conditions, this oven is operated isothermally.

This oven is designed to mount on top of the Hewlett-Packard 6890 gas chromatograph. A heated transfer line connects Valve 3, in this oven, to Valve 2 in the Middle Valve Oven (see 6.2.1.2, page 8).

#### 6.2.1.8 Rear Injector EFC Type 3

This setting controls the programmed H<sub>e</sub> capillary column flow rate (Section [4.1.3](#), page 4).

#### 6.2.1.9 Column Oven

This setting controls the programmed temperature of the GC Column oven. In the current configuration, the GC column is not installed in the Varian Concentrator (Section [6.1](#), page 7).

6.2.1.10 Since the Varian 3800 Concentrator is not used for data acquisition, method sections dealing with these functions are not used.

### 6.3 Hewlett-Packard 6890 Gas Chromatograph / 5973 Mass Selective Detector

6.3.1 The Hewlett-Packard 6890/5973 GC/MS System functions normally in this application. The only departure is that the column carrier gas flow is not controlled by this system (Section [4.2.1.1](#), page 5).

6.3.2 A complete listing of the current Hewlett-Packard GC/MS ChemStation method, which includes all of the setpoints controlled by the ChemStation, is given in [Appendix IV](#), page 70. A description of each major item in the method follows.

#### 6.3.2.1 Oven

This setting controls the gas chromatographic column oven temperature. It includes the column temperature program.

#### 6.3.2.2 Front Inlet (HP PTV) and Back Inlet (Split/Splitless)

This setting controls the temperature and gas flows for both of these injectors. Neither is used in this configuration.

#### 6.3.2.3 Column 1 and Column 2

These are text entries describing the GC column.

#### 6.3.2.4 Front/Back Detector, Signal 1/2, and Column Comp 1/2

These settings are used for GC detectors. They are not used in this configuration.

#### 6.3.2.5 Thermal AUX 2

This controls the temperature of the transfer line connecting the GC column to the MSD.

#### 6.3.2.6 7673 Injector

This injector is not used in this configuration.

#### 6.3.2.7 MS Acquisition Parameters

These values control when the filament is turned on, the electron multiplier voltage, the mass range to be scanned, the MSD temperature, and when the filament is turned off.

#### 6.3.2.8 Data Analysis Parameters

These values include reporting and qualitative/quantitative options for the processing of acquired data. The compound information is updated during the processing cycle.

- 6.4 The sample volume for the column injection is automated by the Varian GC Star Workstation software. The function of the valves in the Varian 3800 Concentrator are shown in [Table 3](#), page 23. The setpoint for the MFC is shown in [Appendix I](#), page 51.

## 7.0 DAILY OPERATION

### 7.1 Instrument Performance Check

- 7.1.1 The MSD must be tuned with FC43 to meet the tuning and standard mass spectral abundance criteria prior to initiating any data collection. The detector is tuned using the Autotune program once a week, and is checked on a daily basis using the Quick Autotune program. The procedure and criteria for the FC43 tune can be found in the Hewlett-Packard system manuals referenced on page 70.
- 7.1.2 The tune values, with regard to positions and abundance ratios of the tune m/z's and their corresponding isotope m/z's, are reviewed.

7.1.3 The system leak and electron multiplier voltage are also checked and evaluated.

7.1.4 An example of a tune evaluation report is shown in [Table 5](#), page 29.

#### 7.1.5 BFB Tuning Verification

7.1.5.1 The mass calibration and resolution of the system may be verified by the analysis of the instrument performance check standard, bromofluorobenzene (Section [5.8](#), page [7](#)).

7.1.5.2 This procedure is not a requirement of this SOP. If performed, the mass spectral ion abundance criteria for BFB analysis are shown in [Table 6](#), page 30.

### 7.2 Initial Setup

7.2.1 The Varian 3800 Concentrator method (. mth), sample list (. smp), and sequence list (. seq) are set up on the Star GC Workstation. [Appendix III](#), page 53, has further details, including a listing of the method, and examples of the sample and sequence list screens.

7.2.2 The Hewlett-Packard 6890/5973 data acquisition method (. M) and sequence list (. S) are set up on the Hewlett-Packard GC/MS Chem-Station. [Appendix IV](#), page 70, has further details, including a listing of the method and an example of the sequence list screen.

The sample flow rate setting is confirmed on the MFC's Control/-Digital Readout module. The sample volume is determined as the product of the trapping time, in minutes, times the flow rate, in  $\text{cm}^3/\text{min}$ , set on the MFC. Confirmation of the actual flow rate can be done with an external flow meter. For example:

Trapping Time: 3.0 minutes  
Flow Rate: 50.0  $\text{cm}^3/\text{min}$   
Volume: 3.00 min x 50.0  $\text{cm}^3/\text{min}$  = 150  $\text{cm}^3$

7.2.3 Canister samples are connected to the canister sampling manifold using appropriate tubing and fittings (Section [4.1.5](#), page 4). The sample canister valves are opened and the canister pressure gauge is monitored to assure a leak-free connection. The initial canister pressure is recorded.

### 7.3 Sample Concentration and Analysis

- 7.3.1 Samples are introduced onto the Varian 3800 Concentrator's sorbent trap under control of the Star Chromatography Workstation method. The gas and sample flow and automation configurations for the sorbent trap loading steps are shown [Figure 1](#), page 32, through [Figure 5](#), page 36. The program times, relay # and status, and events are shown in [Table 4](#), page 25.
- 7.3.2 After the Concentrator's multisorbent trap has finished loading, it is dry purged with nitrogen gas, heated and the contents are transferred to the cryofocuser. The cryofocuser loading and subsequent direct transfer of the trapped sample onto the GC column steps are shown in [Figure 1](#), page 32, and [Figure 6](#), page 36, through [Figure 8](#), page 39.
- 7.3.3 A graphical representation of the concentration steps is shown in [Figure 9](#), page 40.

### 7.4 Samples

- 7.4.1 A system blank (defined in [Section 5.1](#), page 6) is analyzed prior to calibration standards, controls and samples.
  - 7.4.1.1 A system blank run must be performed at least once every 24 hours.
  - 7.4.1.2 System blanks should also be run after samples which contains high concentrations (>100 times a target compound's LOD) to detect and eliminate possible carry-over.
  - 7.4.1.3 Trip blanks, if available, are analyzed like samples and their results are documented and evaluated.
- 7.4.2 A daily calibration standard, for each standard mixture in use (defined in [Section 5.2](#), page 6), is analyzed after the system blank, prior to controls or samples.
- 7.4.3 A control standard, for each standard mixture in use (defined in [Section 5.3](#), page 6), is analyzed after the system blank and calibration standards, prior to ambient air samples.
- 7.4.4 Ambient samples are analyzed using the same sample volume as used for the calibration standard and control standard.

- 7.4.4.1 A smaller volume is analyzed for samples containing concentrations of target analytes that exceed the linear range of the analysis.
- 7.4.4.2 Smaller volumes are obtained by reducing the trapping time while keeping the MFC setpoint constant.
- 7.4.5 Duplicate analyses are performed on 10% of all ambient samples analyzed.

## 8.0 DATA ANALYSIS

- 8.1 After data acquisition, the raw data files (data.ms) collected on the Hewlett-Packard GC/MS ChemStation are processed by the software to produce result files (MLD066.res). The result files contain the integrated Primary Quantitation Ion peak areas, retention times, and mass spectra.
- 8.2 Chromatographic peaks found in the Total Ion Chromatogram (TIC) in the result files for calibration standards are qualitatively identified based on matching the mass spectrum to a reference spectra and the retention time to the reference retention time. Both of these references are stored in the method.
- 8.3 After analyte identification, the integrated calibration standard areas for the Primary Quantitation Ions are used to calibrate the ChemStation method for both retention time and concentration. The latter is based on the peak areas and the known analyte concentration in the standards.
- 8.4 After calibration of the method, chromatographic peaks from the TIC in blank, control, and ambient sample result files are qualitatively identified based on matching the mass spectrum to a reference spectra and the retention time to the reference retention time. They are quantified using the Primary Quantitation Ion response factor stored in the method.
- 8.5 A typical Calibration Standard TIC, Ambient Air TIC, and Mass Spectrum are shown in [Figure 10](#), page 41, through [Figure 12](#), page 42.

## 9.0 QUALITY CONTROL

- 9.1 System Blank
  - 9.1.1 A system blank is analyzed before any standard or sample is run to evaluate the system cleanliness.

- 9.1.2 If the individual concentrations of any target analytes detected in the system blank are less than two (2) times their LOD, no action is taken.
- 9.1.3 If the concentration of any target analyte detected in the system blank is greater than two (2) times it's LOD, the analytical run associated with the system blank should be invalidated and the cause investigated.
- 9.1.4 All actions taken in response to system blank results should be approved by the OLS Supervisor.
- 9.1.5 The actions taken in response to system blank results may be modified by the most current version of the Laboratory Quality Control Manual in effect.

## 9.2 Daily Calibration

- 9.2.1 A single point calibration is performed daily by analyzing the calibration standard, or standards.
- 9.2.2 Retention times, spectra and the Primary Quantitation Ion integration for each target analyte in the calibration standard run should be thoroughly checked prior to calibration.
  - 9.2.2.1 The retention times should fall within  $\pm 0.1$  minute of the preceding runs retention times. This difference may be modified if historical data indicates a larger difference is more appropriate (i.e., volatile early eluting compounds, or wider, later eluting compounds).
  - 9.2.2.2 The Primary Quantitation ion response factors should fall within  $\pm 20\%$  of the preceding runs response factors.
  - 9.2.2.3 If either retention times or the response factors are outside these ranges, the analyst must investigate the cause.
- 9.2.3 The ChemStation method is updated after every run with the new calibration information.
  - 9.2.3.1 The method and response factors can be printed for a hardcopy record.

- 9.2.3.2 Some typical single point calibration concentrations and instrument responses can be found in the Hewlett-Packard GC/MS ChemStation method listing in [Appendix IV](#), under compound information, page 70.

### 9.3 Control Standard

- 9.3.1 In order to evaluate the accuracy of the calibration and the overall performance of the system, a control standard is analyzed daily following the system blank and the calibration standard and prior to sample analysis.
- 9.3.2 Analysis results of the target analytes in this standard are recorded and used to generate control charts.
  - 9.3.2.1 At least 20 data points are needed for the initial set of control limits, and any subsequent adjustment of these limits. This is a requirement for this SOP.
  - 9.3.2.2 Typical Control Charts for the five target analytes are shown in Figure 18, page 49, through [Figure 22](#), page 50.
  - 9.3.2.3 A typical dataset used for calculating control limits is given in [Table 7](#), page 31.
- 9.3.3 The control standard results must be within the established Control Limits for sample analyses to be valid. Control standard results are evaluated as follows.
  - 9.3.3.1 Should any analysis of the control standard yield a result that falls outside the established Control Limits, the control standard shall be reanalyzed.
  - 9.3.3.2 If the second result is also outside the Control Limits, the analysis shall be discontinued and the problem investigated.
  - 9.3.3.3 All data generated during the out of control period shall be invalidated, and the samples reanalyzed after the analysis has been reestablished.
  - 9.3.3.4 If reanalysis is not possible, results may be invalidated on a compound by compound basis.

- 9.3.4 All actions taken in response to control standard analysis results should be approved by the OLS Supervisor.
- 9.3.5 The actions taken in response to control standard results may be modified by the most current version of the Laboratory Quality Control Manual in effect.

#### 9.4 Method Precision

- 9.4.1 Sample precision is measured by the analysis of ambient duplicate samples and the analysis of ambient collocated samples.
- 9.4.2 The percent difference (PD) of the duplicate analyses, for samples with target analyte concentrations greater than five (5) times the Limit of Detection (LOD), are recorded and included in the method quality control report.
  - 9.4.2.1 The control limits for the PD of the duplicate sample analyses are the same as the control limits for the Control Standard. The duplicate criteria limit is three (3) times the percent relative standard deviation.
  - 9.4.2.2 For this analysis, if the duplicate results do not meet the quality control criteria, the samples associated with the duplicate pair should be reanalyzed, or invalidated if reanalysis is not possible.
- 9.4.3 The PD for collocated sample analyses is used to evaluate method precision for both sampling and analysis procedures.
  - 9.4.3.1 The PD for collocated sample analyses should be within  $\pm 25\%$ .
  - 9.4.3.2 Collocated sample results that do not meet the criteria are reported to the Air Quality Surveillance Branch for action.
  - 9.4.3.3 Results for collocated samples that do not meet the criteria are not invalidated by the Laboratory.
- 9.4.4 All actions taken in response to duplicate sample results should be approved by the OLS Supervisor.
- 9.4.5 The actions taken in response to duplicate sample results may be modified by the most current version of the Laboratory Quality Control Manual in effect.

## 9.5 Multipoint Analysis Verification

- 9.5.1 A multipoint verification must be performed every year, as dictated in the most current version of the Laboratory Quality Control Manual, to verify the precision and the calibration working range.
- 9.5.1.1 A multipoint verification is also required, as dictated in the most current version of the Laboratory Quality Control Manual, whenever a system change occurs that is defined by the analyst as major (i.e., a change in instrument or measurement technique that would likely change the method LOD, linearity, or measured concentrations).
- 9.5.1.2 This is done by analyzing at least three (3) concentration levels of the NIST standard, using at least three (3) replicates at each level.
- 9.5.1.3 One of the multipoint verification points must be at the same concentration level as the daily calibration standard level.
- 9.5.1.4 One of the points should be near the LOD concentration of the target analytes.
- 9.5.1.5 The highest concentration point determines the upper limit of the analytical concentration range.
- 9.5.2 In order to verify that the system is linear:
- 9.5.2.1 The plot of response vs. concentration must appear linear;  
and
- 9.5.2.2 The correlation coefficient,  $r$ , calculated from a least square fit of the response/concentration data must be 0.98 or greater. This corresponds to a coefficient of determination,  $r^2$ , of 0.96 or greater.
- 9.5.3 Typical multipoint data and graphs for target analytes are presented in Figure 13, page 44 through Figure 17, page 48. Correlation coefficient and highest calibrated concentration values for each target analyte are shown in [Appendix II](#), page 52.

- 9.5.4 If the verification is considered substantially different from an initial or immediately preceding check, by either the analyst or the OLS Supervisor, the analytical system should be evaluated for problems and the procedure repeated.
- 9.5.5 All actions taken in response to the multipoint verification should be approved by the OLS Supervisor.
- 9.5.6 The actions taken in response to the multipoint verification may be modified by the most current version of the Laboratory Quality Control Manual in effect.

9.6 Limit of Detection (LOD) Verification

- 9.6.1 The LOD verification must be performed every year, as dictated in the most current version of the Laboratory Quality Control Manual,
  - 9.6.1.1 It must also be verified when the conditions as listed under multipoint calibration verification occur (Section 9.5.1.1, page 17).
  - 9.6.1.2 This is done by analyzing at least seven (7) replicates of the NIST standard.
  - 9.6.1.3 The concentration must be no more than five (5) times the published LOD.
  - 9.6.1.4 The calculated LODs must be equal to or less than the published LOD values.
- 9.6.2 The LOD is calculated using the following equation, as specified in most current version of the Laboratory Quality Control Manual in use.

$$\mathbf{MDL} = \mathbf{T}_{(n - 1, 1 - \alpha = 0.99)} \times \mathbf{s}$$

where

- n** = the number of replicates
- T** = the Students' t-value at the 99% confidence level (1 -  $\alpha$ ) for n - 1 degrees of freedom
- s** = the Standard Deviation of the sample Mean

- 9.6.3 The published LODs for most target analytes analyzed by this method and example verification values are presented in [Appendix II](#), page 52.
- 9.6.4 If the verification is considered substantially different from an initial or immediately preceding check, by either the analyst or the OLS Supervisor, the analytical system should be evaluated for problems and the procedure repeated.
- 9.6.5 All actions taken in response to the LOD verification should be approved by the OLS Supervisor.
- 9.6.6 The actions taken in response to the LOD verification may be modified by the most current version of the Laboratory Quality Control Manual in effect.

## 9.7 Method Accuracy

- 9.7.1 Providing performance audits to the NLB, in order to assess the accuracy of the generated data, is the responsibility of the Quality Assurance Section (QAS) of the Quality Management Branch (QMB).
  - 9.7.1.1 The analysis of performance audit materials shall follow the same procedures as the analysis of regular samples, where possible.
  - 9.7.1.2 Several replicate analyses of the performance audit material should be performed to provide an estimate of precision (i.e., the sample standard deviation).
  - 9.7.1.3 The concentration results of audit sample analyses, including the sample standard deviation and the number of replicate analyses, shall be provided as quickly as possible to the QAS staff, and shall be included in the quarterly QC reports.
  - 9.7.1.4 If after receiving the QAS Audit Report any results are considered substantially different from the preceding audit results, the OLS Supervisor in conjunction with the QAS Supervisor shall formulate an appropriate course of action.
  - 9.7.1.5 All actions taken in response to the performance audit should be approved by the OLS Supervisor.

- 9.7.1.6 The actions taken in response to the performance audit may be modified by the most current version of the Laboratory Quality Control Manual in effect.
- 9.7.2 Providing blind Through the Probe audit samples to the NLB, in order to assess the accuracy of the entire sampling and analysis system, is the responsibility of the Quality Assurance Section (QAS) of the Quality Management Branch (QMB).
  - 9.7.2.1 Through the Probe audit samples shall be treated as regular ambient air samples.
  - 9.7.2.2 Replicate analyses of Through the Probe audit samples, unless the sample is picked as the analytical duplicate, should not be performed.
  - 9.7.2.3 The concentration results of Through the Probe audit sample analysis shall be provided as quickly as possible to the QAS staff, and shall be included in the quarterly QC reports.
  - 9.7.2.4 If after receiving the QAS Through the Probe Audit Report any results are considered substantially different from the preceding audit results, the OLS Supervisor in conjunction with the QAS Supervisor shall formulate an appropriate course of action.
  - 9.7.2.5 All actions taken in response to Through the Probe audit should be approved by the OLS Supervisor.
  - 9.7.2.6 The actions taken in response to the Through the Probe may be modified by the most current version of the Laboratory Quality Control Manual in effect.
- 9.7.3 The analysis of any audit samples provided by other sources should be performed as directed by the OLS Supervisor.
- 9.7.4 Method accuracy may also be assessed by periodically analyzing other standard reference materials (i.e., other NIST Standards). The results of replicate analysis of these materials should be consistent with the estimated uncertainty of the sample, the standard, and the analytical replicates.

**Table 1: Target Compounds and Characteristic Masses (m/z) for Quantification**

Compound Name	Compound		Chemical Formula	CAS No.	Primary Ion	Secondary Ion(s)
	Type	Abbr. <sup>(1)</sup>				
Acrolein	T		C3H4O	000107-02-8	56	55,44
Ethanol	T	EtOH	C2H6O	000115-10-6	45	46,43
Acetone	T		C3H6O	000067-64-1	43	58,42
Acetonitrile	T	ACN	C2H3N	000075-05-8	41	40,39
Acrylonitrile	T	AcryN	C3H3N	0001071-13-1	53	52,51
Benzene			C6H6	71-43-2	78	77
Hexane			C6H14	000110-54-3	57	56,86

<sup>(1)</sup> Abbr. = Abbreviation – sometimes used in lieu of the full name in the analytical software.  
T = Target compound

**Table 2: Thermal Zones for the Varian 3800 Concentrator**

<b>Thermal Zone #</b>	<b>Status Label</b>	<b>GC Control Label</b>	<b>Function</b>
1	Front: 1079	Front 1079	Multisorbent trap Temperature (Front Cold Trap)
2	Middle: 1079	Middle 1079	Cryofocuser Temperature (Middle Cold Trap)
3	Rear Valve Oven	Large Valve Oven	Sampling Manifold to SSV Line Heater Temperature
4	Front Valve Oven	Small Valve Oven	Nafion Dryer Heater Temperature (not used in this SOP)
5	Middle Valve Oven	Large Valve Oven	SSV, Valve 1, Valve 2, and Valve M Heated Valve Oven Temperature
6	Rear: 1041	Rear 1041	Valve 3 and Valve 4 Heated Valve Oven Temperature

**Table 3: Function of Valves for the Varian 3800 Concentrator**

<b>Valve/ Relay #</b>	<b>Name</b>	<b>Relay Event and Description</b>	<b>Function</b>
1	Sample Valve	- Off	Sample Flow Blocked Internal Standard Inlet to Vent Purge N2 Flow through Loop to Valve 2 – Purge Lines or Transfer Internal Standard from Loop to Valve 2
		+ On	Sample Flow to Valve 2 Internal Standard Flow through Loop to Vent Purge N2 Flow to Vent
2	Sample Preconcentration Trap Valve	- SPT Desorb	Flow from Valve 1 to Vacuum Purge He Flow through Multisorbent trap to Valve 3
		+ SPT Trap	Flow from Valve 1 through Multisorbent trap to Vacuum Purge He Flow to Valve 3
3	Sample Preconcentration Trap Valve	- SPT Desorb	Flow from Valve 2 to Valve 4 Column Carrier He Flow to Column
		+ SPT Trap	Flow from Valve 2 to Vent Column Carrier He Flow to Valve 4 then Column
4	Series Bypass Valve	- Series	Cryofocuser in Series with Flow from Valve 3
		+ Bypass	Cryofocuser Isolated
5	Event A Valve	- Off	No Action

**Table 3: Function of Valves for the Varian 3800 Concentrator**

<b>Table 3: Function of Valves for the Varian 3800 Concentrator</b>			
		+ On	Start Hewlett-Packard GC and MS Data Acquisition
6	Event B Valve	- Off	Sample Line to Vent
		+ On	Enable Leak test
7	Event C Valve	- Off	N2 Pressurization Gas Off
		+ On	N2 Pressurization Gas On

**Table 4: Program Times, Relay #'s, and Status for the Concentrator**

Time (minutes)	Relay # & Status	Events
0.00	-1-2-3-4-5-6-7	<p>All Valves are off (-):</p> <p>The sample flow is blocked and N<sub>2</sub> purge gas flows through the loop to Valve 2 and then through the MFC to vacuum.</p> <p>He purge gas flows through Valve 2, through the Multisorbent trap (Front Cold Trap), through Valve 3, through the cryofocuser (Middle Cold Trap), back through Valve 3 to vent.</p> <p>He carrier gas flows through Valve 3 to the column.</p>
0.01	+1-2-3-4-5-6-7	<p>Valve 1 is turned on (+1):</p> <p>This allows the sample to flow through Valve 1 then through the MFC to vacuum, purging the lines with new sample. The N<sub>2</sub> purge gas flow is blocked.</p> <p>He purge gas flows through Valve 2, through the Multisorbent trap, through Valve 3, through the cryofocuser, back through Valve 3 to vent.</p> <p>He carrier gas flows through Valve 3 to the column.</p>
2.00	+1+2-3-4-5-6-7	<p>Valve 2 is turned on (+2) and Valve 1 remains on (+1):</p> <p>This allows the sample to flow through Valve 1, through Valve 2, through the multisorbent trap and then through the MFC to vacuum. The N<sub>2</sub> purge gas flow remains blocked. <b><i>This starts sample loading of the multisorbent trap.</i></b></p> <p>He purge gas flows through Valve 2, through Valve 3, through the cryofocuser, back through Valve 3 to vent.</p> <p>He carrier gas flows through Valve 3 to the column.</p>

**Table 4: Program Times, Relay #'s, and Status for the Concentrator**

Time (minutes)	Relay # & Status	Events
3.00	+1+2-3-4+5-6-7	<p>Sample loading of the multisorbent trap continues. Valve 5 is turned on (+5) and starts the HP GC/MS data acquisition..</p> <p>He purge gas flows through Valve 2, through Valve 3, through the cryofocuser, back through Valve 3 to vent.</p> <p>He carrier gas flows through Valve 3 to the column.</p> <p><b>Note: The sample volume is varied by controlling the actions of Valve 1.</b></p>
4.00	+1+2-3-4-5-6-7	<p>Same as at 3.0 min. except that Valve 5 is turned off(-5). HP GC/MS data acquisition continues.</p> <p>He carrier gas flows through Valve 3 to the column.</p>
5.00	-1-2-3-4-5-6-7	<p>All Valves but Valve 2 are off. <b>This terminates sample loading of the multisorbent trap.</b> The sample flow is blocked and N2 purge gas flows through the loop to Valve 2, through the multisorbent trap, then through the MFC to vacuum. This purges the multisorbent trap of possible contaminants and moisture for six minutes.</p> <p>He purge gas flows through Valve 2 to Valve 3 then to Valve 4, through the cryofocuser, back to Valve 3 and to vent.</p> <p>He carrier gas flows through Valve 3 to the column.</p>

**Table 4: Program Times, Relay #'s, and Status for the Concentrator**

Time (minutes)	Relay # & Status	Events
11.00	-1-2-3-4-5-6-7	<p>All Valves are off. Stops N2 purging of multisorbent trap. He purge gas flows through Valve 2, through the multisorbent trap, through Valve 3, to Valve 4, through the cryofocuser and to Vent.</p> <p>He carrier gas flows through Valve 3 and to the column.</p> <p><b><i>This starts transfer and loading of sorbent trap contents to the cryofocuser.</i></b></p>
14.00	-1-2-3+4-5-6-7	<p>Valve 4 is turned on(+). He purge gas flows through Valve 2, through the multisorbent trap, through Valve 3 to Valve 4 and to Vent.</p> <p>He carrier gas flows through Valve 3 to column.</p> <p><b><i>Transfer of analytes to cryofocuser stops, and cryofocuser is isolated.</i></b></p>
14.90	-1-2+3+4-5-6-7	<p>Valve 3 is turned on(+) and Valve 4 remains on. All other valves are off. He purge gas flows through Valve 2, through the multisorbent trap (Front Cold Trap), through Valve 3, and to Vent.</p> <p>He carrier gas flows through Valve 3 to Valve 4 and back to Valve 3 to Column.</p>
15.0	-1-2+3-4-5-6-7	<p>Valve 3 remains on (+3) and Valve 4 is off (-4). All other Valves are off. He purga gas flows through Valve 2, through the multisorbent trap, to Valve 3 and to Vent. He carrier gas flows through Valve 3 to Valve 4, through the cryofocuser, back to Valve 3 and to Column.</p> <p><b><i>This backflushes the contents of the cryofocuser to GC column.</i></b></p>

**Table 4: Program Times, Relay #'s, and Status for the Concentrator**

Time (minutes)	Relay # & Status	Events
20.0	-1-2-3-4-5-6-7	<p>All Valves are off. Sample flow is blocked, N2 purge gas flows through sample loop through MFC to Vacuum.</p> <p>He purge gas flushes the multisorbent trap and cryofocuser to Vent.</p>

### Table 5: Autotune Evaluation Report

Instrument Name:	GC/MS Instrument #3 (HP6890/HP5973)	
DC Polarity:	Positive	
Filament:	1	
Basepeak should be 69 or 219		OK
Position of mass 69	69.00	OK
Position of mass 219	219.00	OK
Position of isotope mass 70	70.00	OK
Position of isotope mass 220	219.99	OK
Position of isotope mass 503	502.91	OK
Ratio of mass 70 to mass 69 (0.5 – 1.6%)	1.11	OK
Ratio of mass 220 to mass 219 (3.2 – 5.4%)	4.30	OK
Ratio of mass 503 to mass 502 (7.9 – 12.3%)	9.98	OK
Ratio of 219 to 69 should be >40% and is	66.88	OK
Ratio of 502 to 69 should be >2.4% and is	5.69	OK
Mass 69 Precursor (<= 3%)	0.08	OK
Mass 219 Precursor (<= 6%)	0.33	OK
Mass 502 Precursor (<= 12%)	3.32	OK
Testing for a leak in the system		
Ratio of 18 to 69 (<20%)	2.12	OK
Ratio of 28 to 69 (<10%)	2.67	OK
Electron Multiplier Voltage	1341	OK
Tune portion of system verification passed		

**Table 6: BFB Ion Abundance Criteria**

<b>m/z</b>	<b>Ion Abundance Criteria</b>
50	8.0 to 4.0 Per cent of m/z 95
75	30.0 to 66.0 Percent of m/z 95
95	Base peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/z 95 (see note)
173	Less than 2.0 Percent of m/z 174
174	50.0 to 120 Percent of m/z 95
175	4.0 to 9.0 Percent of m/z 174
176	93.0 to 101.0 Percent of m/z 174
177	5.0 to 9.0 Percent of m/z 176

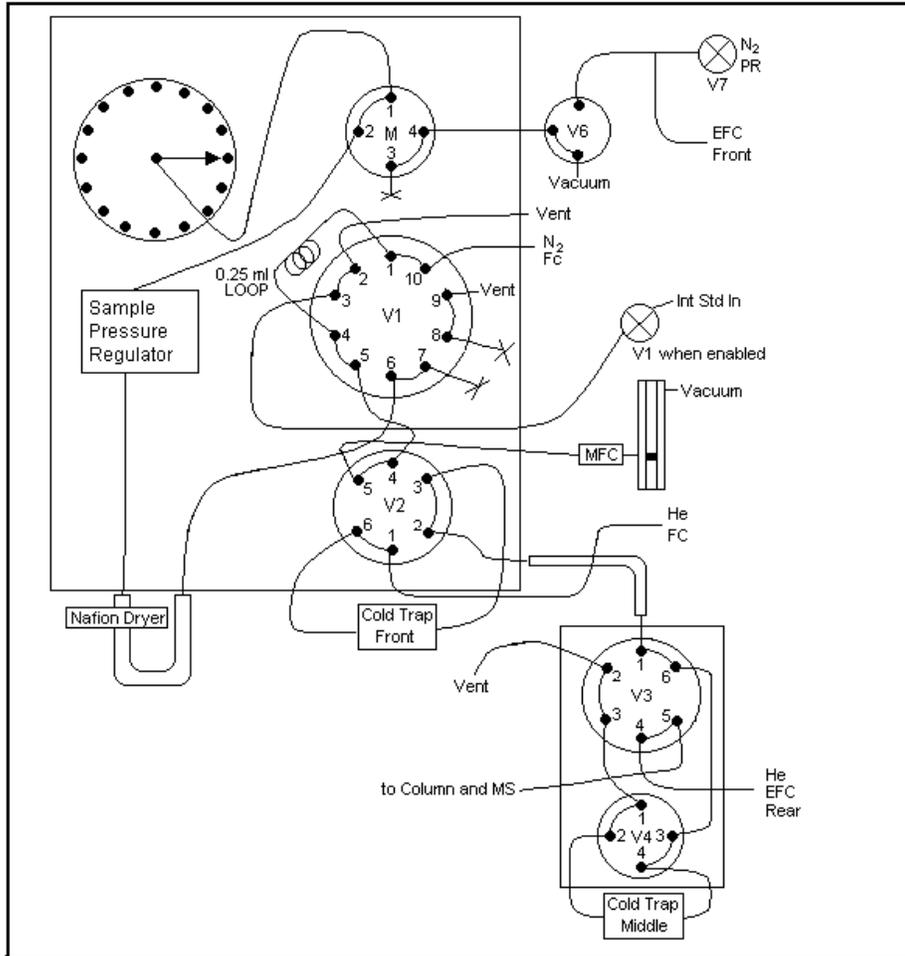
All ion abundances must be normalized to m/z 95, the nominal base peak, even if the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

**Table 7: Precision Measurements and Control Limits for MLD066  
ALM069798**

<b>Compound</b>	<b>Acrolein</b>	<b>Ethanol</b>	<b>Acetone</b>	<b>Acetonitrile</b>	<b>Acrylonitrile</b>
<b>File Name</b>	<b>ppb</b>	<b>ppb</b>	<b>ppb</b>	<b>ppb</b>	<b>ppb</b>
SE1911.D	3.08	20.81	19.14	8.54	4.37
SE1912.D	3.14	21.02	18.91	8.51	4.25
SE1913.D	3.11	19.20	18.76	8.40	4.15
SE1914.D	2.99	19.33	19.18	8.46	4.20
SE1915.D	3.04	18.39	18.81	8.43	4.16
SE1917.D	3.08	17.37	19.85	7.90	3.95
SE1918.D	2.96	17.06	19.85	7.86	3.94
SE1919.D	2.94	16.66	19.89	7.92	4.03
SE1920.D	3.03	16.94	19.85	7.89	4.04
SE1921.D	2.91	18.29	19.93	7.90	3.93
SE1922.D	3.04	18.72	19.48	8.32	4.08
SE1923.D	3.07	20.82	19.58	8.11	4.15
SE1924.D	3.06	20.83	19.60	8.40	4.16
SE1925.D	2.94	20.29	20.76	8.31	4.11
SE1926.D	2.96	18.82	19.73	8.12	3.88
SE2210.D	3.12	18.98	19.69	8.50	4.19
SE2211.D	3.00	20.14	19.28	8.51	4.43
SE2223.D	2.92	19.41	19.01	8.27	4.17
SE2224.D	3.05	20.01	18.73	8.38	4.07
SE2225.D	2.77	19.42	18.80	8.26	4.16
Ave.	3.01	19.13	19.44	8.25	4.12
Std. Dev.	0.089	1.363	0.527	0.241	0.140
%RSD	2.95	7.12	2.71	2.93	3.40
UCL	3.28	23.21	21.02	8.97	4.54
UWL	3.19	21.85	20.50	8.73	4.40
LWL	2.83	16.40	18.39	7.77	3.84
LCL	2.74	15.04	17.86	7.53	3.70

**Figure 1: All Valves OFF**

**Time: Idle state at 0.00 min, Transfer of Trap Contents to Cryofocuser at 11.00 min, and flushes sampling system at 20.00 min**

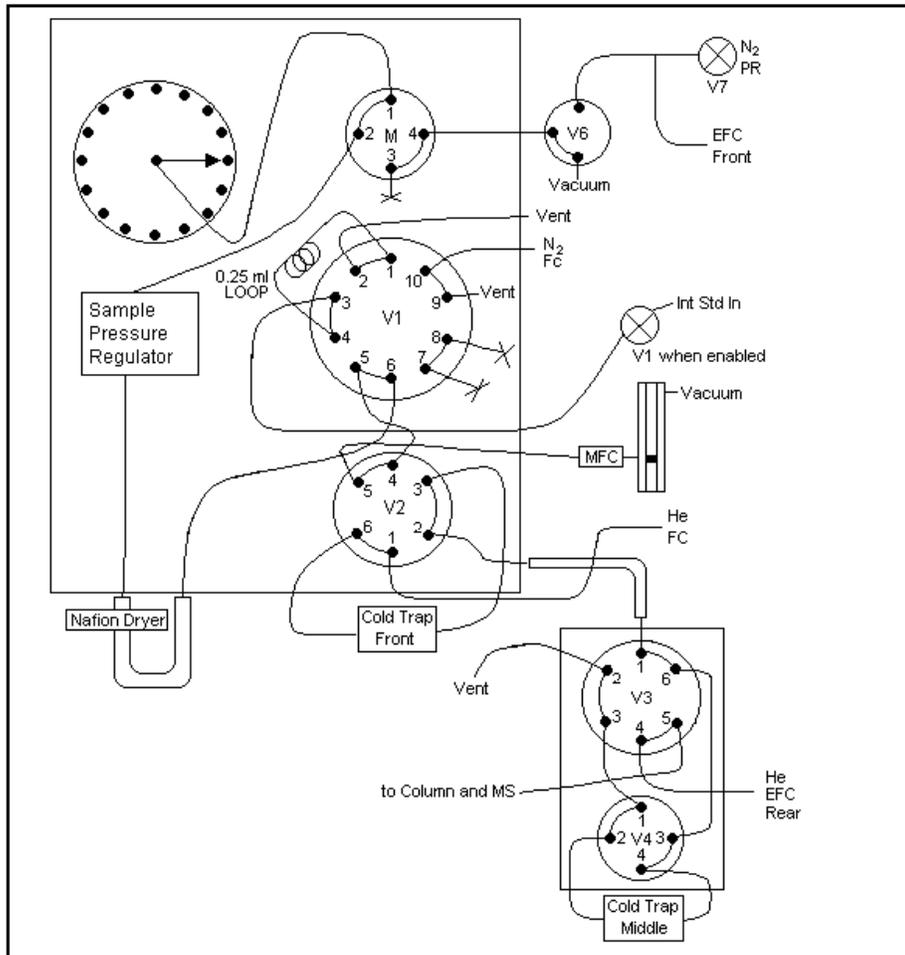


**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
* 0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	-	+	-	-	-	-	-
* 11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	-	-	-	-
* 20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 2: Purges Sample Line  
Time: 0.01**

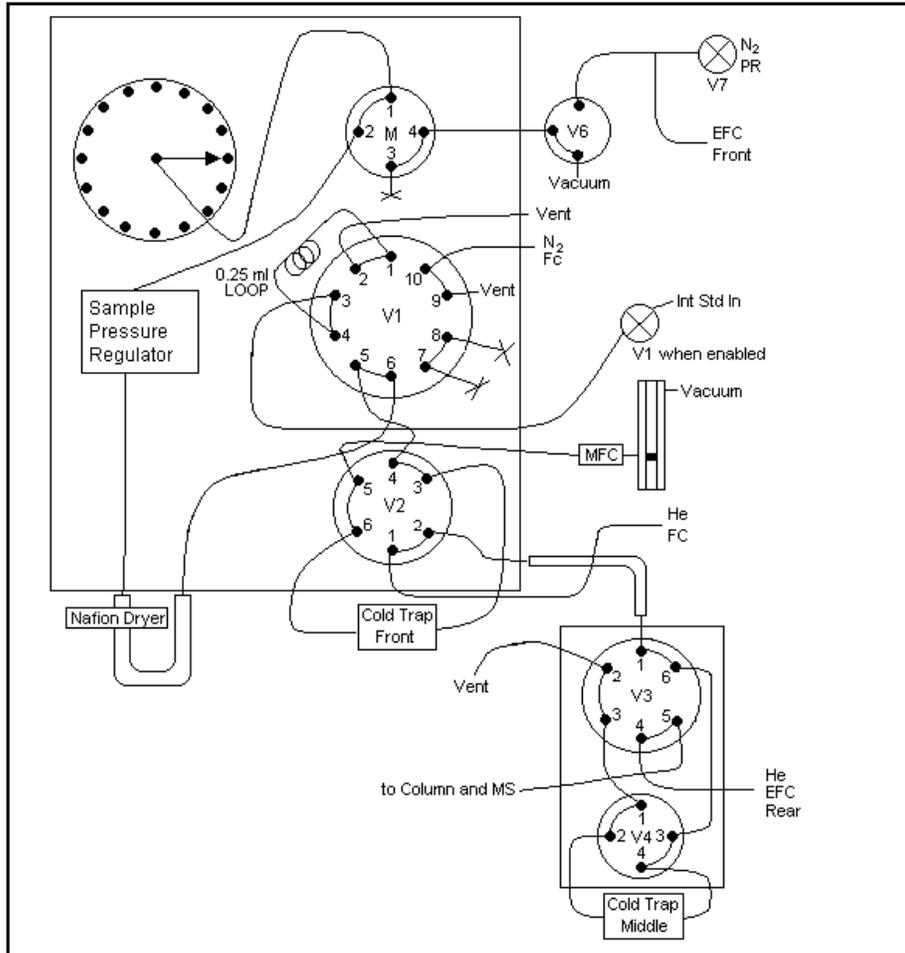


**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	-	+	-	-	-	-	-
11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	+	-	-	-
20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 3: Starts Loading Multisorbent Trap**  
**Time: 2.00**

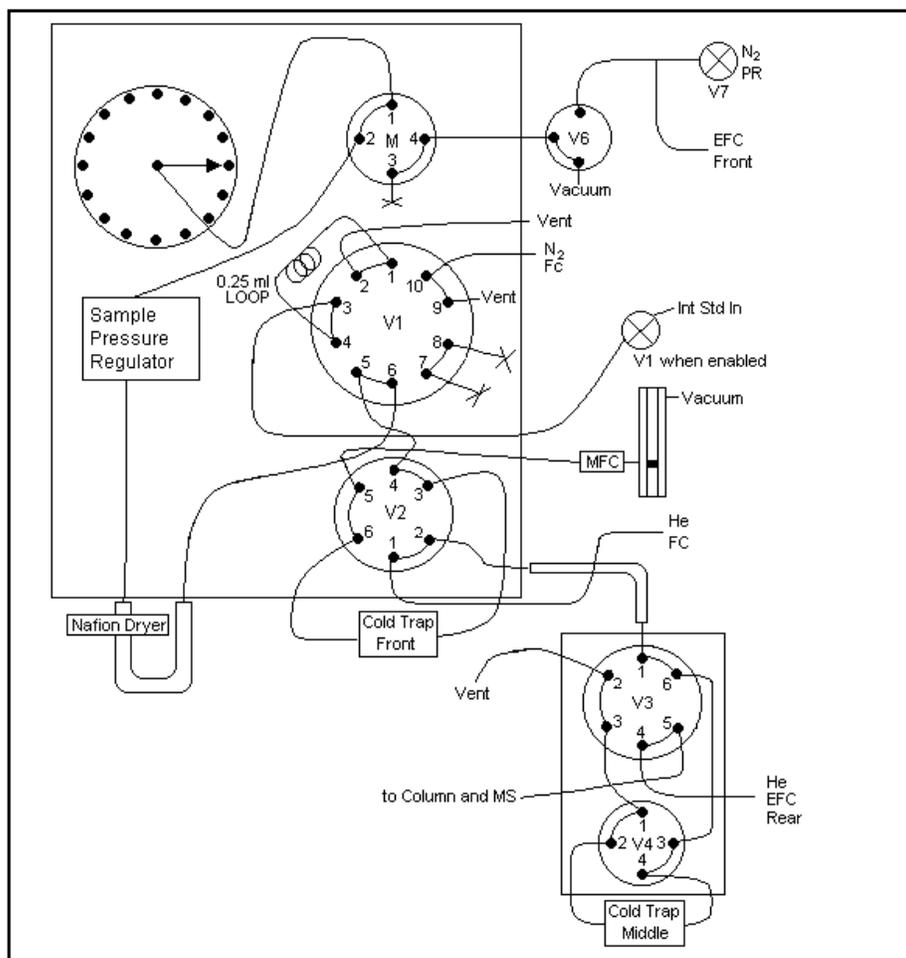


**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	+	+	-	-	-	-	-
11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	-	-	-	-
20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 4: Loading Multisorbent Trap and starts GC/MS Data Acquisition  
Time: 3.00 min**

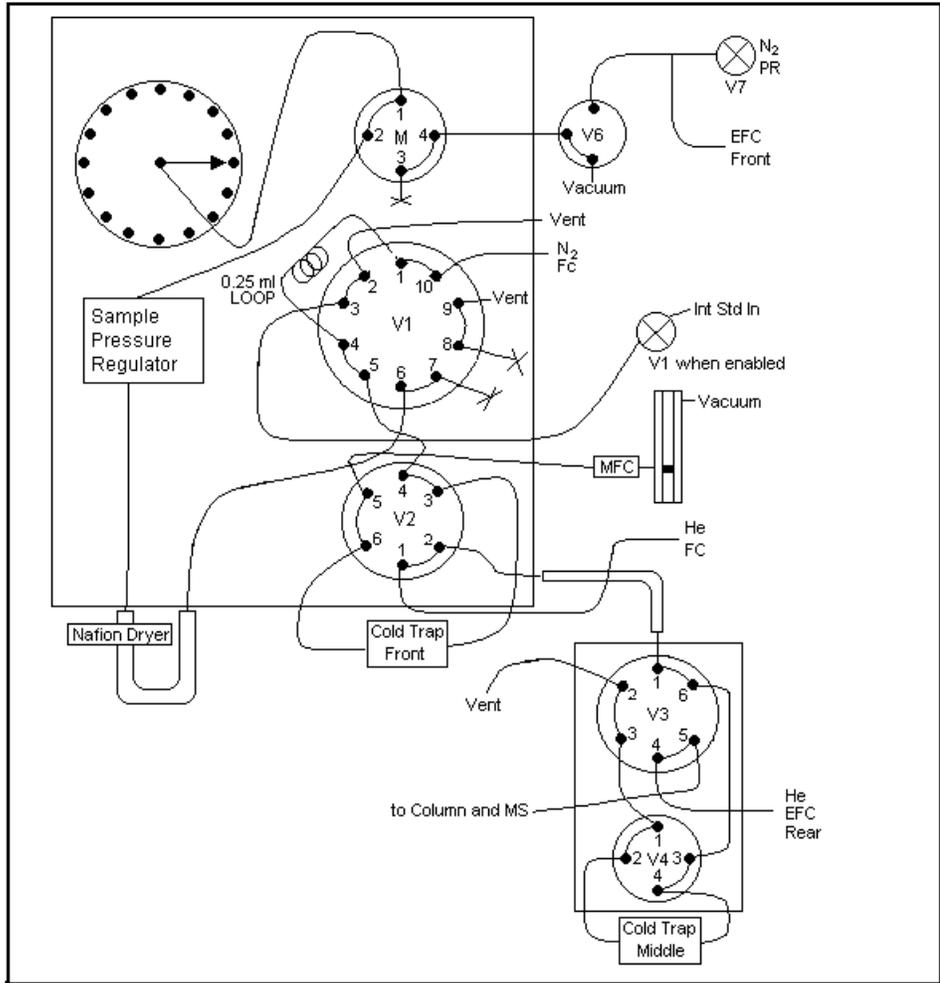


**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	-	+	-	-	-	-	-
11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	-	-	-	-
20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 5: Stops loading and starts N2 Purge of Sorbent Trap  
Time: 5.00 min**

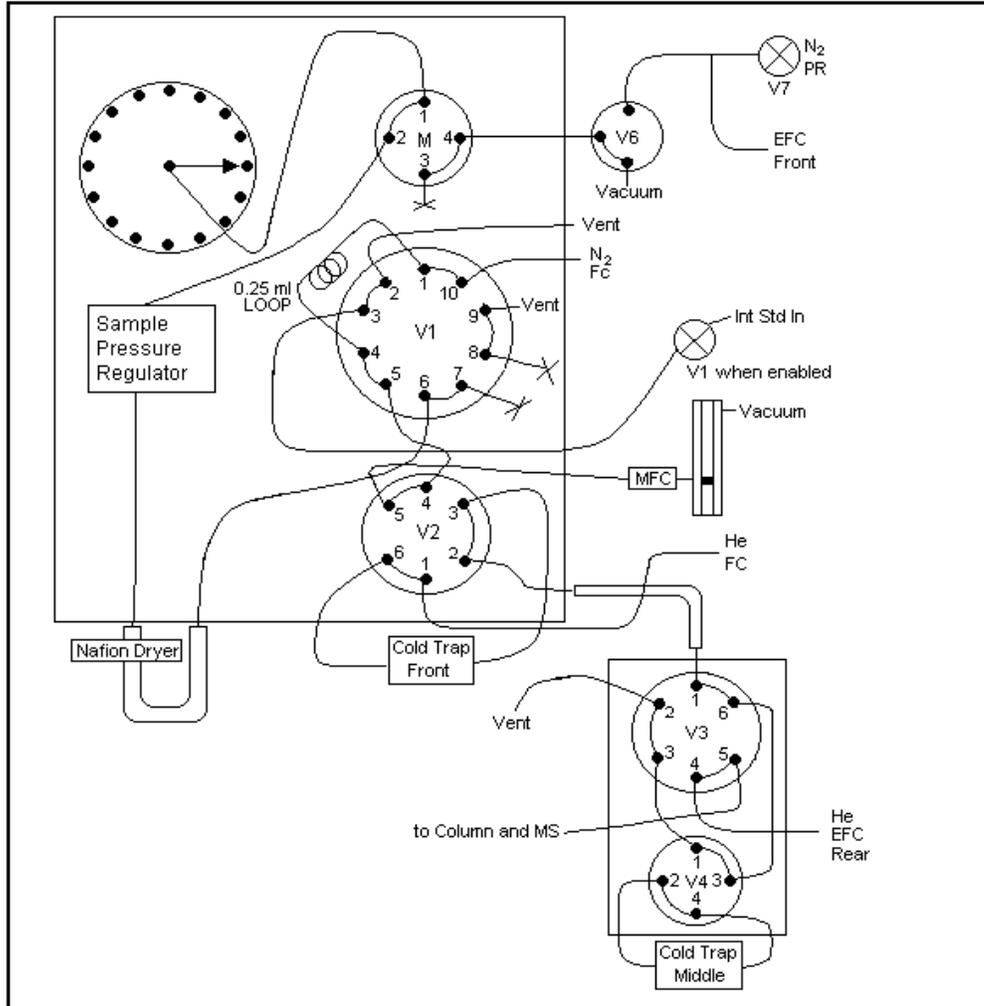


**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	-	+	-	-	-	-	-
11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	-	-	-	-
20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 6: Stops Transfer of Trap contents to Cryofocuser**  
**Time: 14.00 min**

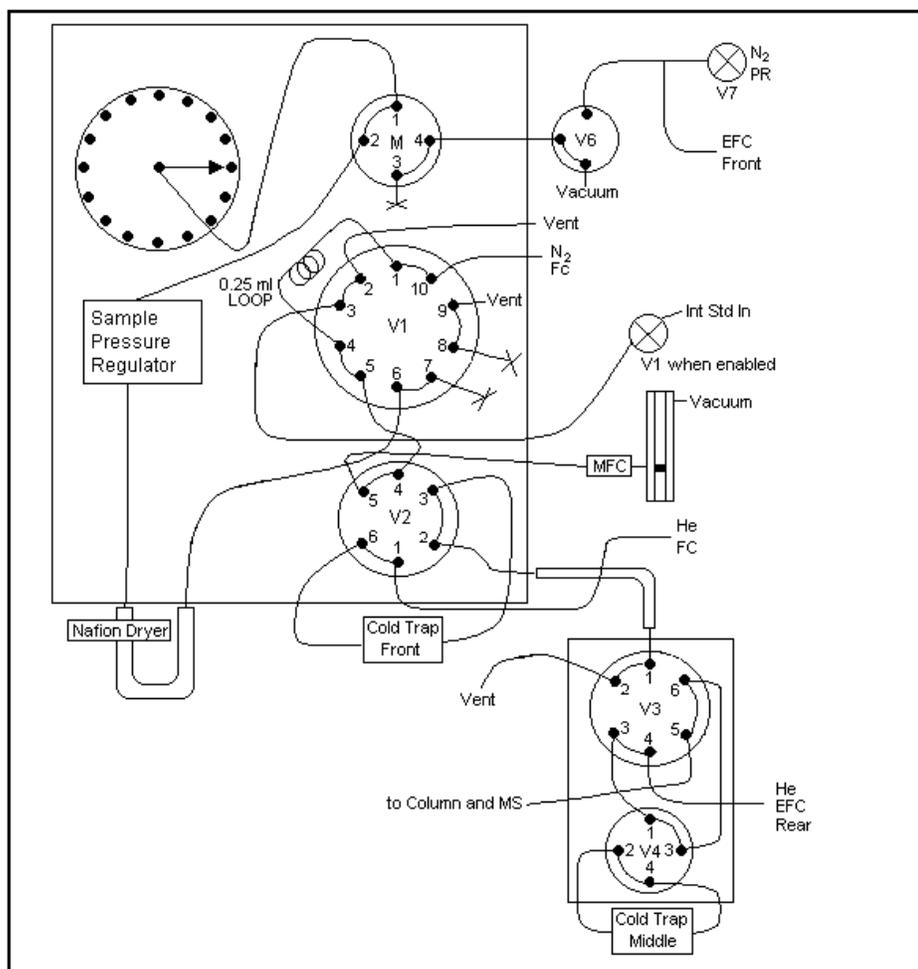


**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	-	+	-	-	-	-	-
11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	-	-	-	-
20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 7: Isolates Cryofocuser**  
**Time: 14.90 min**

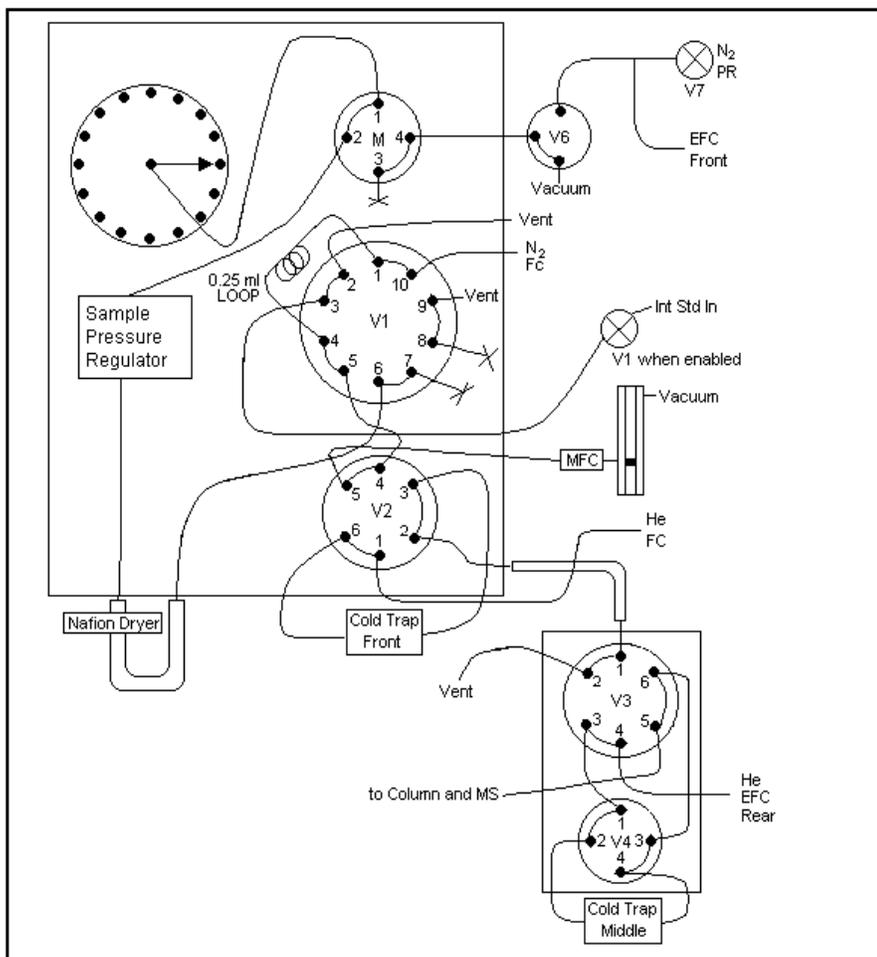


**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	-	+	-	-	-	-	-
11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	-	-	-	-
20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 8: Desorbs Cryofocuser Contents to GC Column and MS**  
**Time: 15.0 mins**



**Sample Delivery Program**

Time	V1	V2	V3	V4	V5	V6	V7
0.00	-	-	-	-	-	-	-
0.01	+	-	-	-	-	-	-
2.00	+	+	-	-	-	-	-
3.00	+	+	-	-	+	-	-
4.00	+	+	-	-	-	-	-
5.00	-	+	-	-	-	-	-
11.00	-	-	-	-	-	-	-
14.00	-	-	-	+	-	-	-
14.90	-	-	+	+	-	-	-
15.00	-	-	+	-	-	-	-
20.00	-	-	-	-	-	-	-

- V1 Sample Valve
- V2 Sample Preconcentration Trap Valve
- V3 Sample Preconcentration Trap Valve
- V4 Series Bypass Valve
- V5 Event A Valve
- V6 Event B Valve
- V7 Event C Valve

**Figure 9. Concentrator Programming Sequence**

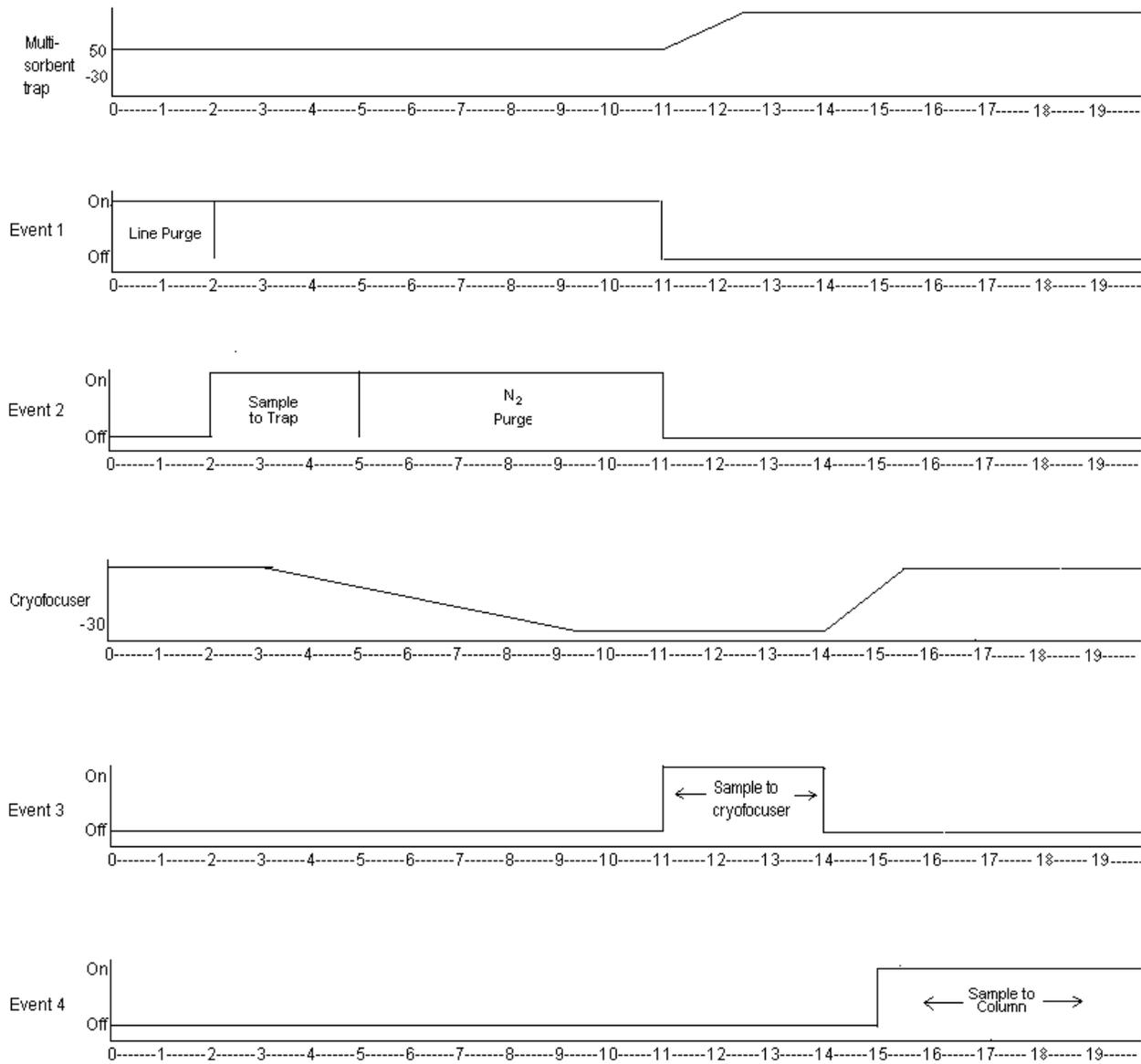


Figure 10. Typical Calibration Standard TIC, CC50604 Gas Mixture

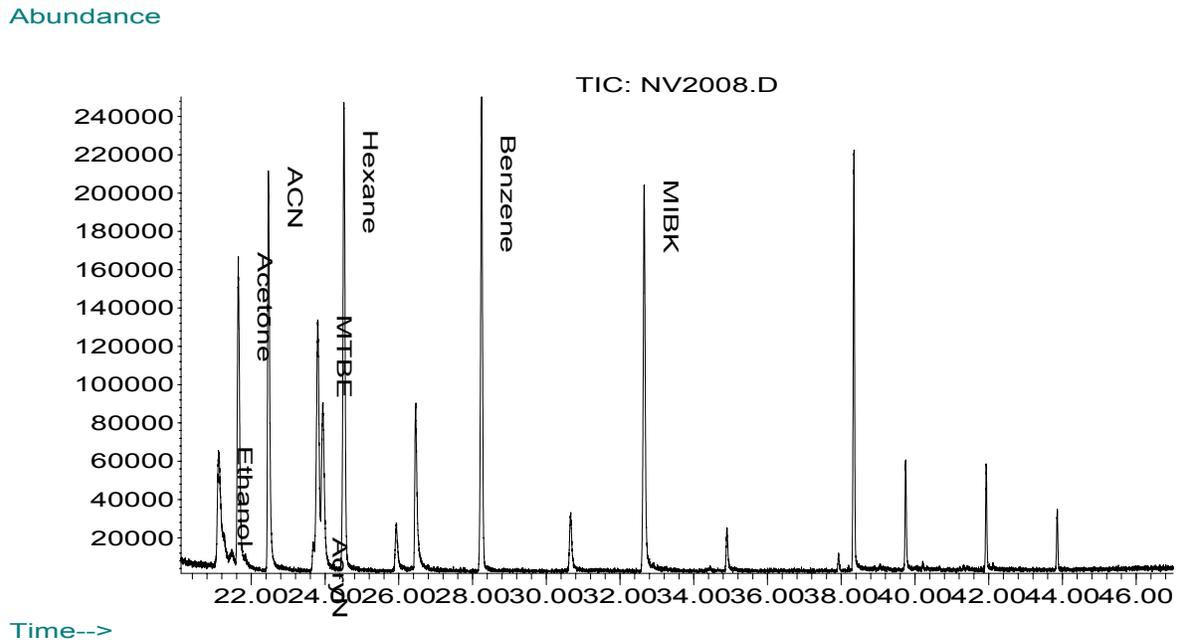


Figure 11: Typical Ambient Air TIC, Long Beach Sample.

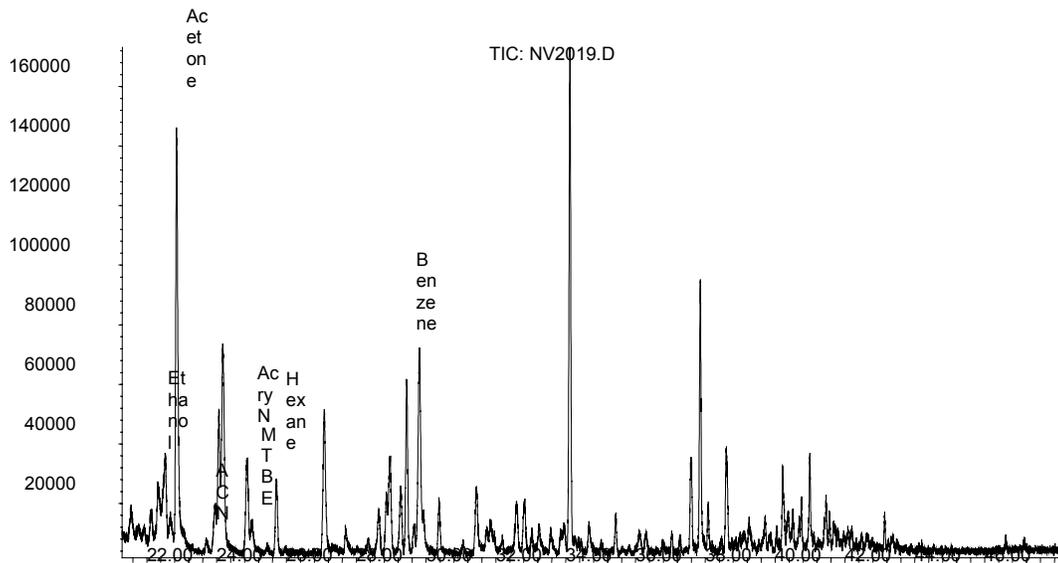
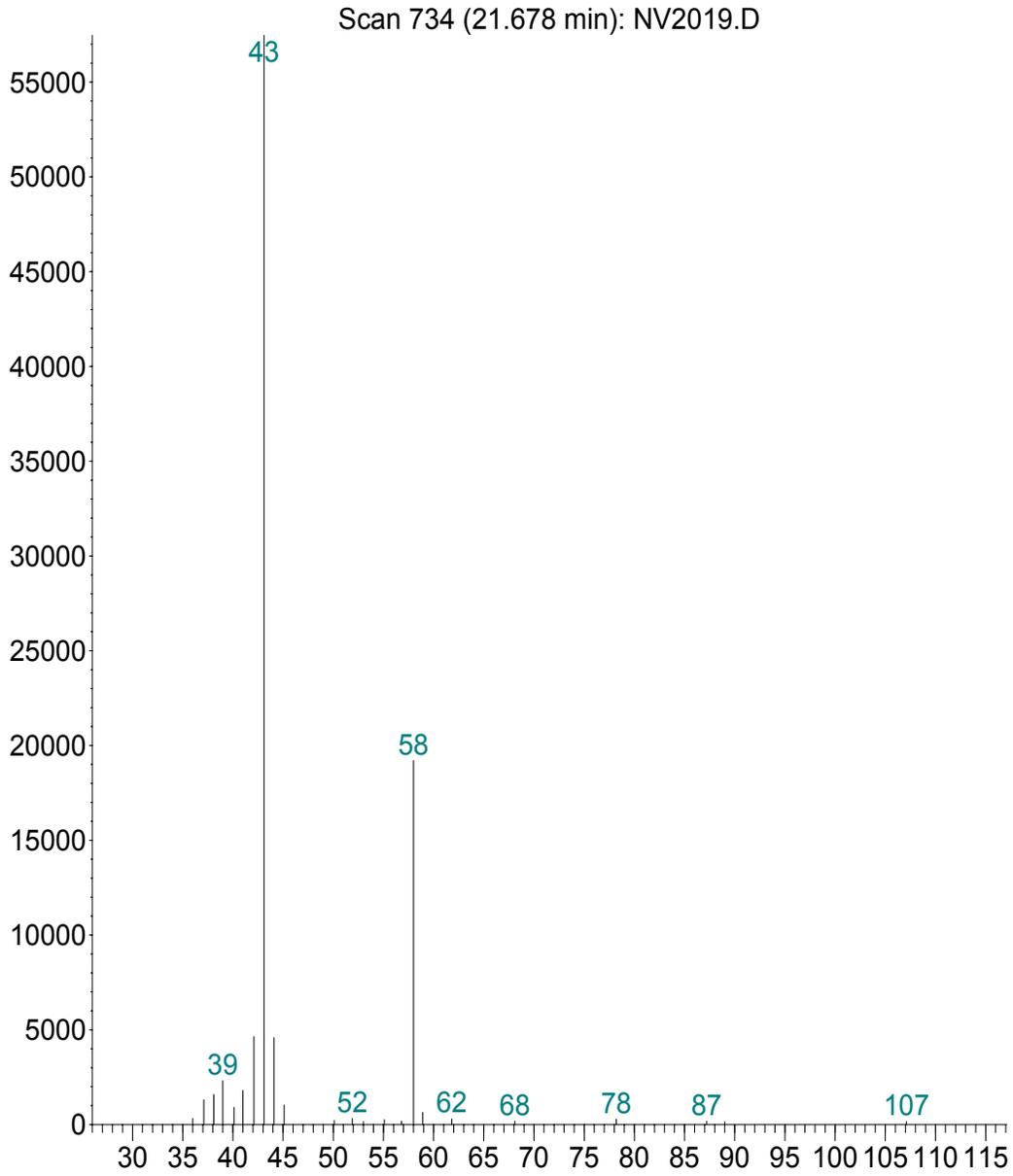


Figure 12: Typical Mass Spectrum, Acetone

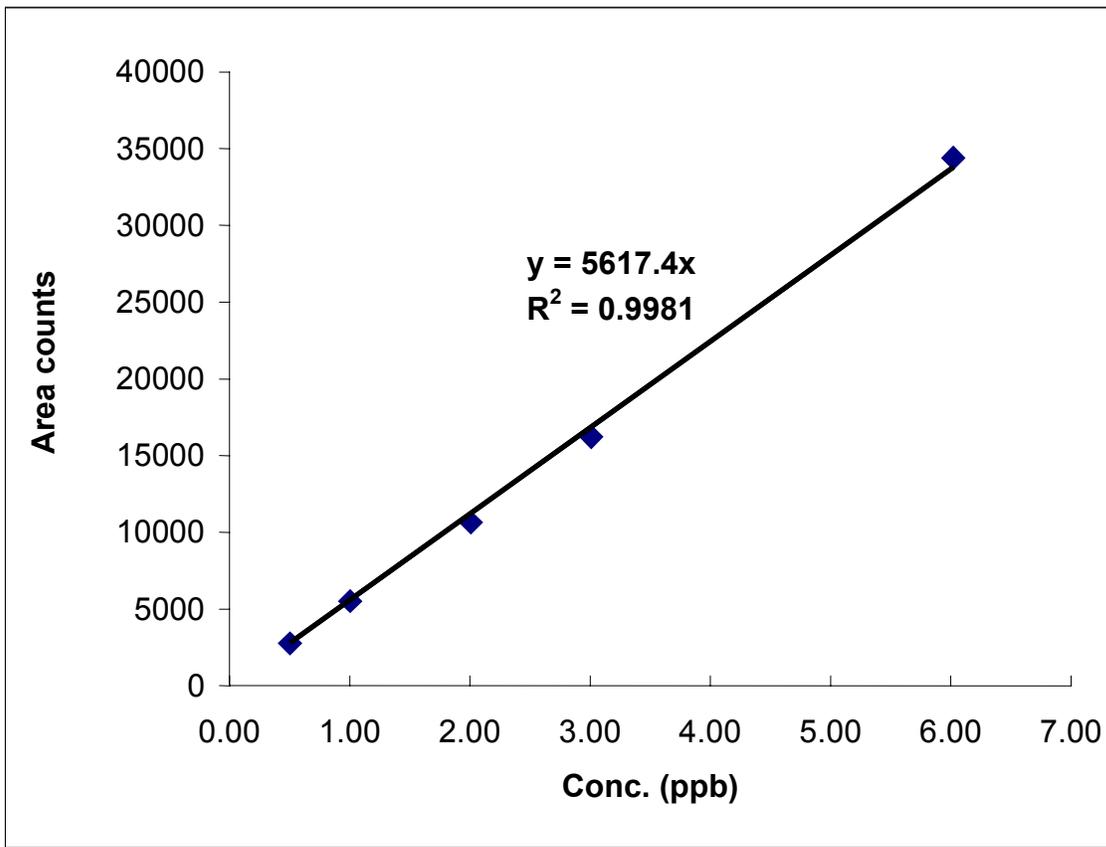
Abundance



m/z-->

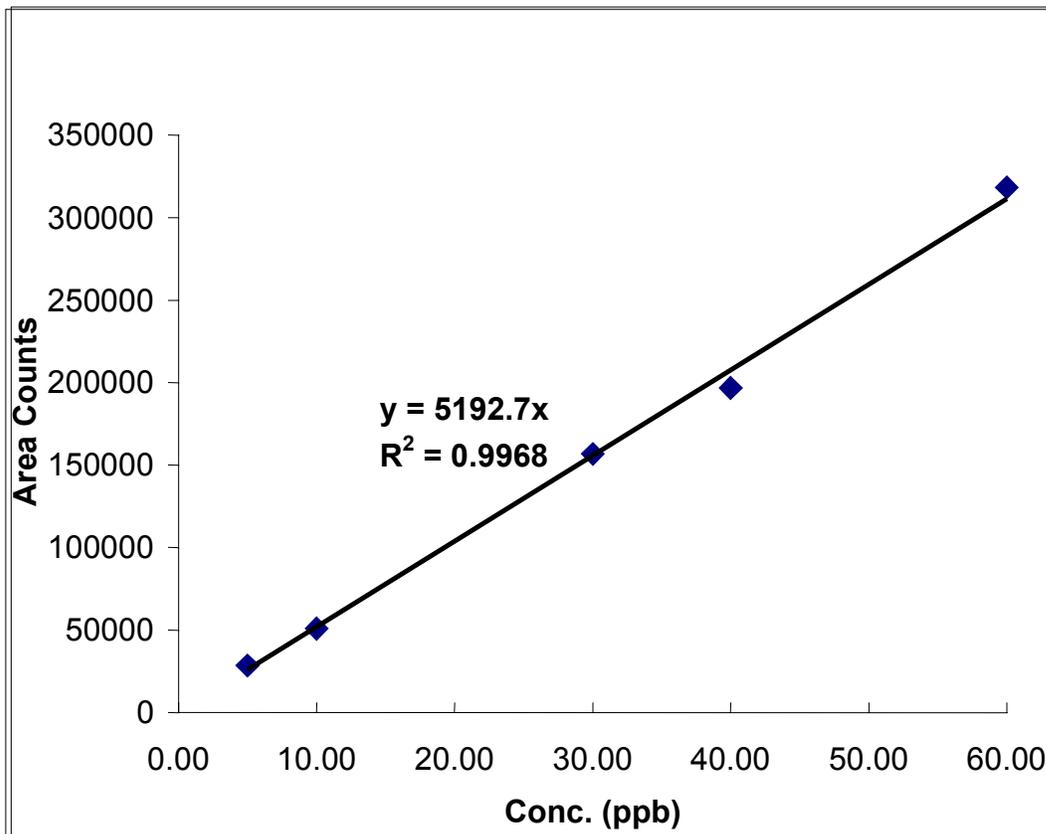
**Figure 13: Acrolein Multipoint Analysis (10/22/03 – ALM069798  
3.01 ppb**

LEVELS OF CONCENTRATION (PPB)					
cc	25	50	100	150	300
ppb	0.502	1.003	2.007	3.010	6.020
1st Run	2725	5297	10963	16647	34836
2nd	2956	5660	10621	16159	34068
3rd	2574	5570	10353	15872	34279
Mean=	2752	5509	10646	16226	34394
Std.Dev.=	192	189	306	392	397
%RSD=	7.0	3.4	2.9	2.4	1.2
# Obs. =	3	3	3	3	3



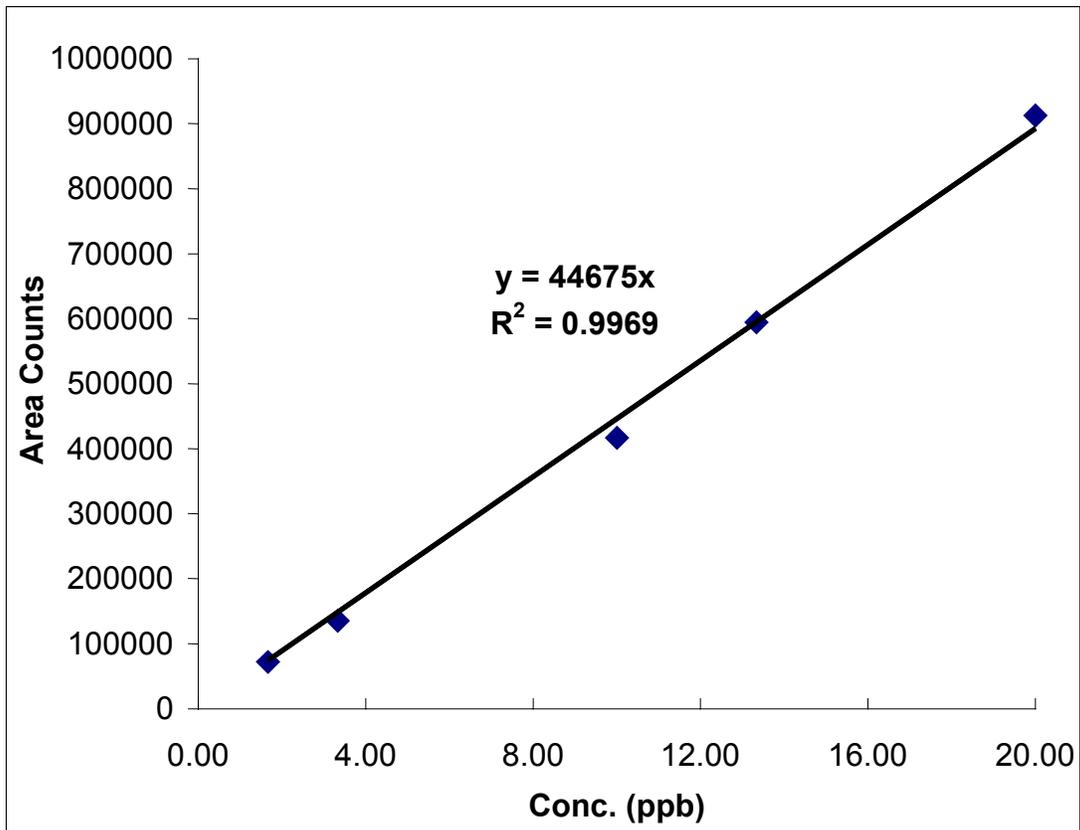
**Figure 14: Ethanol Multipoint Analysis (03/11/03) – CC50604  
30 ppb**

LEVELS OF CONCENTRATION (PPB)					
cc	25	50	150	200	300
ppb	5.000	10.000	30.000	40.000	60.000
1st Run	29009	53893	153431	201777	308912
2nd	28321	50630	156982	189902	317477
3rd	28304	48100	160072	198264	328727
Mean=	28545	50874	156828	196648	318372
Std.Dev.=	402	2904	3323	6100	9938
%RSD=	1.4	5.7	2.1	3.1	3.1
# Obs. =	3	3	3	3	3



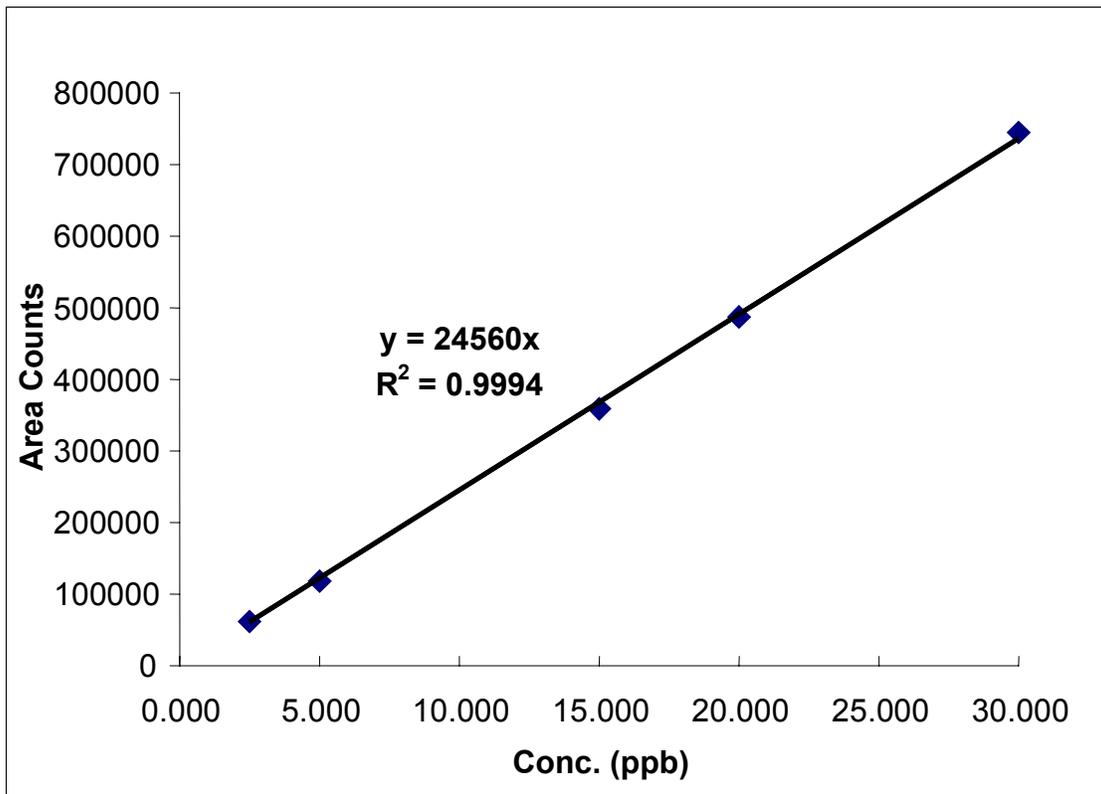
**Figure 15: Acetone Multipoint Analysis (3/11/03) – CC50604**  
**10.0 ppb**

LEVELS OF CONCENTRATION (PPB)					
cc	25	50	150	200	300
ppb	1.667	3.333	10.000	13.333	20.000
1st Run	75092	135818	415917	592473	906216
2nd	71419	135000	418240	599978	910896
3rd	68762	133866	414744	590280	919560
Mean=	71758	134895	416300	594244	912224
Std.Dev.=	3179	980	1779	5086	6770
%RSD=	4.4	0.7	0.4	0.9	0.7
# Obs. =	3	3	3	3	3



**Figure 16: Acetonitrile Multipoint Analysis (3/11/03) – CC50604  
15.0 ppb**

LEVELS OF CONCENTRATION (PPB)					
cc	25	50	150	200	300
ppb	2.500	5.000	15.000	20.000	30.000
1st Run	65828	119753	358820	482681	739275
2nd	61177	120424	353132	490058	743130
3rd	58558	114277	364630	489153	752261
Mean=	61854	118151	358861	487297	744889
Std.Dev.=	3682	3372	5749	4023	6669
%RSD=	6.0	2.9	1.6	0.8	0.9
# Obs. =	3	3	3	3	3



**Figure 17: Acrylonitrile Multipoint Analysis (3/11/03) – CC50604  
2.00 ppb**

LEVELS OF CONCENTRATION (PPB)					
cc	25	50	150	200	300
ppb	0.333	0.667	2.000	2.667	4.000
1st Run	6721	8068	23069	29957	48953
2nd	5129	8131	22359	31677	49398
3rd	4565	7303	21673	31164	50342
Mean=	5472	7834	22367	30933	49564
Std.Dev.=	1118	461	698	883	709
%RSD=	20.4	5.9	3.1	2.9	1.4
# Obs. =	3	3	3	3	3

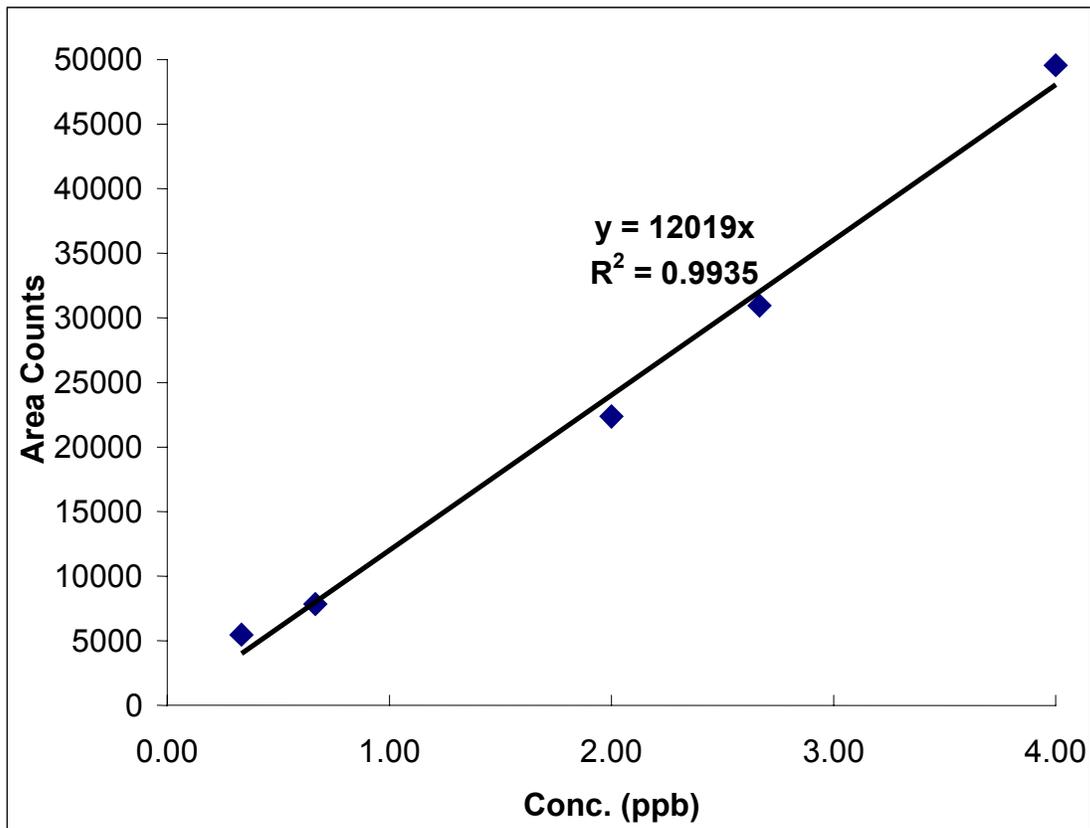


Figure 18: Acrolein Control Chart

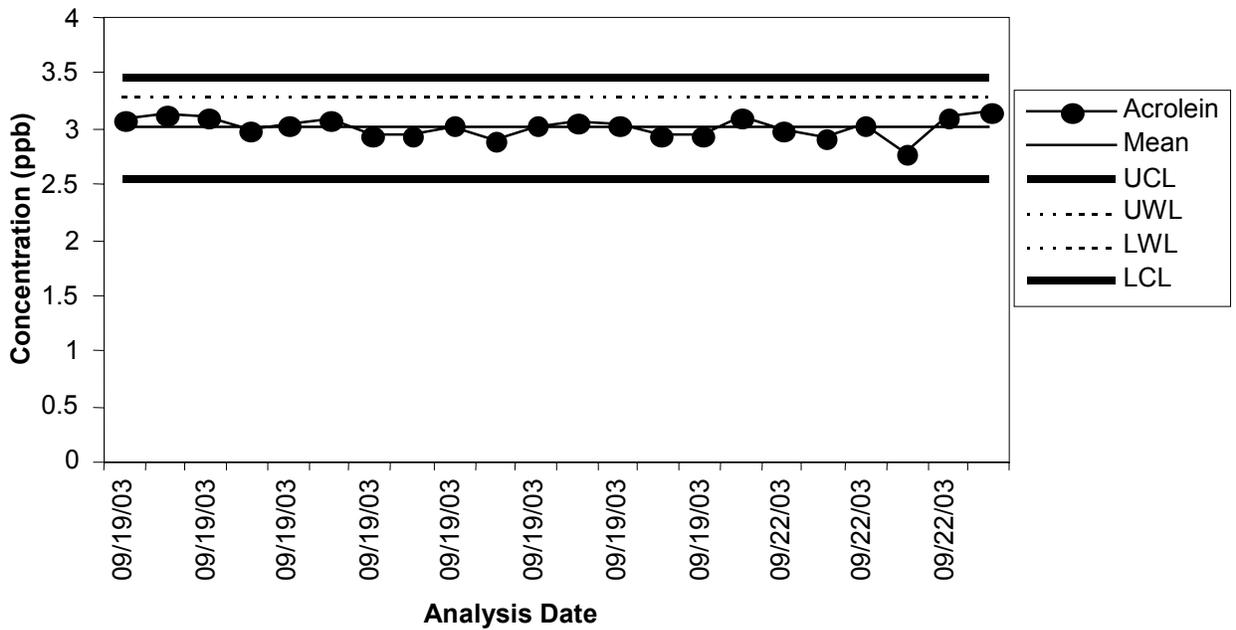


Figure 19: Ethanol Control Chart

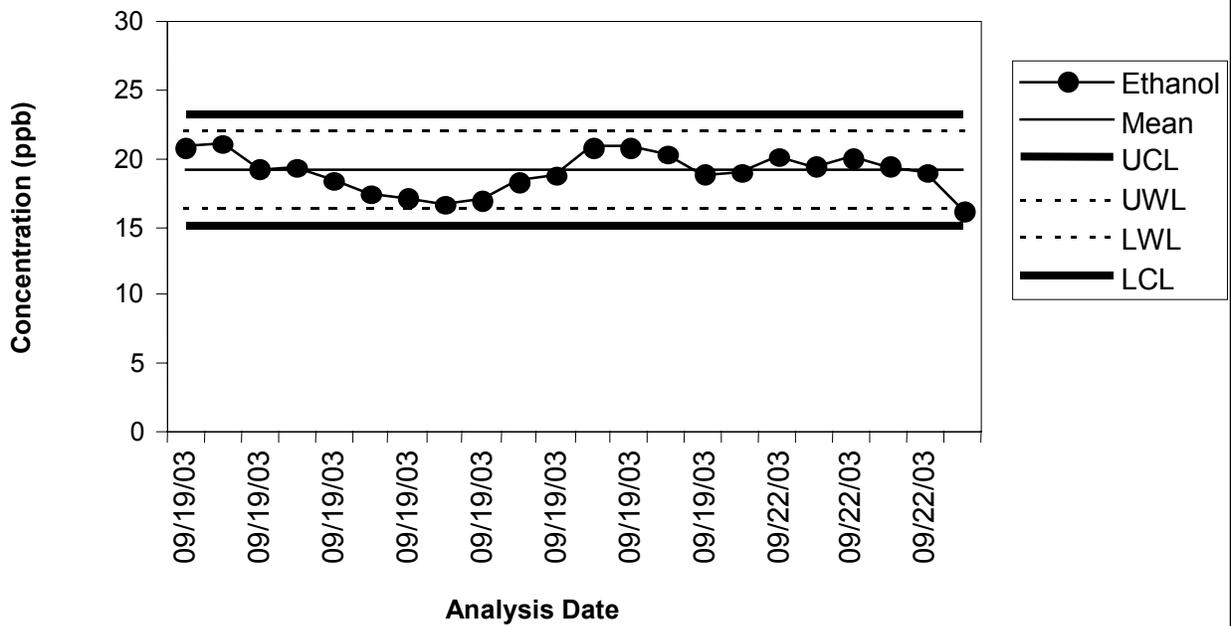
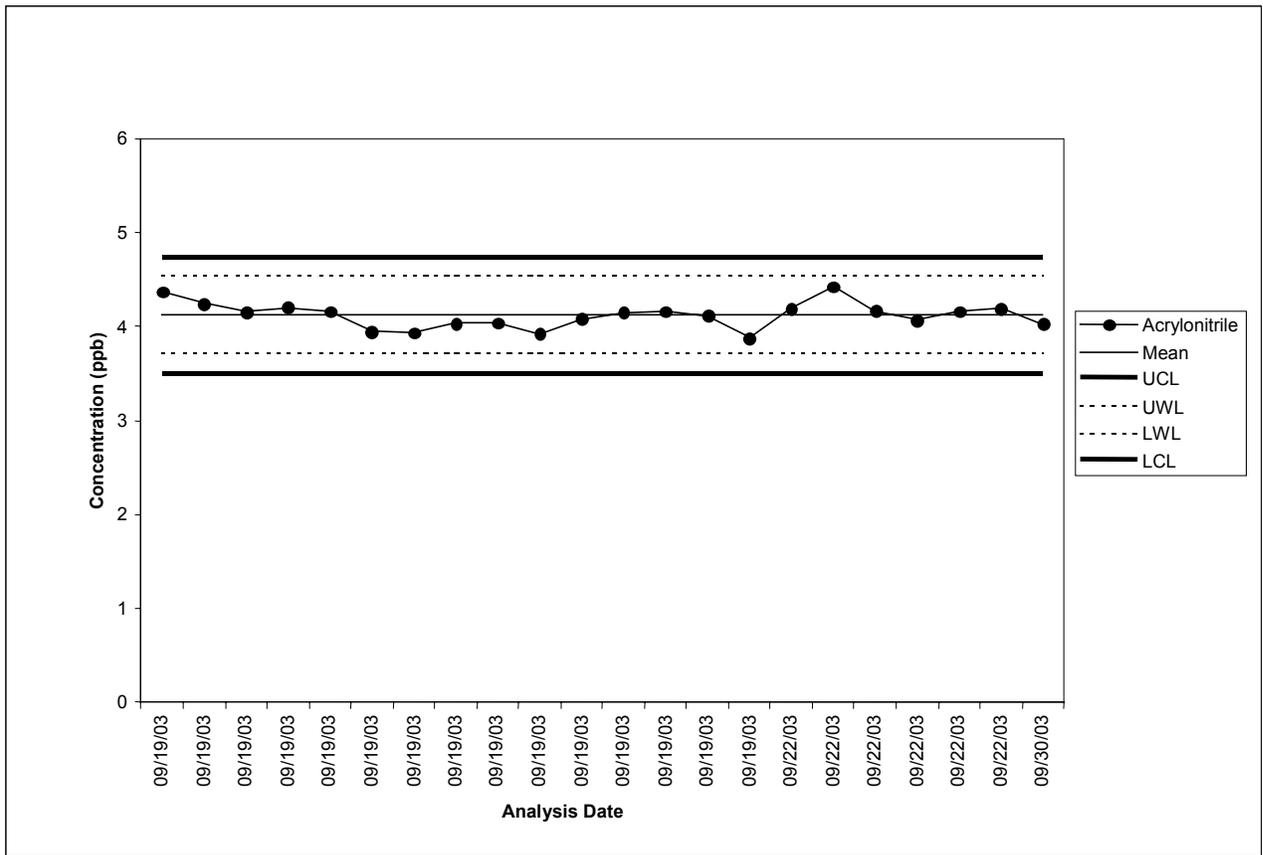




Figure 22: Acrylonitrile Control Chart



# Appendix I: Additional Setpoints

## He Carrier Gas:

Set Rear Type 3 Electronic Flow Controller to 1.2 cm<sup>3</sup>/minute

## N<sub>2</sub> Purge Gas:

Set digital gauge on Flow Controller to 16.0 (~ cm<sup>3</sup>/minute)

## He Purge Gas:

Set digital gauge on Flow Controller to 7.05 (~ cm<sup>3</sup>/minute)

## Nafion Dryer Purge:

Set digital gauge on Flow Controller to 7.05 (~ cm<sup>3</sup>/minute)

## Mass Flow Controller (MFC):

Set sampling flow rate to 50 cm<sup>3</sup>/minute

Set .....50.1% of full scale  
Read .....50.6% of full scale

Note: 100 cm<sup>3</sup>/minute equals 100% full scale

## Required Regulator Pressures:

He - Carrier Gas and Purge Gas .....60 psi

N<sub>2</sub> - Purge Gas and Nafion™ Dryer Gas .....60 psi

## Appendix II: Target Analyte LODs and Highest Calibration Concentration

Target compound	Recommended Published LOD	Replicate LOD	Multipoint Analysis Data	
			Correlation coefficient, R	Linear range
Acrolein	0.30	0.14	0.999	6.
Ethanol	0.50	0.28	0.9984	60
Acetone	0.30	0.12	0.9984	20
Acetonitrile	0.30	0.12	0.9997	30
Acrylonitrile	0.30	0.05	0.9967	4

## Appendix III: Varian Star Chromatography Workstation

A Varian GC Star Workstation includes an Intel compatible PC, an Ethernet network adapter, Microsoft 9.X or NT 4.0 operating system, and Varian Star Chromatography software, Version 5.51. The chromatography software operates under Microsoft Windows 9.X or Microsoft Windows NT 4.0. The Star Workstation automates control of the Varian/Lotus Sorbent Concentration and Cryofocuser System, including concentration of the sample, introduction of the concentrated sample onto the gas chromatographic column, and setting the column carrier gas flow. For a more detailed discussion of the Star Workstation software, including setting up methods, sequences, and sample lists, refer to the manuals on the "Varian Star Chromatography Workstation", Version 5.51 CD-ROM and the "Varian Saturn GC/MS Workstation – System Software", Version 5.51 CD-ROM. Additional resources are the "Ultra Trace Hydrocarbon System Operator's Manual", the "Stream Selector Valve Control Software for Varian Workstation Operator's Manual", and the "Varian GC Star Workstation Manual", all by Randall Bramston-Cook of Lotus Consulting.

A Hewlett-Packard ChemStation, running Hewlett-Packard Analytical MSD Productivity ChemStation Software, is used to automate the control of the Hewlett-Packard Model 6890 Gas Chromatograph with a Model 5973 Mass Selective Detector (MSD).

Each Gas Chromatograph (GC) serviced by the Star Chromatography Workstation is assigned a separate address. Each Workstation can be linked to a maximum of four (4) GCs. In a single GC environment, the Varian/Lotus 3800 Cryogenic Concentrator would normally have an address of 44. The instrument setpoints are stored on the Workstation as methods. Method MLD066S.MTH is used for normal operation. Other methods include IDLE66.MTH for system standby, SBAKEOUT.MTH for conditioning/bakeout of the system. They are used in automated sequences along with method MLD066S.

Copies of the current Star GC Chromatography Workstation analytical, idle and bakeout methods are listed. Although there are sections for data handling and reporting, they are not used in this analysis, and are shown in lighter type. Examples of a sample list and a sequence list are also shown.

Varian Star Workstation

Star Chromatography Workstation - Method Listing    Mon Dec 15  
09:41:10 2003

Method: MLD066S.mth

\*\*\*\*\*

3800 GC

\*\*\*\*\*

Module Address: 44

Middle Valve Oven

-----

Oven Power: On  
Temperature: 120 C

Rear Valve Oven

-----

Oven Power: On  
Temperature: 50 C

Valve Table

-----

Valve 1: Sample Valve

Initial: Off  
0.01 min: On  
2.00 min: On  
3.00 min: On  
4.00 min: On  
5.00 min: Off  
11.00 min: Off  
14.00 min: Off  
14.90 min: Off  
15.00 min: Off  
20.00 min: Off

Valve 2: Sample Preconcentration Trap Valve

Initial: SPT Desorb  
0.01 min: SPT Desorb  
2.00 min: SPT Trap  
3.00 min: SPT Trap  
4.00 min: SPT Trap  
5.00 min: SPT Trap  
11.00 min: SPT Desorb  
14.00 min: SPT Desorb  
14.90 min: SPT Desorb  
15.00 min: SPT Desorb  
20.00 min: SPT Desorb

## Varian Star Workstation

### Valve 3: Sample Preconcentration Trap Valve

Initial: SPT Desorb  
0.01 min: SPT Desorb  
2.00 min: SPT Desorb  
3.00 min: SPT Desorb  
4.00 min: SPT Desorb  
5.00 min: SPT Desorb  
11.00 min: SPT Desorb  
14.00 min: SPT Desorb  
14.90 min: SPT Trap  
15.00 min: SPT Trap  
20.00 min: SPT Desorb

### Valve 4: Series Bypass Valve

Initial: Series  
0.01 min: Series  
2.00 min: Series  
3.00 min: Series  
4.00 min: Series  
5.00 min: Series  
11.00 min: Series  
14.00 min: Bypass  
14.90 min: Bypass  
15.00 min: Series  
20.00 min: Series

### Valve 5: Event A Valve

Initial: Off  
0.01 min: Off  
2.00 min: Off  
3.00 min: On  
4.00 min: Off  
5.00 min: Off  
11.00 min: Off  
14.00 min: Off  
14.90 min: Off  
15.00 min: Off  
20.00 min: Off

### Valve 6: Event B Valve

Initial: Off  
0.01 min: Off  
2.00 min: Off  
3.00 min: Off  
4.00 min: Off

## Varian Star Workstation

5.00 min: Off  
11.00 min: Off  
14.00 min: Off  
14.90 min: Off  
15.00 min: Off  
20.00 min: Off

### Valve 7: Event C Valve

Initial: Off  
0.01 min: Off  
2.00 min: Off  
3.00 min: Off  
4.00 min: Off  
5.00 min: Off  
11.00 min: Off  
14.00 min: Off  
14.90 min: Off  
15.00 min: Off  
20.00 min: Off

### Front Injector Type 1079

-----

Oven Power: On  
Coolant: On  
Enable Coolant at: 300 C  
Coolant Timeout: 20.00 min

Temp (C)	Rate (C/min)	Hold (min)	Total (min)
125	0	11.00	11.00
325	200	28.00	40.00

### Middle Injector Type 1079

-----

Oven Power: On  
Coolant: On  
Enable Coolant at: 300 C  
Coolant Timeout: 20.00 min

Temp (C)	Rate (C/min)	Hold (min)	Total (min)
200	0	3.00	3.00
-30	200	9.85	14.00

## Varian Star Workstation

325 200 24.23 40.00

Rear Injector Type 1041

-----  
Oven Power: On  
Temperature: 150 C

Rear Injector EFC Type 3

-----  
Flow Rate Hold Total  
(ml/min) (ml/min/min) (min) (min)  
-----  
2.0 0.0 45.00 45.00

Column Oven

Coolant: Off  
Enable Coolant at: 50 C  
Coolant Timeout: 20.00 min  
Stabilization Time: 0.50 min

Temp Rate Hold Total  
(C) (C/min) (min) (min)  
-----  
50 0.0 48.00 48.00

Front FID Detector

-----  
Oven Power: Off  
Temperature: 50 C  
Electronics: On  
Time Constant: Fast

Time Range Autozero  
(min)  
-----  
Initial 12 no

Output Port A

-----  
Time Signal Attenuation  
(min) Source  
-----  
Initial Front 1

## Varian Star Workstation

### Output Port B

-----

Time (min)	Signal Source	Attenuation
Initial	Front	1

### Output Port C

-----

Time (min)	Signal Source	Attenuation
Initial	Front	1

### Data Acquisition

-----

Detector Bunch Rate : 128 points (0.3 Hz)  
Monitor Length : 16 bunched points (51.2 sec)  
Front FID/TSD Scale: 1 Volts  
Middle FID/TSD Scale: 1 Volts  
Rear FID/TSD Scale: 1 Volts

## Varian Star Workstation

Star Chromatography Workstation - Method Listing    Mon Dec 15  
09:43:53 2003

Method: idle66.mth

\*\*\*\*\*

3800 GC

\*\*\*\*\*

Module Address: 44

Middle Valve Oven

-----

Oven Power: On  
Temperature: 120 C

Rear Valve Oven

-----

Oven Power: On  
Temperature: 50 C

Valve Table

-----

Valve 1: Sample Valve

Initial: Off  
0.20 min: Off  
15.00 min: Off  
15.10 min: Off

Valve 2: Sample Preconcentration Trap Valve

Initial: SPT Desorb  
0.20 min: SPT Desorb  
15.00 min: SPT Desorb  
15.10 min: SPT Desorb

Valve 3: Sample Preconcentration Trap Valve

Initial: SPT Desorb  
0.20 min: SPT Desorb  
15.00 min: SPT Desorb  
15.10 min: SPT Desorb

Valve 4: Series Bypass Valve

Initial: Series  
0.20 min: Series  
15.00 min: Series  
15.10 min: Series

## Varian Star Workstation

Valve 5: Event A Valve

Initial: Off  
0.20 min: On  
15.00 min: Off  
15.10 min: Off

Valve 6: Event B Valve

Initial: Off  
0.20 min: Off  
15.00 min: Off  
15.10 min: Off

Valve 7: Event C Valve

Initial: Off  
0.20 min: Off  
15.00 min: Off  
15.10 min: Off

Front Injector Type 1079

-----

Oven Power: On  
Coolant: On  
Enable Coolant at: 250 C  
Coolant Timeout: 20.00 min

Temp (C)	Rate (C/min)	Hold (min)	Total (min)
180	0	0.20	0.20

Middle Injector Type 1079

-----

Oven Power: On  
Coolant: On  
Enable Coolant at: 250 C  
Coolant Timeout: 20.00 min

Temp (C)	Rate (C/min)	Hold (min)	Total (min)
200	0	0.10	0.10

Rear Injector Type 1041

-----

## Varian Star Workstation

Oven Power: On  
Temperature: 150 C

### Rear Injector EFC Type 3

```
-----  
      Flow      Rate      Hold      Total  
(ml/min) (ml/min/min) (min)      (min)  
-----  
      2.0       0.0       1.00      1.00
```

### Column Oven

```
-----  
                Coolant: Off  
      Enable Coolant at: 50 C  
      Coolant Timeout: 20.00 min  
      Stabilization Time: 0.10 min
```

```
-----  
      Temp      Rate      Hold      Total  
      (C)      (C/min)    (min)    (min)  
-----  
      50       0.0      45.00   45.00
```

### Front FID Detector

```
-----  
      Oven Power: Off  
      Temperature: 50 C  
      Electronics: Off  
      Time Constant: Fast
```

```
-----  
      Time      Range  Autozero  
      (min)  
-----  
      Initial   12    yes
```

### Output Port A

```
-----  
      (min)      Source  
-----  
      Initial   Front      1
```

### Output Port B

```
-----  
      Time      Signal  Attenuation
```

### Varian Star Workstation

(min)	Source	
Initial	Front	1

### Output Port C

Time (min)	Signal Source	Attenuation
Initial	Front	1

### Data Acquisition

Detector Bunch Rate : 4 points (10.0 Hz)  
Monitor Length : 64 bunched points (6.4 sec)  
Front FID/TSD Scale: 1 Volts  
Middle FID/TSD Scale: 1 Volts  
Rear FID/TSD Scale: 1 Volts

## Varian Star Workstation

Star Chromatography Workstation - Method Listing    Mon Dec 15  
09:42:41 2003

Method: SBAKEOUT.mth

\*\*\*\*\*

### Notes

\*\*\*\*\*

THIS IS A 20 MIN RUN TO CONDITION THE VARIAN3800 STAR ATD SYATEM  
BEFORE AN ANALYTICAL METHOD IS ACTIVATED TO RUN A SAMPLE LIST.

\*\*\*\*\*

3800 GC

\*\*\*\*\*

Module Address: 44

### Middle Valve Oven

-----

Oven Power: On  
Temperature: 120 C

### Rear Valve Oven

-----

Oven Power: On  
Temperature: 50 C

### Valve Table

-----

Valve 1: Sample Valve  
  Initial: Off  
  1.00 min: Off  
  20.00 min: Off

Valve 2: Sample Preconcentration Trap Valve  
  Initial: SPT Desorb  
  1.00 min: SPT Desorb  
  20.00 min: SPT Desorb

Valve 3: Sample Preconcentration Trap Valve  
  Initial: SPT Desorb  
  1.00 min: SPT Desorb  
  20.00 min: SPT Desorb

Valve 4: Series Bypass Valve  
  Initial: Series  
  1.00 min: Series  
  20.00 min: Series

## Varian Star Workstation

Valve 5: Event A Valve  
Initial: Off  
1.00 min: On  
20.00 min: Off

Valve 6: Event B Valve  
Initial: Off  
1.00 min: Off  
20.00 min: Off

Valve 7: Event C Valve  
Initial: Off  
1.00 min: Off

### Front Injector Type 1079

-----

Oven Power: On  
Coolant: On  
Enable Coolant at: 250 C  
Coolant Timeout: 20.00 min

Temp (C)	Rate (C/min)	Hold (min)	Total (min)
325	0	20.00	20.00

-----

### Middle Injector Type 1079

-----

Oven Power: On  
Coolant: On  
Enable Coolant at: 250 C  
Coolant Timeout: 20.00 min

Temp (C)	Rate (C/min)	Hold (min)	Total (min)
325	0	20.00	20.00

-----

### Rear Injector Type 1041

-----

Oven Power: On  
Temperature: 150 C

### Rear Injector EFC Type 3

-----

## Varian Star Workstation

Flow (ml/min)	Rate (ml/min/min)	Hold (min)	Total (min)
2.0	0.0	20.00	20.00

### Column Oven

-----  
Coolant: Off  
Enable Coolant at: 50 C  
Coolant Timeout: 20.00 min  
Stabilization Time: 0.10 min

Temp (C)	Rate (C/min)	Hold (min)	Total (min)
50	0.0	20.00	20.00

### Front FID Detector

-----  
Oven Power: Off  
Temperature: 50 C  
Electronics: Off  
Time Constant: Fast

Time (min)	Range	Autozero
Initial	12	no

### Output Port A

Time (min)	Signal Source	Attenuation
Initial	Front	1

### Output Port B

-----  

Time (min)	Signal Source	Attenuation
Initial	Front	1

## Varian Star Workstation

Output Port C

-----

Time (min)	Signal Source	Attenuation
Initial	Front	1

Data Acquisition

-----

Detector Bunch Rate : 4 points (10.0 Hz)  
Monitor Length : 64 bunched points (6.4 sec)  
Front FID/TSD Scale: 1 Volts  
Middle FID/TSD Scale: 1 Volts  
Rear FID/TSD Scale: 1 Volts

# Varian Star Workstation

Varian Star Workstation - Generic SampleList Mon Dec 15  
09:46:23 2003

SampleList: C:\Star\data\DEC0403.smp

Created: Thu Dec 04 13:52:08 2003

Modified: Fri Dec 05 09:47:56 2003

Amount	Line	Sample Type	Sample Name	Inj.	AutoLink	Standard	
	1	AutoLink	c:\star\ssvauto.exe	Other	parameters: -i		
	2	Analysis	BLANK	1	below	1	
		AutoLink	Command c:\star\ssvauto.exe				
	3	Activate Method	C:\Star\METHODS\MLD066S25cc.mth				
	4	Analysis	AAL069919#1054	1	below	2	
		AutoLink	Command c:\star\ssvauto.exe				
	5	Analysis	CC50604 25CC #1061	1	below	3	
		AutoLink	Command c:\star\ssvauto.exe				
	6	Activate Method	C:\Star\METHODS\MLD066S50cc.mth				
	7	Analysis	CC50604 50CC	1	below	3	
		AutoLink	Command c:\star\ssvauto.exe				
	8	Activate Method	C:\Star\METHODS\MLD066S75cc.mth				
	9	Analysis	CC50604 75CC	1	below	3	
		AutoLink	Command c:\star\ssvauto.exe				
	10	Activate Method	C:\Star\METHODS\MLD066S100.mth				
	11	Analysis	CC50604 100CC	1	below	3	
		AutoLink	Command c:\star\ssvauto.exe				
	12	Activate Method	C:\Star\METHODS\MLD066S.mth				
	13	Analysis	CC50604 150CC	1	below	3	
		AutoLink	Command c:\star\ssvauto.exe				

## Varian Star Workstation

14	Analysis	ALM069798 #1072	1	below	4
	AutoLink	Command c:\star\ssvauto.exe			
15	Analysis	5669BFREG	1	below	5
	AutoLink	Command c:\star\ssvauto.exe			
16	Analysis	5670BFCOL	1	below	6
17	Analysis	5671RUREG	1	below	7
	AutoLink	Command c:\star\ssvauto.exe			
18	Analysis	5672RUCOL	1	below	8
	AutoLink	Command c:\star\ssvauto.exe			
19	Analysis	5673RS	1	below	9
	AutoLink	Command c:\star\ssvauto.exe			
20	Analysis	5674CX	1	below	11
	AutoLink	Command c:\star\ssvauto.exe			
21	Analysis	5675CV	1	below	12
	AutoLink	Command c:\star\ssvauto.exe			
22	Analysis	5654BFREG	1	below	13
	AutoLink	Command c:\star\ssvauto.exe			
23	Analysis	5655BFCOL	1	below	14
	AutoLink	Command c:\star\ssvauto.exe			
24	Activate Method	C:\Star\METHODS\MLD066S25cc.mth			
25	Analysis	5642SV RR 25CC	1	below	10
	AutoLink	Command c:\star\ssvauto.exe			
26	Activate Method	C:\Star\METHODS\MLD066S.mth			
27	Analysis	5669DUP	1	below	5
	AutoLink	Command c:\star\ssvauto.exe			
	AutoLink	Command c:\star\ssvauto.exe			

# Varian Star Workstation

Varian Star Workstation - Sequence Listing Mon Dec 15 09:47:38  
2003

Sequence: C:\Star\voc.seq

Created: Wed Aug 13 12:32:26 2003

Modified: Fri Dec 05 10:41:12 2003

\*\*\*\*\*

Line	Action	Method
====	=====	=====
1	Inject	c:\star\methods\sbaekout.mth
2	Inject	c:\star\methods\mld066s.mth
3	Print Message Log	
4	Inject	c:\star\methods\idle66.mth

## Appendix IV: Hewlett-Packard GC/MS ChemStation

A Hewlett-Packard GC/MS ChemStation includes an Intel compatible PC, an Ethernet network adapter, a GPIB interface card, Microsoft 9.X or NT 4.0 operating system, and Hewlett-Packard Analytical MSD Productivity ChemStation Software, Version A.03.00 or B.03.00. The A software operates under Microsoft Windows 9.X, and the B operates under Microsoft Windows NT 4.0. They are functionally equivalent.

The Hewlett-Packard ChemStation automates control of the Hewlett-Packard Model 6890 Gas Chromatograph and its associated Model 5973 Mass Selective Detector (MSD). This includes controlling the temperature of the GC column oven, the GC to MSD transfer line, and all operating and data acquisition parameters of the MSD. This software is also used for the analysis and reporting of the acquired MS data. For a more detailed discussion of the ChemStation software, including setting up methods, sequences, and sample lists, and data analysis, refer to the manuals, on the CD-ROM, "HP 5973 MSD Reference Collection", Revision C.00.00, by Hewlett-Packard. A Varian Star GC Chromatography Workstation, running Varian Star Chromatography software, is used to automate the control of the Lotus/Varian 3800 Concentrator.

The instrument setpoints are stored on the ChemStation as methods. Method MLD066.M is used for normal operation. Method IDLE.M and IDLE1 are used for system standby and bakeout steps. Both methods are used in automated sequences. Method MLD066.M also includes data handling and reporting sections.

A copy of the current Hewlett-Packard ChemStation analytical and idle methods, including sections for data handling and reporting, are listed. An example SAMPLE.S sequence list screen is also shown.

The analytical data files collected by the Hewlett-Packard ChemStation are named in the following manner:

**DC 04 03**                    =            **DECEMBER 04, 2003**

/                    |                    \  
**Month**            **Day**                    **Year**  
**Code**            **Code**

The applicable month codes are:

January	=	JA	May	=	MY	September	=	SE
February	=	FB	June	=	JN	October	=	OC
March	=	MR	July	=	JL	November	=	NV
April	=	AP	August	=	AG	December	=	DC

# HP GC/MS Chemstation

Method Information For: C:\HPCHEM\1\METHODS\MLD066.M

Method Sections To Run:

- ( ) Save Copy of Method With Data
- ( ) Pre-Run Cmd/Macro =
- (X) Data Acquisition
- (X) Data Analysis
- ( ) Post-Run Cmd/Macro =

Method Comments:

This is a method for the analysis of ambient air for OXYGENATES, ACETONITRILE AND ACRYLONITRILE.

END OF TOPLEVEL PARAMETERS

-----  
INSTRUMENT CONTROL PARAMETERS  
-----

Sample Inlet: GC  
Injection Source: External Device  
Injection Location: Front  
Mass Spectrometer: Enabled

=====  
HP6890 GC METHOD  
=====

OVEN

Initial temp: 60 'C (On)                   Maximum temp: 260 'C  
Initial time: 8.00 min                    Equilibration time: 0.50 min

Ramps:

#	Rate	Final temp	Final time	CRYO (N2)
1	40.00	-20	4.00	Cryo: On
2	5.00	80	0.00	Cryo fault: On
3	10.00	200	2.00	Cryo timeout: 60.00 min (On)
4	0.0(Off)			Quick cryo cool: On

Post temp: 0 'C  
Post time: 0.00 min  
Run time: 48.00 min

Ambient temp: 55 'C

FRONT INLET (HP PTV)

Mode: Split  
Initial temp: 50 'C (Off)

BACK INLET (SPLIT/SPLITLESS)

Mode: Split  
Initial temp: 50 'C (Off)

## HP GC/MS Chemstation

Cryo: On  
Cryo use temp: 25 'C  
Cryo Timeout: 30.00 min (On)  
Cryo Fault: On  
Pressure: 1.10 psi (Off)  
Split ratio: 50:1  
Split flow: 61.3 mL/min  
Total flow: 65.7 mL/min  
Gas saver: Off  
Gas type: Helium

Pressure: 0.00 psi (Off)  
Total flow: 45.0 mL/min  
Gas saver: Off  
Gas type: Helium

### COLUMN 1

Capillary Column  
Model Number: J & W DB-624  
30m x 0.32 mm i.d. and 1.8 um thick  
Max temperature: 260 'C  
Nominal length: 30.0 m  
Nominal diameter: 320.00 um

### COLUMN 2

(not installed)

Method: MLD066.M

Mon Dec 15 09:52:18 2003

Page: 1

Mode: constant pressure  
Pressure: 1.10 psi  
Nominal initial flow: 1.2 mL/min  
Average velocity: 41 cm/sec  
Inlet: Front Inlet  
Outlet: MSD  
Outlet pressure: vacuum

FRONT DETECTOR (NO DET)

BACK DETECTOR (NO DET)

### SIGNAL 1

Data rate: 20 Hz  
Type: test plot  
Save Data: Off  
Zero: 0.0 (Off)  
Range: 0  
Fast Peaks: Off  
Attenuation: 0

### SIGNAL 2

Data rate: 20 Hz  
Type: test plot  
Save Data: Off  
Zero: 0.0 (Off)  
Range: 0  
Fast Peaks: Off  
Attenuation: 0

### COLUMN COMP 1

(No Detectors Installed)

### COLUMN COMP 2

(No Detectors Installed)

### THERMAL AUX 2

Use: MSD Transfer Line Heater

## HP GC/MS Chemstation

Description: MSD  
Initial temp: 280 'C (On)  
Initial time: 0.00 min  
# Rate Final temp Final time  
1 0.0 (Off)

POST RUN  
Post Time: 0.00 min

### TIME TABLE

Time	Specifier	Parameter & Setpoint
------	-----------	----------------------

7673 Injector

Front Injector:  
No parameters specified

Back Injector:

Sample Washes	0
Sample Pumps	0
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
PostInj Solvent A Washes	0
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

### MS ACQUISITION PARAMETERS

#### General Information

-----  
Tune File : ATUNE.U  
Acquisition Mode : Scan

#### MS Information

--  
Solvent Delay : 20.00 min  
EM Absolute : False  
EM Offset : 106  
Resulting EM Voltage : 2435.3  
  
Low Mass : 33  
High Mass : 200  
Threshold : 150  
Sample # : 2            A/D Samples    4

## HP GC/MS Chemstation

[MSZones]

MS Quad : 150 C maximum 200 C  
MS Source : 230 C maximum 250 C  
Timed Events

-----

[Timed MS Detector Entries]

Time (min)	State (MS on/off)
47.00	Off

END OF MS ACQUISITION PARAMETERS  
END OF INSTRUMENT CONTROL PARAMETERS

-----

DATA ANALYSIS PARAMETERS

-----

Method Name: C:\HPCHEM\3\METHODS\MLD066.M

Percent Report Settings

-----

Sort By: Retention Time

Output Destination

Screen: No

Printer: Yes

File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

-----

Peak Location of Unknown: Apex

Library to Search Minimum Quality

C:\DATABASE\NIST98.L 0

Integration Events: RTEINT.p

Report Type: Summary

## HP GC/MS Chemstation

### Output Destination

Screen: No  
Printer: Yes  
File: No

Generate Report During Run Method: No

### Quantitative Report Settings

-----

Report Type: Summary

### Output Destination

Screen: Yes  
Printer: No  
File: No

Generate Report During Run Method: Yes

### Analysis Method for Oxygenates

Calibration Last Updated: Mon Dec 08 09:16:24 2003

Reference Window: 10.00 Percent  
Non-Reference Window: 5.00 Percent  
Correlation Window: 0.02 minutes  
Default Multiplier: 1.00  
Default Sample Concentration: 0.00

### Compound Information

---

1) Acrolein ( )  
Ret. Time 21.04 min., Extract & Integrate from 20.84 to 21.24 min.  
Signal Rel Resp. Pct. Unc.(abs) Integration  
Tgt 56.00 \*\*\* METH DEFAULT \*\*\*  
Q1 55.00 72.90 20.0 \*\*\* METH DEFAULT \*\*\*  
Lvl ID Conc (ppbv) Response  
1 9.120 72782  
2 not used for this compound  
3 not used for this compound  
4 not used for this compound  
5 not used for this compound  
Qualifier Peak Analysis ON  
Curve Fit: Linear, forced through origin

---

## HP GC/MS Chemstation

2) Ethanol ( )  
Ret. Time 21.10 min., Extract & Integrate from 20.90 to 21.30 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 45.00			*** METH DEFAULT ***
Q1 46.00	10.00	20.0	*** METH DEFAULT ***
Q2 43.00	8.60	20.0	*** METH DEFAULT ***

Lvl ID	Conc (ppbv)	Response
1	30.000	189728
2	not used for this compound	
3	not used for this compound	
4	not used for this compound	
5	not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear, forced through origin

-----  
-----  
3) Acetone ( )  
Ret. Time 21.63 min., Extract & Integrate from 21.43 to 21.83 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 58.00	29.90	20.0	*** METH DEFAULT ***

Lvl ID	Conc (ppbv)	Response
1	10.000	454778
2	not used for this compound	
3	not used for this compound	
4	not used for this compound	
5	not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear, forced through origin

-----  
-----  
4) ACN ( )  
Ret. Time 22.46 min., Extract & Integrate from 22.26 to 22.66 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 39.00	10.60	20.0	*** METH DEFAULT ***

Lvl ID	Conc (ppbv)	Response
1	15.000	440488
2	not used for this compound	
3	not used for this compound	
4	not used for this compound	
5	not used for this compound	

## HP GC/MS Chemstation

Qualifier Peak Analysis ON

Curve Fit: Linear, forced through origin

-----  
-----  
5) AcryN ( )  
Ret. Time 23.67 min., Extract & Integrate from 23.47 to 23.87 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 53.00			*** METH DEFAULT ***
Q1 52.00	43.20	20.0	*** METH DEFAULT ***
Q2 51.00	17.70	20.0	*** METH DEFAULT ***

Lvl ID	Conc (ppbv)	Response
1	2.000	19894
2	not used for this compound	
3	not used for this compound	
4	not used for this compound	
5	not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear, forced through origin

-----  
-----  
6) MTBE ( )  
Ret. Time 23.79 min., Extract & Integrate from 23.59 to 23.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 57.00	7.30	10.0	*** METH DEFAULT ***
Q2 41.00	15.70	10.0	*** METH DEFAULT ***

Lvl ID	Conc (ppbv)	Response
1	0.830	19234
2	1.670	45037
3	2.500	78114
4	3.330	118736
5	5.000	213904

Qualifier Peak Analysis ON

Curve Fit: Quadratic, forced through origin

-----  
-----  
7) Hexane ( )  
Ret. Time 24.51 min., Extract & Integrate from 24.31 to 24.71 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 57.00			*** METH DEFAULT ***
Q1 56.00	61.60	20.0	*** METH DEFAULT ***
Q2 86.00	18.50	20.0	*** METH DEFAULT ***

## HP GC/MS Chemstation

Lvl ID	Conc (ppbv)	Response
1	5.000	156081
2	not used for this compound	
3	not used for this compound	
4	not used for this compound	
5	not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear, forced through origin

-----  
-----  
8) Benzene ( )  
Ret. Time 28.24 min., Extract & Integrate from 28.04 to 28.44 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
--------	-----------	----------------	-------------

Tgt	78.00		*** METH DEFAULT ***	
Q1	77.00	10.50	10.0	*** METH DEFAULT ***

Lvl ID	Conc (ppbv)	Response
1	5.000	380868
2	not used for this compound	
3	not used for this compound	
4	not used for this compound	
5	not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear, forced through origin

-----  
-----  
9) MIBK ( )  
Ret. Time 32.65 min., Extract & Integrate from 32.45 to 32.85 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
--------	-----------	----------------	-------------

Tgt	43.00		*** METH DEFAULT ***	
Q1	58.00	16.30	20.0	*** METH DEFAULT ***
Q2	85.00	7.00	20.0	*** METH DEFAULT ***
Q3	100.00	5.80	20.0	*** METH DEFAULT ***

Lvl ID	Conc (ppbv)	Response
1	5.000	241219
2	not used for this compound	
3	not used for this compound	
4	not used for this compound	
5	not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear, forced through origin

# HP GC/MS Chemstation

-----

Method Information For: C:\HPCHEM\1\METHODS\IDLE.M

## Method Sections To Run:

- ( ) Save Copy of Method With Data
- ( ) Pre-Run Cmd/Macro =
- (X) Data Acquisition
- (X) Data Analysis
- ( ) Post-Run Cmd/Macro =

## Method Comments:

This is a method for setting temperature zones to standby conditions.

END OF TOPLEVEL PARAMETERS

-----  
INSTRUMENT CONTROL PARAMETERS  
-----

Sample Inlet: GC  
Injection Source: External Device  
Injection Location: Front  
Mass Spectrometer: Enabled

=====  
HP6890 GC METHOD  
=====

## OVEN

Initial temp: 100 'C (On)      Maximum temp: 260 'C  
Initial time: 33.00 min      Equilibration time: 0.50 min  
Ramps:  
# Rate Final temp Final time CRYO (N2)  
1 0.0(Off)      Cryo: On  
Post temp: 0 'C      Cryo fault: On  
Post time: 0.00 min      Cryo timeout: 60.00 min (On)  
Run time: 33.00 min      Quick cryo cool: On  
Ambient temp: 55 'C

## FRONT INLET (HP PTV)

Mode: Split  
Initial temp: 87 'C (Off)  
Cryo: Off  
Cryo use temp: 25 'C

## BACK INLET (SPLIT/SPLITLESS)

Mode: Split  
Initial temp: 50 'C (Off)  
Pressure: 0.00 psi (Off)  
Total flow: 45.0 mL/min

## HP GC/MS Chemstation

Cryo Timeout: 30.00 min (On)      Gas saver: Off

Cryo Fault: On      Gas type: Helium

Pressure: 7.32 psi (Off)

Split ratio: 50:1

Split flow: 98.6 mL/min

Total flow: 103.6 mL/min

Gas saver: Off

Gas type: Helium

### COLUMN 1

Capillary Column

Model Number: J & W DB-624

30m x 0.32 mm i.d. and 1.8 um thick

Max temperature: 260 'C

Nominal length: 30.0 m

Nominal diameter: 320.00 um

Nominal film thickness: 1.80 um

Mode: constant pressure

Pressure: 7.32 psi

Average velocity: 52 cm/sec

Inlet: Front Inlet

Outlet: MSD

Outlet pressure: vacuum

### COLUMN 2

(not installed)

### FRONT DETECTOR (NO DET)

#### SIGNAL 1

Data rate: 20 Hz

Type: test plot

Save Data: Off

Zero: 0.0 (Off)

Range: 0

Fast Peaks: Off

Attenuation: 0

### BACK DETECTOR (NO DET)

#### SIGNAL 2

Data rate: 20 Hz

Type: test plot

Save Data: Off

Zero: 0.0 (Off)

Range: 0

Fast Peaks: Off

Attenuation: 0

### COLUMN COMP 1

(No Detectors Installed)

### COLUMN COMP 2

(No Detectors Installed)

### THERMAL AUX 2

Use: MSD Transfer Line Heater

Description: MSD

Initial temp: 280 'C (On)

Initial time: 0.00 min

# Rate Final temp Final time

# HP GC/MS Chemstation

1 0.0 (Off)

POST RUN  
Post Time: 0.00 min

## TIME TABLE

Time	Specifier	Parameter & Setpoint
------	-----------	----------------------

7673 Injector

Front Injector:  
No parameters specified

### Back Injector:

Sample Washes	0
Sample Pumps	0
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
PostInj Solvent A Washes	0
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

## MS ACQUISITION PARAMETERS

### General Information

-----

Tune File : ATUNE.U  
Acquisition Mode : Scan

### MS Information

--

Solvent Delay : 3.00 min  
EM Absolute : False  
EM Offset : 106  
Resulting EM Voltage : 2435.3

### [Scan Parameters]

Low Mass : 35  
Threshold : 150  
Sample # : 2      A/D Samples      4

### [MSZones]

## HP GC/MS Chemstation

MS Quad : 150 C maximum 200 C  
MS Source : 230 C maximum 250 C

### Timed Events

-----

[Timed MS Detector Entries]

Time (min)	State (MS on/off)
45.00	Off

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

-----  
DATA ANALYSIS PARAMETERS  
-----

Method Name: C:\HPCHEM\1\METHODS\IDLE.M

### Percent Report Settings

-----  
Sort By: Retention Time

### Output Destination

Screen: No  
Printer: Yes  
File: No

Integration Events: AutoIntegrate  
Generate Report During Run Method: No  
Signal Correlation Window: 0.020

### Qualitative Report Settings

-----  
Peak Location of Unknown: Apex  
Library to Search Minimum Quality  
DEMO.L 0

Integration Events: AutoIntegrate  
Report Type: Summary  
Output Destination  
Screen: No  
Printer: Yes

## HP GC/MS Chemstation

File: No

Generate Report During Run Method: No

### Quantitative Report Settings

-----

Report Type: Summary

Output Destination

Screen: Yes

Printer: No

File: No

Generate Report During Run Method: No

Calibration Last Updated:

Reference Window: 10.00 Percent

Non-Reference Window: 5.00 Percent

Correlation Window: 0.02 minutes

Default Multiplier: 1.00

Default Sample Concentration: 0.00

Compound Information

-----

\*\*\* Empty Quantitation Database \*\*\*

END OF DATA ANALYSIS PARAMETERS

-----

# HP GC/MS Chemstation

Method Information For: C:\HPCHEM\1\METHODS\IDLE1.M

## Method Sections To Run:

- ( ) Save Copy of Method With Data
- ( ) Pre-Run Cmd/Macro =
- (X) Data Acquisition
- (X) Data Analysis
- ( ) Post-Run Cmd/Macro =

## Method Comments:

This is a method for setting temperature zones to standby conditions.

END OF TOPLEVEL PARAMETERS

-----  
INSTRUMENT CONTROL PARAMETERS  
-----

Sample Inlet: GC  
Injection Source: External Device  
Injection Location: Front  
Mass Spectrometer: Enabled

=====  
HP6890 GC METHOD  
=====

### OVEN

Initial temp: 200 'C (On)           Maximum temp: 260 'C  
Initial time: 20.00 min           Equilibration time: 0.50 min  
Ramps:

#	Rate	Final temp	Final time	CRYO (N2)
1	0.0(Off)			Cryo: On
	Post temp:	0 'C		Cryo fault: On
	Post time:	0.00 min		Cryo timeout: 60.00 min (On)
	Run time:	20.00 min		Quick cryo cool: On
				Ambient temp: 55 'C

### FRONT INLET (HP PTV)

Mode: Split  
Initial temp: 87 'C (Off)  
Cryo: Off  
Cryo use temp: 25 'C  
Cryo Timeout: 30.00 min (On)

### BACK INLET (SPLIT/SPLITLESS)

Mode: Split  
Initial temp: 50 'C (Off)  
Pressure: 0.00 psi (Off)  
Total flow: 45.0 mL/min  
Gas saver: Off

## HP GC/MS Chemstation

Cryo Fault: On  
Pressure: 7.32 psi (Off)  
Split ratio: 50:1  
Split flow: 66.2 mL/min  
Total flow: 70.5 mL/min  
Gas saver: Off  
Gas type: Helium

Gas type: Helium

COLUMN 1	COLUMN 2
Capillary Column	not installed)
Model Number: J & W DB-624	
30m x 0.32 mm i.d. and 1.8 um thick	
Max temperature: 260 'C	
Nominal length: 30.0 m	
Nominal diameter: 320.00 um	
Nominal film thickness: 1.80 um	
Mode: constant pressure	
Pressure: 7.32 psi	
Average velocity: 45 cm/sec	
Inlet: Front Inlet	
Outlet: MSD	
Outlet pressure: vacuum	

FRONT DETECTOR (NO DET)	BACK DETECTOR (NO DET)
-------------------------	------------------------

SIGNAL 1	SIGNAL 2
Data rate: 20 Hz	Data rate: 20 Hz
Type: test plot	Type: test plot
Save Data: Off	Save Data: Off
Zero: 0.0 (Off)	Zero: 0.0 (Off)
Range: 0	Range: 0
Fast Peaks: Off	Fast Peaks: Off
Attenuation: 0	Attenuation: 0

COLUMN COMP 1	COLUMN COMP 2
(No Detectors Installed)	No Detectors Installed)

THERMAL AUX 2  
Use: MSD Transfer Line Heater  
Description: MSD  
Initial temp: 280 'C (On)  
Initial time: 0.00 min  
# Rate Final temp Final time  
1 0.0(Off)

POST RUN

HP GC/MS Chemstation

Post Time: 0.00 min

TIME TABLE

Time                    Specifier                    Parameter & Setpoint

7673 Injector

Front Injector:  
No parameters specified

Back Injector:  
Sample Washes                    0  
Sample Pumps                    0  
Injection Volume                1.0 microliters  
Syringe Size                    10.0 microliters  
PostInj Solvent A Washes        0  
PostInj Solvent B Washes        0  
Viscosity Delay                 0 seconds  
Plunger Speed                    Fast  
PreInjection Dwell               0.00 minutes  
PostInjection Dwell              0.00 minutes

**MS ACQUISITION PARAMETERS**

General Information

-----

Tune File                        : ATUNE.U  
Acquisition Mode                : Scan

MS Information

--

Solvent Delay                    : 3.00 min  
EM Absolute                      : False  
EM Offset                        : 106  
Resulting EM Voltage            : 2435.3

[Scan Parameters]

Low Mass                         : 35  
Threshold                        : 150  
Sample #                         : 2                    A/D Samples        4

[MSZones]

MS Quad                         : 150 C                maximum 200 C  
MS Source                        : 230 C                maximum 250 C

Timed Events

# HP GC/MS Chemstation

-----  
[Timed MS Detector Entries]

Time (min)	State (MS on/off)
15.00	Off

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS  
-----

DATA ANALYSIS PARAMETERS  
-----

Method Name: C:\HPCHEM\1\METHODS\IDLE1.M

Percent Report Settings  
-----

Sort By: Retention Time

Output Destination

Screen: No

Printer: Yes

File: No

Integration Events: AutoIntegrate

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings  
-----

Peak Location of Unknown: Apex

Library to Search Minimum Quality

DEMO.L 0

Integration Events: AutoIntegrate

Report Type: Summary

Output Destination

Screen: No

Printer: Yes

File: No

Generate Report During Run Method: No

Quantitative Report Settings  
-----

## HP GC/MS Chemstation

Report Type: Summary

Output Destination

Screen: Yes

Printer: No

File: No

Generate Report During Run Method: Yes

Calibration Last Updated:

Reference Window: 10.00 Percent

Non-Reference Window: 5.00 Percent

Correlation Window: 0.02 minutes

Default Multiplier: 1.00

Default Sample Concentration: 0.00

Compound Information

-----

\*\*\* Empty Quantitation Database \*\*\*

END OF DATA ANALYSIS PARAMETERS

-----

# HP GC/MS Chemstation

Sequence list  
Comment: DEC0403.s  
Operator: NRL  
Data Path: C:\HPCHEM\1\DATA\  
Pre-Seq Cmd:  
Post-Seq Cmd:

Method Sections To Run            On A Barcode Mismatch  
(X) Full Method                    (X) Inject Anyway  
( ) Reprocessing Only            ( ) Don't Inject

Line	Type	Vial	DataFile	Method	Sample Name
1	Sample	1	DE0401	IDLE1	bakeout
2	Sample	1	DE0402	MLD066	BLANK
3	Sample	2	DE0403	MLD066	AAL069919 #1054
4	Sample	3	DE0404	MLD066	CC50604A #1061 25CC
5	Sample	3	DE0405	MLD066	CC50604B #1061 50CC
6	Sample	3	DE0406	MLD066	CC50604C #1061 75CC
7	Sample	3	DE0407	MLD066	CC50604D #1061 100CC
8	Sample	3	DE0408	MLD066	CC50604E #1061 150CC
9	Sample	4	DE0409	MLD066	ALM069798 #1072
10	Sample	5	DE0410	MLD066	TX005669 BFREG
11	Sample	6	DE0411	MLD066	TX005670 BFCOL
12	Sample	7	DE0412	MLD066	TX005671 RUREG
13	Sample	8	DE0413	MLD066	TX005672 RUCOL
14	Sample	9	DE0414	MLD066	TX005673 RS
15	Sample	11	DE0415	MLD066	TX005674 CX
16	Sample	12	DE0416	MLD066	TX005675 CV
17	Sample	13	DE0417	MLD066	TX005654 BFREG
18	Sample	14	DE0418	MLD066	TX005655 BFCOL
19	Sample	10	DE0419	MLD066	XX5642 SV 25CC
20	Sample	5	DE0420	MLD066	TX005669 DUP
23	Sample	1	DE0423	IDLE	BLANK

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## Appendix V: Calibration and Control Standards

Compounds	Calibration Standard			Control Standards	
	AAL070488 ppbv	ALM069798 ppbv	CA06063 ppbv	CC50604 ppbv	AAL069919 ppbv
Acrolein	2.18	5.6	11.2		54.7
Ethanol	40.5	22.9	41.3	30.0	
Acetone	27.2	20.5	30.0	10.0	
Acetonitrile	9.3	10.0	11.0	15.0	
Acrylonitrile	6.78	5.0	3.37	2.0	
<i>t-butylmethyl ether (MTBE)</i>	4.69	4.6	5.25	5.0	
<i>Benzene</i>	6.25	5.5	6.16	5.0	

**ALM069798** - Calibration standard used from 7/01/2003 through 9/18/2003 and has been used as control standard from 9/19/2003 to the present

**CC50604 and AAL069919** - Control standards used from 7/01/2003 through 9/18/2003 and used as calibration standards from 9/19/2003 through 11/14/2004.

**AAL070488** - Calibration standard from 9/15/2004 to the present.

## Appendix VI: Revision History

Revision Number	Approval Date	Comments
1.00	July 1, 2003	Initial SOP

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