

State of California
AIR RESOURCES BOARD

Notice of Public Availability of Modified Text

Public Hearing to Consider Amending the Test Methods Designated for Determining the Benzene, Aromatic Hydrocarbon, Olefin and Sulfur Content of Phase 2 Gasoline

Public Hearing Date: October 26, 1995

Public Availability Date: December 11, 1995

Deadline for Public Comment: December 26, 1995

At a public hearing held October 26, 1995, the Air Resources Board (the "Board") considered amendments to section 2263, title 13, California Code of Regulations. The purpose of the proposed amendments was to update the test methods designated for determining the benzene, aromatic hydrocarbon, olefin and sulfur content of Phase 2, or California reformulated (CaRFG), gasoline. The amendments were developed through a cooperative effort with members of the regulated industry and are described in detail in the Staff Report released on September 8, 1995.

At the hearing the Board approved the adoption of amendments to section 2263, title 13, California Code of Regulations, with modifications to the original proposal as follows:

(1) Add language to clarify the reporting limit for all test methods with conforming changes to proposed reporting limits for the primary sulfur test method, ASTM D 2622-94, and the olefin test method, ASTM D 1319-9X.

(a) The modifications include a general statement regarding reporting limits. This statement is set out in a new note "a" to Table 1 of section 2263(b). Note "a" is intended to ensure that no values below the limit of detection (LOD), or where the test method does not specify an LOD the lower limit of the scope of the test method, may be reported. Neither the existing regulation or the amendments as originally proposed included a general provision relating to reporting limits for any of the designated test methods. Such a provision has not been necessary in the past because the concentrations of concern for all measured components of gasoline under the existing regulations are significantly above any limit of detection (LOD) or lower limit of the scope of the designated test methods. Staff discovered the need for this clarification during discussions regarding the proposed reporting limit for ASTM D 2622-94. These discussions revealed that under the averaging provisions of the CaRFG regulations the concentration of sulfur, and possibly other fuel components, could be at or below the LOD or scope of the designated test methods. Clarification of the reporting limits is necessary under these circumstances because it is not appropriate to use a test method to report concentrations below its useful range as indicated by the LOD or scope. Staff has agreed to work with industry to redefine these lower limits based on future interlaboratory testing.

(b) As proposed the amendments would have precluded reporting sulfur concentrations below 30 ppm using ASTM D 2622-94. This reporting limit was proposed due to concerns about

reproducibility of this method between 10 ppm and 30 ppm. Based on additional data submitted during the 45-day notice period, staff determined that ASTM D 2622-94 had adequate reproducibility down to 10 ppm when carried out with a modified calibration procedure and so proposed to delete the reporting limitation found in proposed note "b" to Table 1 of the regulation. New note "a" will allow reporting down to 10 ppm, the lower limit of the scope of the test method.

(c) Proposed note "f" can be deleted because it simply limited reporting under ASTM D 5453-93 to 1 ppm, the lower limit of the scope of the test method. This limit duplicates new note "a."

(2) Modify the proposed calibration procedure as shown in note "c" of Table 1. This modification reflects the method used in the most recent interlaboratory testing to show improved reproducibility at lower sulfur concentration levels. (See (1)(b) above.)

(3) Delete the bias correction requirement for ASTM D 5453-93 as set out in note "e" of Table 1. The original proposal would have allowed the use of ASTM D 5453-93 as an alternative to ASTM D 2622-94 for all sulfur concentrations. If the alternative method was to be used for gasoline with sulfur concentration of 30 ppm and above, however, the amendments required the user to establish the correlation with ASTM D 2622 to account for any bias within this range. During the 45-day comment period additional data was obtained showing that there was no bias between the two methods and therefore the correlation requirement is not necessary.

(4) Modify note "g" to extend the applicability of the reproducibility equation of ASTM D 1319-9X from 4.0 volume percent to 0.3 volume percent. Because the CaRFG regulations allow averaging and alternative gasoline formulations as compliance options, it is likely that gasoline with olefin content well below the flat limit of 4.0 volume percent will be produced. For this reason, the reproducibility of ASTM D 1319-9X must be established for these lower levels. ARB staff and industry have agreed that extrapolating the equation relating reproducibility and concentration to the lower limit of the method (0.3 volume percent) is the most practical solution until testing at the lower levels can be accomplished. The reproducibilities of aromatics and saturates in gasoline as measured by ASTM D 1319-9X has been deleted because they are not relevant to the measurement of olefin content.

In the resolution the Board also directed the Executive Officer to consider any new or additional data regarding the reproducibility, accuracy or scope of the approved test methods submitted by December 1, 1995 and to make additional modifications to the regulations based on that data, if appropriate. The Western States Petroleum Association (WSPA) timely submitted additional data regarding the reproducibility of both the primary and alternative sulfur test methods. The Executive Officer has determined that it is appropriate to modify the reproducibility values for ASTM D 2622-94 to reflect the new data. These changes are contained in note "d" to Table 1 of section 2263(b). The Executive Officer further determined that the additional data regarding ASTM D 5453-93, which was collected in an effort to determine whether a bias exists between ASTM D 2622-94 and ASTM D 5453-93 and which showed a significant drop in reproducibility

over the existing reproducibility, does not support modification of the reproducibility statement without further investigation.

In addition staff has made a number of nonsubstantive editorial and formatting changes to the amendments as originally proposed. Most notably ASTM D 5580-9X has been redesignated because the revisions to the test method that were pending at the time the proposed regulations were released were recently given final approval by ASTM, the American Society for Testing and Materials. The test method is now designated ASTM D 5580-95. (Note: ASTM D 1319-9X, which references ASTM D 1319-95 with proposed revisions currently pending will be redesignated if final approval of those revisions occurs before the final regulation is adopted. Otherwise the "9X" designation will be retained.) Additionally, new notes "e" and "g" have been added to Table 1 regarding the reproducibility statement for ASTM D 5580-95 to provide reproducibility in volume percent. This modification is necessary because the benzene and aromatic hydrocarbon specifications (13 CCR §§ 2262.3 and 2262.7) are expressed in volume percent and the reproducibility statement of ASTM D 5580-95 is expressed as mass percent.

In the resolution the Board directed the Executive Officer to adopt the amendments with modifications after making the modified regulatory language available to the public for written comment for a period of 15 days, provided that "the Executive Officer shall consider such written comments as may be submitted during this period, shall make such modifications as may be appropriate in light of the comments received, and shall present the regulations to the Board for further consideration if he determines that this is warranted."

The modified regulations and additional data submitted by WSPA are being made available by this notice for public comment prior to final action by the Executive Officer as required by Government Code section 11346.8(c) and (d). The modified text of section 2263, title 13, California Code of Regulations, is contained in Attachment A. The originally proposed amendments are shown in underline and strikeout to indicate additions and ~~deletions~~ from the existing regulation. Modifications to the original proposal are shown in shaded underline and ~~strikeout~~. The additional data submitted by WSPA is contained in Attachment B.

Written comments must be submitted to the Board Secretary, Air Resources Board, P.O. Box 2815, Sacramento, California 95812, no later than December 26, 1995, for consideration by the Executive Officer prior to final action. Only comments relating to the modifications described in this notice will be considered by the Executive Officer.

Attachments

**Attachment A
REGULATION ORDER**

Amend section 2263, title 13, California Code of Regulations, as follows:

Section 2263. Sampling Procedures and Test Methods

(a) *Sampling Procedures.* In determining compliance with the standards set forth in this subarticle 2, an applicable sampling methodology set forth in 13 C.C.R. section 2296 shall be used.

(b) *Test Methods.*

(1) In determining compliance with the standards set forth in this subarticle 2, the test methods presented in Table 1 shall be used. All identified test methods are incorporated herein by reference.

Table 1

| <i>Section</i> | <i>Gasoline Specification</i> | <i>Test Method</i> [§] |
|----------------|--|--|
| 2262.1 | Reid Vapor Pressure | ASTM D 323-58 [§] or 13 C.C.R. Section 2297 |
| 2262.2 | Sulfur Content 1 ppm to 30 ppm | ASTM D 2622-87 ASTM D 2622-94 [§] or ASTM D 5453-93 [§] |
| 2262.3 | Benzene Content | ASTM 3606-87D 5580-9X [§] or ARB MLD 116 ^b |
| 2262.4 | Olefin Content | ASTM D 1319-89-9X [§] |
| 2262.5 | Oxygen Content | ASTM D 4815-94 |
| 2262.6 | T90 and T50 | ASTM D 86-90 |
| 2262.7 | Aromatic Hydrocarbon Content | ARB MLD 116 ^b ASTM D 5580-9X [§] |

a ~~Do not report values below the limit of detection (LOD) specified in the test method. Where a test method does not specify a LOD, do not report values below the lower limit of the score of the test method.~~

~~§~~ Delete paragraph 4(b) concerning sampling.

- b Air Resources Board, Monitoring and Laboratory Division, "Procedure for the Analysis of Benzene and Other Aromatic Components of Gasoline," dated November 1991. This method is to be used instead of ASTM 3606-87 to determine benzene content if ethanol is present.

~~Results showing sulfur concentration of all types of gasoline by this method shall be reported as 0 ppm.~~

- c Make the following modifications to paragraph 9.1:

Low Level Sulfur Calibration Procedure

Reagents

Thiophene, at least 99% purity

2-Methylthiophene, at least 98% purity

Toluene, reagent grade

2,2,4 - Trimethylpentane, reagent grade

Preparation of Stock Standard

Weigh standard materials thiophene (~0.7290 gm) and 2-methylthiophene (~0.7031 gm) separately into a tared volumetric flask and record the individual mass to 0.1 mg. Add "mixed solvent" containing 25% toluene and 75% iso-octane (by volume) into the flask to a net weight of approximately 50 gm and record the weight. This "Stock Standard" contains approximately 10 mg/gm sulfur. The actual sulfur concentration can be calculated as follows:

$$\text{Sulfur from thiophene (gm)} = \text{Weight of thiophene} * 32.06 * \text{purity} / 84.14$$

$$\text{Sulfur from 2-methylthiophene (gm)} = \text{Weight of 2-methylthiophene} * 32.06 * \text{purity} / 98.17$$

$$\text{Sulfur concentration of Stock Standard (gm/gm)} = (\text{sulfur from thiophene} + \text{sulfur from 2-methylthiophene}) / \text{net weight of the stock standard}$$

Multiply the sulfur concentration by 1000 to convert the unit to mg/gm.

Preparation of Calibration Standards

Pipet 2.5 ml of the Stock Standard to 250 ml flask and dilute with the "mixed solvent" to the mark. The "Diluted Standard" contains approximately 100 mg/kg sulfur. Prepare ~~5, 10, 20, 30, 40, 50~~ ppm calibration standards by pipetting ~~5, 10, 20, 30, 40, 50~~ ml of the Diluted Standard into a 100 ml flask, respectively, and diluting with the "mixed solvent" to the mark. The actual concentration of the calibration standard should be determined from the stock standard. The standards with concentration ranging from ~~5~~ to 100 ppm and the mixed solvent are to be used for calibrating the instrument.

- d Replace ASTM D 2622-94 reproducibility values with the following:

Sulfur Content, ppm

Reproducibility

~~10 to 30~~

~~30 to 60~~

~~60 to 100~~

~~±0.5 to 1% X Sulfur Content (ppm)~~

~~±0.4 to 0.7% X Sulfur Content (ppm)~~

~~±0.3 to 0.5% X Sulfur Content (ppm)~~

As an alternative to the designated test method, ASTM D 2622-94, D 5453-93 may be used for gasoline with sulfur concentrations of 30 ppm or above provided the results from testing with D 5453-93 are correlated with ASTM D 2622-94 as modified in c. above.

Report results as sulfur content no less than 1 ppm.

The reproducibility of benzene is as follows:

$$\text{Reproducibility} = 0.1409 (X^{1.2}) \text{ where } X = \text{vol} \%$$

Add the following reproducibility statements for oxygenate-containing samples:

| | Range | Repeatability | Reproducibility |
|-----------|---------|-------------------|-------------------|
| Aromatics | 13-40 | 13 | 13 |
| Olefins | 40.1-33 | $0.258 (X)^{0.6}$ | $0.819 (X)^{0.6}$ |
| Saturates | 13-68 | 13 | 13 |

X = Volume %

The reproducibility of total aromatic hydrocarbon is as follows:

$$\text{Reproducibility} = 1.4 \text{ vol} \%$$

(c) **Equivalent Test Methods.** Whenever this section provides for the use of a specified test method, another test method may be used following a determination by the executive officer that the other method produces results equivalent to the results with the specified method.

NOTE: Authority cited: sections 39600, 39601, 43013, 43018, and 43101, Health and Safety Code; and Western Oil and Gas Ass'n. v. Orange County Air Pollution Control District, 14 Cal.3d 411, 121 Cal.Rptr. 249 (1975). Reference: sections 39000, 39001, 39002, 39003, 39010, 39500, 39515, 39516, 41511, 43000, 43016, 43018, and 43101, Health and Safety Code; and Western Oil and Gas Ass'n. v. Orange County Air Pollution Control District, 14 Cal.3d 411, 121 Cal.Rptr. 249 (1975).

Attachment B



BP OIL

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November 9, 1995

To All WSPA Sulfur Round Robin Participants,

Attached is a series of 14 pages of various information on the most recent WSPA sponsored round robin of D-2622 and D-5453 for sulfur in California Phase II Type gasoline. A preliminary report of this information was presented to the California Air Resources Board prior to the Public Hearing on October 25 and had a positive impact on the Board's actions at that meeting.

The first page is a list of the sample ID's and their contents. The next four pages is a list of the participants. This is followed by a summary of the data showing the means, reproducibility, the %R for each sample and an indication of bias between the two methods.

The next four pages are graphs of the %R vs sulfur concentrations for the ranges of 10-30 and 10-150 mg/kg. These graphs also show the calculated "best fit" line and calculated regression for each set of data. The final four sheets are the data from each laboratory. If you can not determine your labs numbers I can give those to you if you contact my by FAX or phone.

As a result of this data, CARB has accepted the equivalency of the two methods for the range of 10 mg/kg to 300 mg/kg. They agree that there is no bias! They also are now allowing the use of D-2622 in the 10-30 range where they previously were allowing only Antek.

CARB has adopted the revised calibration procedure for D-2622. A couple of labs indicated that there was no significant difference in their results but when five samples from the previous round robin were compared their concentrations dropped 1.6 mg/kg (the amount of bias seen earlier) and the % Reproducibility dropped 39%.

We are still in discussion with CARB about the MDL (Method Detection Limit) for D-2622 and what do we use as an averaging number if we find less than that level. They presently say the MDL is 10 mg/kg and when you report <10 you must use 10 for averaging.

We are also questioning the use of the D-5453 reproducibility for Phase II gasoline. Our data indicates that it should be about the same as D-2622 but the method report about 20% at 30/mg/kg. We will be requesting ASTM to rerun this in the California Phase II range (10-40 mg/kg) using gasoline type samples. We have learned that this was not done in the original work on this method.

If anyone has further questions concerning this data or the program please feel free to contact me at any time.

Sincerely,



Hal Knieriemen
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(216) 441-8131

SULFUR REPRODUCIBILITY

Proposed
Reproducibility
D-2622 & D-5453

D-2622

D-5453

RANGE, ppm

1+

10+

Reproducibility

1-10 rpm

50.70

50 (D5453 only)

10-30 ppm

43.1 (57.0)

40.5 (56.2)

42

30+ ppm

24.2 (47.4)

19.2 (27.2)

22

() Gasoline Only

Description of Gasoline Samples for D-2622 Round Robin

| <u>Sample ID #'s</u> | <u>Description & Source</u> |
|----------------------|--|
| 8594 - 96 | Blank Mixed Solvent (25% Toluene and 75% Isooctane) used for preparation of the "known" samples. |
| 8597 - 99 | About 20 mg/kg sulfur* added to the Mixed Solvent. |
| 8627 - 29 | About 140 mg/kg sulfur* added to the Mixed Solvent. |
| 8600 - 02 | California Phase II Gasoline and reported at 14.5 mg/kg sulfur from previous round robin. |
| 8603 - 05 | BP Lab Blended Gasoline reported to contain 12.9 mg/kg sulfur from previous round robin. |
| 8606 - 08 | BP Lab Blend plus 10 Vol% Ethanol. Sulfur reported at 11.2 mg/kg from previous round robin. |
| 8609 - 11 | BP Lab Blend plus 15 Vol% MTBE. |
| 8612 - 14 | About 90 mg/kg sulfur* added to the Mixed Solvent.. |
| 8615 - 17 | About 45 mg/kg sulfur* added to the Mixed Solvent. |
| 8618 - 20 | California Phase II Gasoline plus 20 mg/kg sulfur*. This was reported at 35.5 mg/kg for the first round robin. |
| 8621 - 23 | MTBE |
| 8624 - 26 | Blending ETBE |
| 8630 - 33 | 5 mg/kg sulfur* added to the Mixed Solvent. |
| 8634 - 37 | 10 mg/kg sulfur* added to the Mixed Solvent. |
| 8638 - 41 | 30 mg/kg sulfur* added to the Mixed Solvent. |

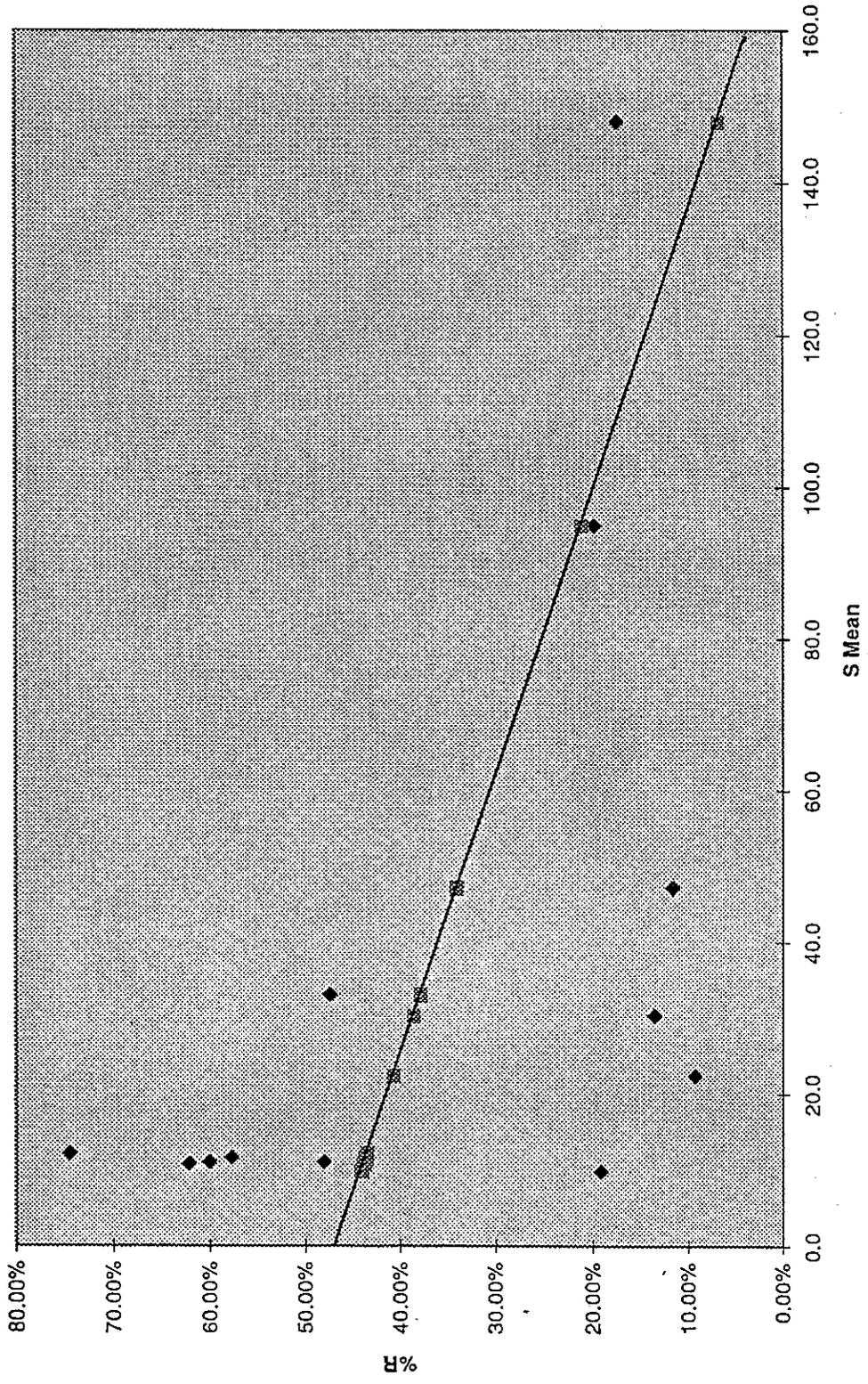
* Mixed Sulfur Standard

This was prepared by diluting 55% Thiophene and 45% 2-Methylthiophene in iso-octane to a concentration of 10.0 mg sulfur per ml. This material was then added volumetrically to weighed samples of the fuel.

Summary of Sulfur Round Robin Data
Total Data W/ Grubbs Scrub

| Comparison of Sulfur Round Robin Data | | | | | | | | | | | | |
|---|--------|-------|---------|-------|--------|---------|---------|---------------------|-----------------------|--------------------------|----------------------------|--------------------------|
| Sample # | D-2622 | | %R | Mean | D-5453 | | %R | % Bias 2622-5453 | %R Diff. 2622-5453 | %R Diff <10 2622-5453 | %R Diff 10-30 2622-5453 | %R Diff 30+ 2622-5453 |
| | Mean | R | | | R | R | | | | | | |
| 8597-8599 | 23.2 | 6.17 | 26.55% | 22.4 | 2.08 | 9.31% | 3.45% | 17.24% | | | 17.24% | |
| 8600-8599 | 12.6 | 6.22 | 49.25% | 11.0 | 6.60 | 60.06% | 12.70% | -10.81% | | | -10.81% | |
| 8603-8605 | 11.4 | 6.95 | 60.82% | 11.1 | 5.33 | 48.05% | 2.63% | 12.77% | | | 12.77% | |
| 8606-8608 | 9.9 | 4.25 | 42.82% | 11.6 | 6.69 | 57.75% | -17.17% | -14.93% | | | -14.93% | |
| 8609-8611 | 12.4 | 8.91 | 72.00% | 10.8 | 6.74 | 62.20% | 12.90% | 9.80% | | | 9.80% | |
| 8612-8614 | 93.2 | 10.33 | 11.07% | 95.0 | 18.84 | 19.83% | -1.93% | -8.76% | | | | -8.76% |
| 8615-8617 | 46.9 | 8.98 | 19.13% | 47.2 | 5.52 | 11.70% | -0.64% | 7.43% | | | | 7.43% |
| 8618-8620 | 35.5 | 9.66 | 27.21% | 33.1 | 15.66 | 47.37% | 6.76% | -20.16% | | | | -20.16% |
| 8621-8623 | 10.5 | 4.19 | 39.78% | 12.1 | 9.05 | 74.49% | -15.24% | -34.71% | | | -34.71% | |
| 8624-8626 | 3.3 | 6.05 | 185.81% | 3.5 | 3.68 | 104.81% | -6.06% | 81.00% | | | | 81.00% |
| 8627-8629 | 138.0 | 24.00 | 17.39% | 148.1 | 25.66 | 17.33% | -7.32% | 0.06% | | | | 0.06% |
| 8594-8596 | 0.0 | 0.00 | 0.00% | 0.0 | 0.00 | 0.00% | 0.00% | 0.00% | | | | |
| 8630-8633 | 5.3 | 4.31 | 81.37% | 5.2 | 2.49 | 48.02% | 1.89% | 33.35% | | | | |
| 8634-8637 | 9.9 | 2.15 | 21.70% | 9.8 | 1.87 | 19.18% | 1.01% | 2.52% | | | 2.52% | |
| 8638-8641 | 31.6 | 3.57 | 11.30% | 30.3 | 4.12 | 13.63% | 4.11% | -2.33% | | | -2.33% | |
| Overall Average | | | 44.41% | | | | | 4.83% | | | 38.12% | |
| Average <5 | | | 89.06% | | | | | -0.19% | | | -2.56% | |
| Average <10 | | | 61.91% | | | | | -1.39% | | | | |
| Average 5-10 | | | 46.42% | | | | | -3.68% | | | | |
| Average 10-30 | | | 40.53% | | | | | -7.38% | | | | |
| Average >30 | | | 19.21% | | | | | 3.10% | | | | |
| Average 5+ | | | 33.25% | | | | | 3.41% | | | | |
| Average 5+ | | | | | | | | 36.74% | | | | |
| Average 5+ | | | | | | | | 0.11% | | | | |
| Average 5+ | | | | | | | | -3.49% | | | | |
| Observations: | | | | | | | | | | | | |
| There is no significant bias between D-2622 and D-5453 at any of the levels tested above 10 mg/kg! | | | | | | | | | | | | |
| The R of D-2622 is significantly improved for the 10 - 30 mg/mg range (40.3% vs 89.5%) | | | | | | | | | | | | |
| The R for D-2622 is only about 3% higher than for D-5453 in this range but is about 20% over the ASTM published level! | | | | | | | | | | | | |
| Results of the modified D-2622 calibration procedure are encouraging! Five samples were repeated from the previous round robin and showed an average reduction of 1.6 mg/kg along with a reduction of about 39% in Reproducibility! | | | | | | | | | | | | |

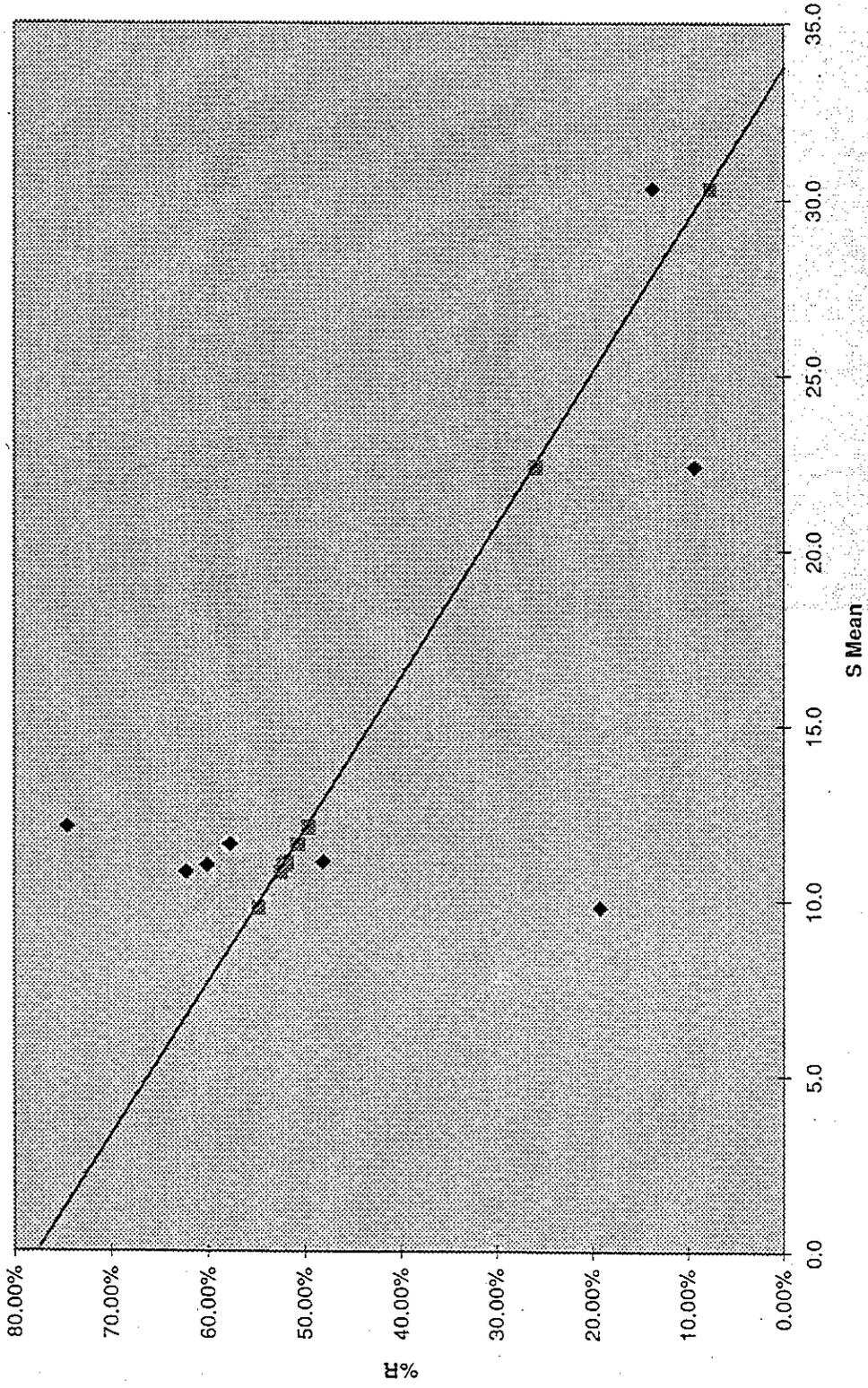
D 5453 10 - 150



%R = 0.467 - 0.0027 (S)

11/2/95

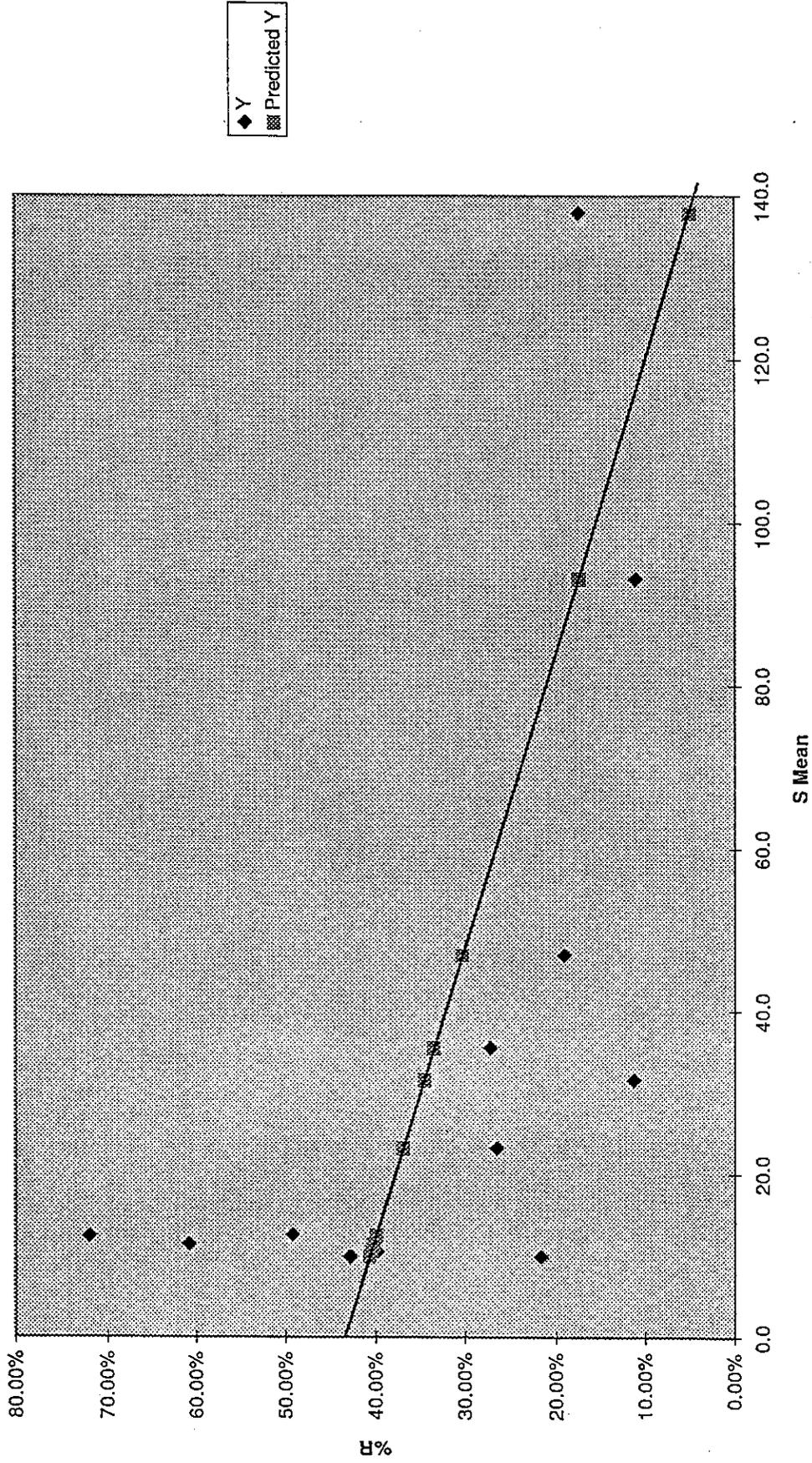
D-5453 10 - 30



%R = 0.773 - 0.023 (S)

11/2/95

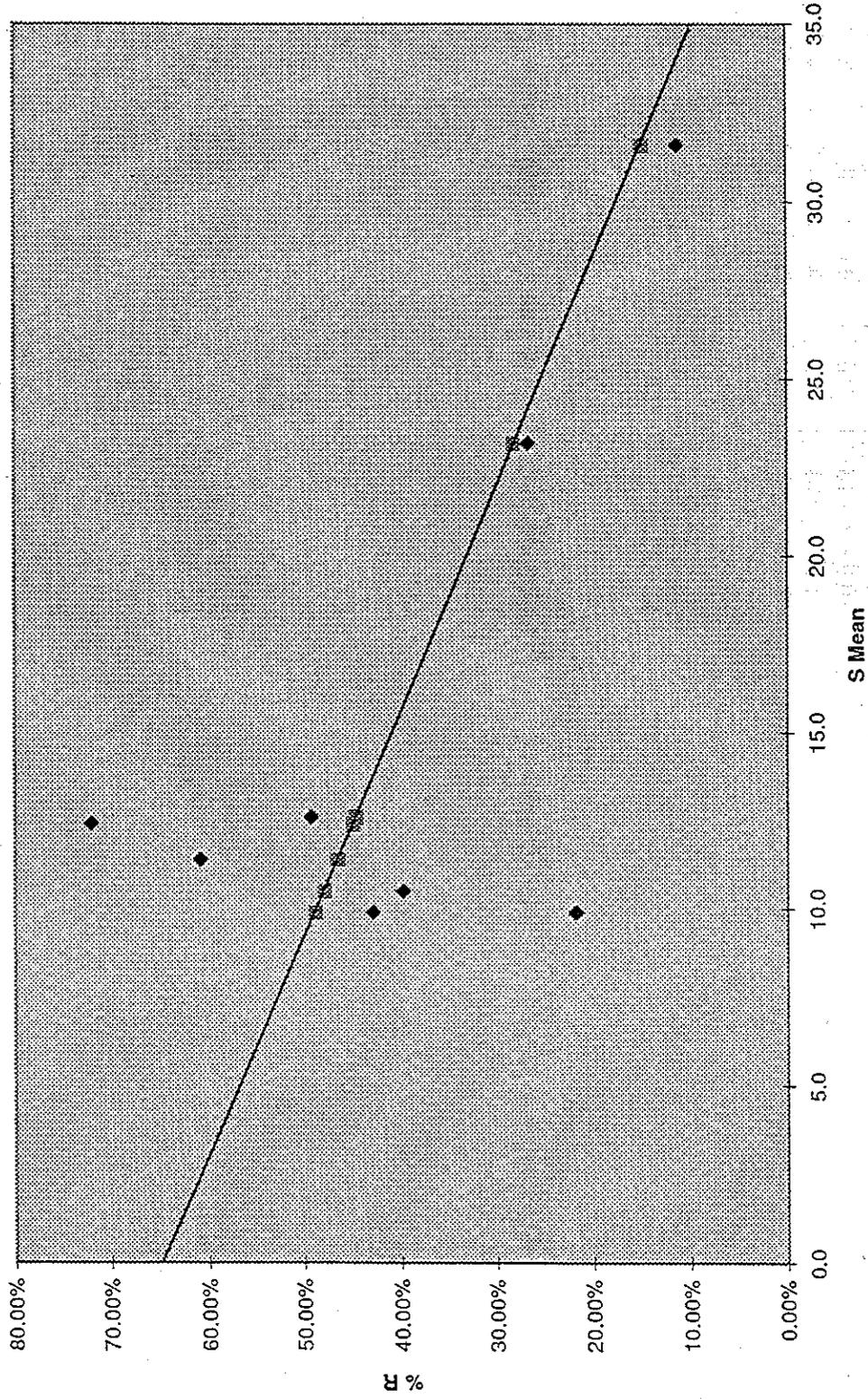
D 2622 10 - 150



%R - 0.433 - 0.0028 (S)

11/2/95

D 2622 10 - 30



%R = 0.642 - 0.0156 (S)

11/2/95

Western States Petroleum Association D-2622 Round Robin

| LAB. ID | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | | | | | | |
|--------------|-----------|--------|----------|-----------|----------|--------|----------|-----------|----------|------|-----------|--------|----------|-----------|----------|------|-----------|--------|------|-----------|--------|------|
| | 8597-8599 | 25 | 1.8 | 8600-8602 | 31* | 9 | -2.4 | 8606-8608 | 9 | -0.9 | 8609-8611 | 15 | 2.6 | 8612-8614 | 94 | 0.8 | 8615-8617 | 50 | 3.1 | 8618-8620 | 35 | -0.5 |
| A | | 20 | -3.2 | | 11 | 10 | 5.3 | | 10 | 0.1 | | 14 | 1.6 | | 92 | -1.2 | | 45 | -1.9 | | 40 | 4.5 |
| C | | 26 | 2.3 | | 14 | 17 | 2.6 | | 11 | 1.3 | | 13 | 0.9 | | 86 | -7.2 | | 44 | -3.4 | | 32 | -3.2 |
| D | | | | | 12 | 14 | 0.6 | | 9 | -0.9 | | 11 | -1.4 | | 95 | 1.8 | | 47 | 0.1 | | 34 | -1.5 |
| E | | 24 | 0.8 | | 11 | 12 | 0.6 | | 9 | -0.9 | | 9 | -3.4 | | 95 | 1.8 | | | | | 34 | -1.5 |
| F | | 27 | 3.8 | | 16 | 15 | 3.6 | | 11 | 1.1 | | 14 | 1.6 | | 99 | 5.8 | | 52 | 5.1 | | 38 | 2.5 |
| G | | 23 | -0.2 | | 12 | 12 | 0.6 | | 10 | 0.1 | | 11 | -1.4 | | 93 | -0.2 | | 48 | 1.1 | | 35 | -0.5 |
| I | | 26 | 2.8 | | 14 | 12 | 0.6 | | 12 | 2.1 | | 13 | 0.6 | | 100 | 6.8 | | 53 | 6.1 | | 40 | 4.5 |
| J | | | | | 16 | 8 | -3.4 | | 17* | | | 19 | 6.6 | | 96 | 2.8 | | 47 | 0.1 | | 40 | 4.5 |
| K | | 20 | -2.9 | | 24* | 11 | -0.6 | | 9 | -1.0 | | 10 | -2.7 | | 94 | 1.2 | | 46 | -1.2 | | 40 | 4.1 |
| L | | 22 | -0.8 | | 12 | 11 | -0.7 | | 12 | 1.7 | | 11 | -1.8 | | 93 | 0.2 | | 46 | -1.3 | | 32 | -3.1 |
| M | | 24 | 0.8 | | 14 | 11 | -0.4 | | 11 | 1.1 | | | | | 93 | -0.2 | | 48 | 1.1 | | 38 | 2.5 |
| N | | 22 | -1.5 | | 12 | 8 | -3.0 | | 9 | -0.9 | | 13 | 0.4 | | 92 | -1.0 | | 48 | 1.3 | | 32 | -3.6 |
| O | | 39.4* | | | 8 | 10 | -1.4 | | 7 | -3.3 | | 7 | -5.7 | | 87 | -6.4 | | 41 | -6.4 | | 28 | -7.3 |
| P | | 21 | -2.1 | | 10 | 9 | -2.7 | | 9 | -1.4 | | 10 | -2.9 | | 89 | -4.1 | | 44 | -3.3 | | 35 | -1.0 |
| Q | | 22 | -1.2 | | 15 | 13 | 1.6 | | 12 | 2.1 | | 17 | 4.6 | | 93 | -0.2 | | 47 | 0.1 | | 35 | -0.5 |
| T | | | | | | | | | | | | | | | | | | | | | | |
| Mean | | 23.2 | | | 12.6 | 11.4 | | | 9.9 | | | 12.4 | | | 93.2 | | | 46.9 | | | 35.5 | |
| Std. Dev. | | 2.23 | | | 2.25 | 2.51 | | | 1.53 | | | 3.22 | | | 3.73 | | | 3.24 | | | 3.49 | |
| # Ratings | | 13 | | | 14 | 15 | | | 15 | | | 15 | | | 16 | | | 15 | | | 16 | |
| # Grubbs Lir | | 5.81 | | | 5.97 | 6.77 | | | 4.14 | | | 8.68 | | | 10.25 | | | 8.75 | | | 9.59 | |
| Max | | 27 | | | 16 | 17 | | | 12.0 | | | 19.0 | | | 100.0 | | | 53.0 | | | 40.0 | |
| Min | | 20 | | | 8 | 8 | | | 6.6 | | | 6.7 | | | 86.0 | | | 40.5 | | | 28.2 | |
| Range | | 7 | | | 8 | 9 | | | 5.4 | | | 12.3 | | | 14.0 | | | 12.5 | | | 11.8 | |
| R | | 6.17 | | | 6.22 | 6.95 | | | 4.25 | | | 8.91 | | | 10.33 | | | 8.98 | | | 9.66 | |
| %R | | 26.55% | | | 49.25% | 60.82% | | | 42.82% | | | 72.00% | | | 11.07% | | | 19.13% | | | 27.21% | |

X-Ray Data with Grubbs Scrub

| Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. |
|-----------|------|-----------|-------|-----------|-------|-----------|-------|-----------|------|-----------|------|-----------|------|
| 8621-8623 | | 8624-8626 | | 8627-8629 | | 8594-8596 | | 8630-8633 | | 8634-8637 | | 8638-8641 | |
| 12 | 1.5 | 6 | 2.7 | 136 | -2.0 | 0 | 0.0 | 7 | 1.7 | 10 | 0.1 | 32 | 0.4 |
| 4* | 2.7 | 6 | 2.7 | 142 | 4.0 | 0 | 0.0 | 16* | 3.7 | 16* | 0.6 | 31 | -0.6 |
| 267* | <6* | <6* | -18.4 | 120 | -18.4 | <6* | 10.2* | 11 | 0.6 | 11 | 0.6 | 31 | -1.0 |
| 9 | -1.5 | 3 | -0.3 | | | | | | | | | | |
| 2* | 0 | 0 | -3.3 | 136 | -2.0 | 2* | 0.0 | 4 | -1.3 | 10 | 0.1 | 33 | 1.4 |
| 12 | 1.5 | 6 | 2.7 | 151 | 13.0 | 0 | 0.0 | 9 | 3.7 | 11 | 1.1 | 33 | 1.4 |
| 10 | -0.5 | 4 | 0.7 | 138 | 0.0 | 0 | 0.0 | 5 | -0.3 | 10 | 0.1 | 32 | 0.4 |
| 12 | 1.5 | 6 | 2.7 | 151 | 13.0 | 1* | 0.0 | 5 | -0.3 | 12* | 0.1 | 33 | 1.4 |
| 13 | 2.5 | 2 | -1.3 | | | | | 5 | -0.3 | 9 | -0.9 | 30 | -1.6 |
| 8 | -2.6 | 1 | -2.7 | 139 | 0.9 | 0 | 0.0 | 3 | -2.0 | 10 | 0.0 | 30 | -1.7 |
| 9 | -1.5 | 1 | -1.9 | 142 | 3.7 | 0 | 0.0 | 5 | -0.3 | 10 | 0.4 | 30 | -1.5 |
| 11 | 0.5 | 4 | 0.7 | 135 | -3.0 | 0 | 0.0 | 4 | -1.3 | 10 | 0.1 | 31 | -0.6 |
| 10 | -0.1 | 2 | -0.9 | 141 | 3.3 | 0 | 0.0 | 4 | -1.1 | 8 | 0.1 | 31 | -0.9 |
| | | 0 | -3.3 | 127 | -11.0 | 0 | 0.0 | 6 | 0.8 | 10 | 0.1 | 33 | 1.8 |
| 10 | -0.4 | 4 | 1.1 | 130 | -8.4 | 0.4* | 0.4* | 6 | 0.7 | 10 | 0.1 | 33 | 1.4 |
| 10 | -0.5 | 3 | -0.3 | 145 | 7.0 | 2* | 2* | | | | | | |
| 10.5 | | 3.3 | | 138.0 | | 0.0 | 0.0 | 5.3 | | 9.9 | | 31.6 | |
| 1.51 | | 2.18 | | 8.66 | | 0.00 | 0.00 | 1.56 | | 0.77 | | 1.29 | |
| 12 | | 15 | | 14 | | 10 | 10 | 12 | | 12 | | 14 | |
| 3.86 | | 5.89 | | 23.04 | | 0.00 | 0.00 | 3.97 | | 1.97 | | 3.43 | |
| 13.0 | | 6.0 | | 151.0 | | 0.0 | 0.0 | 9.0 | | 11.0 | | 33.4 | |
| 7.9 | | 0.0 | | 119.6 | | 0.0 | 0.0 | 3.3 | | 7.9 | | 29.9 | |
| 5.1 | | 6.0 | | 31.4 | | 0.0 | 0.0 | 5.7 | | 3.1 | | 3.5 | |
| 4.19 | | 6.05 | | 24.00 | | 0.00 | 0.00 | 4.31 | | 2.15 | | 3.57 | |
| 39.78% | | 185.81% | | 17.39% | | 0.00% | 0.00% | 81.37% | | 21.70% | | 11.30% | |

**Western States Petroleum Association
D-5453 Round Robin**

| LAB. ID | Sample # | Dev. |
|---------|-----------|------|-----------|------|-----------|------|-----------|------|-----------|------|-----------|------|-----------|------|-----------|------|
| | 8597-8599 | | 8600-8602 | | 8603-8605 | | 8606-8608 | | 8609-8611 | | 8612-8614 | | 8615-8617 | | 8618-8620 | |
| J | 23.0 | 0.6 | 12.0 | 1.0 | 12.0 | 0.9 | 12.0 | 0.4 | 11.0 | 0.2 | 102.0 | 7.0 | 49.0 | 1.8 | 34.0 | 0.9 |
| N | 21.0 | -1.4 | 14.2 | 3.2 | 14.5 | 3.4 | 16.0 | 4.4 | 15.1 | 4.3 | 95.1 | 0.1 | 45.7 | -1.5 | 42.7 | 9.6 |
| O | 22.0 | -0.4 | 9.1 | -1.9 | 9.3 | -1.8 | 10.0 | -1.6 | 10.0 | -0.8 | 91.0 | -4.0 | 47.0 | -0.2 | 27.0 | -6.1 |
| P | 22.6 | 0.2 | 11.8 | 0.8 | 9.4 | -1.7 | 10.6 | -1.0 | 10.5 | -0.3 | 97.3 | 2.3 | 48.5 | 1.3 | 35.1 | 2.0 |
| S | 12.3* | | 7.8 | -3.2 | 9.5 | -1.6 | 8.5 | -3.1 | 9.1 | -1.7 | 104.5 | 9.5 | 50.5 | 3.3 | 35.7 | 2.6 |
| W | 23.0 | 0.6 | 8.0 | -3.0 | 10.0 | -1.1 | | | 7.0 | -3.8 | | | 45.0 | -2.2 | 24.0 | -9.1 |
| X | 22.0 | -0.4 | 12.0 | 1.0 | 11.0 | -0.1 | 11.0 | -0.6 | 11.0 | 0.2 | 89.0 | -6.0 | 45.0 | -2.2 | 33.0 | -0.1 |
| Z | 23.0 | 0.6 | 13.0 | 2.0 | 13.0 | 1.9 | 13.0 | 1.4 | 13.0 | 2.2 | 86.0 | -9.0 | 47.0 | -0.2 | 33.0 | -0.1 |

| | | | | | | | | | | | | | | | | |
|------------|-------|--|--------|--|--------|--|--------|--|--------|--|--------|--|--------|--|--------|--|
| Mean | 22.4 | | 11.0 | | 11.1 | | 11.6 | | 10.8 | | 95.0 | | 47.2 | | 33.1 | |
| Std. Dev. | 0.75 | | 2.38 | | 1.92 | | 2.42 | | 2.43 | | 6.80 | | 1.99 | | 5.65 | |
| # Ratings | 7 | | 8 | | 8 | | 7 | | 8 | | 7 | | 8 | | 8 | |
| Grubbs Lir | 1.58 | | 5.29 | | 4.27 | | 5.07 | | 5.40 | | 14.28 | | 4.43 | | 12.55 | |
| Max | 23 | | 14 | | 15 | | 16.0 | | 15.1 | | 104.5 | | 50.5 | | 42.7 | |
| Min | 21 | | 8 | | 9 | | 8.5 | | 7.0 | | 86.0 | | 45.0 | | 24.0 | |
| Range | 2 | | 6 | | 5 | | 7.5 | | 8.1 | | 18.5 | | 5.5 | | 18.7 | |
| R | 2.08 | | 6.60 | | 5.33 | | 6.69 | | 6.74 | | 18.84 | | 5.52 | | 15.66 | |
| %R | 9.31% | | 60.06% | | 48.05% | | 57.75% | | 62.20% | | 19.83% | | 11.70% | | 47.37% | |

Antek Data with Grubbs Scrub

| Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | Sample # | Dev. | | | | | | | |
|-----------|--------|----------|-----------|----------|------|-----------|--------|----------|-----------|----------|--------|-----------|--------|------|-----------|--------|------|-----------|--------|------|
| 3621-8623 | 11.0 | -1.1 | 8624-8626 | 4.0 | 0.5 | 8627-8629 | 156.0 | 7.9 | 8594-8596 | 0.0 | 0.0 | 8630-8633 | 6.0 | 0.8 | 8634-8637 | 9.0 | -0.8 | 8638-8641 | 31.0 | 0.8 |
| | | | | 4.1 | 0.6 | | 145.6 | -2.5 | | <2 | <10 | | 4.0 | -1.2 | | 11.0 | 1.2 | | 28.0 | -2.3 |
| | 14.0 | 1.9 | | <10 | | | 149.0 | 0.9 | | <10 | | | <10 | | 9.4 | -0.4 | | 31.0 | 0.8 | |
| | 12.4 | 0.3 | | 4.0 | 0.5 | | 147.4 | -0.7 | | 1.7* | 5.3 | | 5.3 | 0.1 | | 10.2 | 0.4 | | 31.0 | 0.8 |
| | 5.6 | -6.5 | | 2.5 | -1.0 | | 160.4 | 12.3 | | 0.0 | 4.0 | | 4.0 | -1.2 | | 9.0 | -0.8 | | 32.0 | 1.8 |
| | 15.0 | 2.9 | | 1.0 | -2.5 | | 147.0 | -1.1 | | 0.4* | 5.0 | | 5.0 | -0.2 | | 9.5 | -0.3 | | 28.0 | -2.3 |
| | 12.0 | -0.1 | | 4.0 | 0.5 | | | | | 0.0 | 6.0 | | 6.0 | 0.8 | | 10.0 | 0.2 | | 31.0 | 0.8 |
| | 15.0 | 2.9 | | 5.0 | 1.5 | | 131.0 | -17.1 | | <3 | 6.0 | | 6.0 | 0.8 | | 10.0 | 0.2 | | 30.0 | -0.3 |
| | 12.1 | | | 3.5 | | | 148.1 | | | 0.0 | 5.2 | | 5.2 | | | 9.8 | | | 30.3 | |
| | 3.27 | | | 1.33 | | | 9.26 | | | 0.00 | 0.90 | | 0.90 | | | 0.68 | | | 1.49 | |
| | | | | | | | | | | 6 | 7 | | 7 | | | 8 | | | | |
| | 6.86 | | | 2.79 | | | 19.45 | | | 0.00 | 1.89 | | 1.89 | | | 1.50 | | | 3.30 | |
| | 15.0 | | | 5.0 | | | 160.4 | | | 0.0 | 6.0 | | 6.0 | | | 11.0 | | | 32.0 | |
| | 5.6 | | | 1.0 | | | 131.0 | | | 0.0 | 4.0 | | 4.0 | | | 9.0 | | | 28.0 | |
| | 9.4 | | | 4.0 | | | 29.4 | | | 0.0 | 2.0 | | 2.0 | | | 2.0 | | | 4.0 | |
| | 9.05 | | | 3.68 | | | 25.66 | | | 0.00 | 2.49 | | 2.49 | | | 1.87 | | | 4.12 | |
| | 74.49% | | | 104.81% | | | 17.33% | | | 0.00% | 48.02% | | 48.02% | | | 19.18% | | | 13.63% | |