EOD TEST PROCEDURE

TP 109C

Title	Page Number
Test for Lead in Gasoline by Atomic Absorption Spectrometry	1 of 26
Originator	**Supersedes**
Carl A. Scarbro	TP 109B
Responsible Organization Engineering Operations Division (EOD) Fuels and Chemical Analysis Branch (FCAB)	**Computer Program** TP 109 Data Base
Type of Test Report Data Form and Computer Report	**Data Form Number** Forms 109-01, 109-02, and 109-03
Report Distribution Project Officer and Chem. Lab File	**Implementation Date** 09-30-94

Implementation Approval

Original Test Procedure Authorized by EPCN #85 on 07-30-93

Revision Description

(1) 09-30-94 The purpose of this change is to revise the procedure as described in EPCN #170.

Note: Specific brand names in EPA/EOD procedures are for reference only and are not an endorsement of those products.

Table of Contents

Attachments

1. Purpose

This method describes the determination of total lead in gasoline. It is the EOD specific version of American Society for Testing and Material's (ASTM) D 3237 and the "Code of Federal Regulation's," Part 80, Appendix B, Method 1, Standard Test For Lead in Gasoline by Atomic Absorption Spectrometry.

The method is suitable for the determination of lead in gasoline in concentrations from 0.010 to 0.100 g Pb/U.S. gallon, independent of lead alkyl type.

2. Test Article Description

Gasoline sample no less than 10 mL in volume

3. References

3.1 ASTM Standard Method D 3237, Test for Lead in Gasoline by Atomic Absorption Spectrometry

3.2 40 CFR Part 80, Appendix B, Method 1, Standard Test For Lead in Gasoline by Atomic Absorption Spectrometry

3.3 ASTM Standard Practice E-177-86, Standard Practice for use of the Terms Precision and Bias in ASTM Test Methods.

3.4 ASTM Manual MNL7, Manual on Presentation of Data and Control Chart Analysis: 6th Edition

3.5 "Operator's Manual for Perkin-Elmer Atomic Absorption Spectrometer Model 306"

3.6 "FileMaker Pro User's Guide," 1990 Edition

4. Required Equipment

4.1 Atomic absorption (AA) spectrophotometer, with 4-inch burner slot fueled by compressed air and acetylene, scale expansion in the absorbency mode, and signal dampening

4.2 Fume hood for wet chemistry preparation of samples

4.3 Fume extractor for AA flame emissions

4.4 Glassware

4.4.1 Class A volumetric pipette with pipette aspirator filling devices

4.4.2 Class A volumetric flasks

4.5 0.10-mL auto-micro-pipettes with an accuracy of ±0.6 percent

4.6 Auto-dispensers for reagents with an accuracy of ±2.0 percent

4.7 Reagents (“reagent grade” or better, or as otherwise specified)

4.7.1 Atomic Absorption Grade Acetylene

4.7.2 Burner air substantially similar to the specification in 40 CFR 86.114 (a) (5)

4.7.3 Methyl isobutyl ketone (MIBK) [108-01]

4.7.4 Toluene [108-88-3]

4.7.5 Iodine [7553-56-2]

4.7.6 Lead Chloride [7758-95-4]

4.7.7 Tricapryl methyl ammonium chloride (Aliquot 336) [6243-39-6]

4.7.8 Lead-sterile gasoline; gasoline containing less than 0.005 g Pb/U.S. gallon and being substantially similar to the emission certification test gasoline specified in 40 CFR 86.113

4.8 Quality Control Standards, such as an NIST Standard Reference Material or other independently prepared sample of known lead concentration.

4.9 Fuels Field Inspection, EPA Form 3500-5 (Attachment A)

4.10 Control Chart for the Range of Duplicate Analyses, Form 109-01 (Attachment B)

4.11 Control Chart for Individual Observations, Form 109-02 (Attachment C)

4.12 Chain of Custody, Form 109-03 (Attachment D)

5. Precautions

5.1 Acetylene, Aliquot 336, gasoline, iodine, lead chloride, MIBK, and toluene are hazardous materials. Specific procedures for handling these materials are listed in their respective Material Safety Data Sheets (MSDS) on file in FCAB.

The analyst must be familiar with these MSDS procedures prior to performing this procedure.

5.2 The AA burner is an open flame.

The fume extractor must be on whenever the burner is lit.

Extreme caution must be taken when handling flammable materials and test samples near the open flame.

5.3 In case of a spill of any of the above reagents or samples, remove all personnel from the area, contact the Emergency Response Team and, if possible, turn the AA's fuel and air supply off.

Spills of a gallon or less can be mopped up with spill control pillows or absorbent found near the AA.

5.4 Under no circumstances should personnel pipette by mouth. Pipette aspirators, auto pipets, or auto dispensers must be used.

5.5 Samples, standards, reagents, and glassware must be at the same temperature when mixed to ensure volumetric accuracy.

5.6 All glassware must be clean and dry before use.

5.7 Before initial use, all auto-micro-pipette and auto-dispensers must be checked for volumetric accuracy with the appropriate class A volumetric glassware.

6. Visual Inspection

6.1 Samples are inspected before analysis for phase separation, leaks, and tampering.

6.2 Before analysis, AA fuel and burner air lines are inspected for leaks with soap solution.

6.3 The nebulizer drain tube is inspected before lighting the burner.

6.4 Glassware is inspected for contamination before use.

7. Test Article Preparation

7.1 Check the acetylene tank supply pressure. It must be greater than 100 psi to proceed.

7.2 Inspect all glassware for cleanliness prior to use. Glassware showing contamination of interior surfaces must not be used.

7.3 Inspect the nebulizer drain tube. It must drain into an MIBK-tolerant container with a secure cap.

The tube must have a loop trap of 15 cm (6 inches) in diameter, filled with water, to act as a vapor barrier between the nebulizer and the container.

The tube's end must be submerged below the surface of the water-waste mix in the container.

7.4 Install a lead hollow cathode ray lamp into the instrument's lamp fixture. Turn the instrument on.

7.5 AA settings vary from instrument to instrument. Perform the following adjustments on the Perkin-Elmer 306:

ADJUSTMENT	SETTING
POWER	ON
FILTER	OFF
PHASE	NORMAL
CHOPPER	OFF
SIGNAL	TC4
SLIT	4
FUNCTION	ABS
RANGE	UV
MODE	10X
MONOCHROMATOR	~ 283.5 nm
SOURCE	installed lamp rating
GAIN	energy meter in the green

7.6 After the lamp has been warmed up for at least 30 minutes, align the lamp with the lamp alignment screws to maximize the gain indicated on the energy meter.

Then readjust the gain such that the needle on the energy meter reads in the green area.

7.7 If a vertical alignment has not been done, raise the nebulizer with the flame off and the source on until the absorbency meter indicates absorbency.

Then lower the nebulizer to where no absorbency occurs. Further alignment of the burner must be done during the aspiration of a calibration standard.

7.8 Prepare the iodine solution. Dissolve and dilute 3 g of iodine crystals with toluene to 100 mL.

7.9 Prepare the Aliquot 336 in MIBK solutions.

7.9.1 Dissolve and dilute 100 mL (88.0 g) of Aliquot 336 with MIBK to 1 L. This makes the 10 volume percent Aliquot 336 in MIBK solution.

7.9.2 Dissolve and dilute 10 mL (8.8 g) of Aliquot 336 with MIBK to 1 L. This makes the 1 volume percent Aliquot 336 in MIBK solution.

7.10 Prepare the lead standards.

7.10.1 Transfer 0.4433 g of lead chloride (previously dried at 105 °C for a minimum of 3 hours) to a 250-mL volumetric flask and dissolve in approximately 200 mL of 10 percent Aliquot 336 in MIBK solution.

Dilute to volume with 10 percent Aliquot 336 in MIBK solution, mix well, and store in a brown polyethylene bottle. This is the 5.0 g Pb/U.S. gallon standard.

7.10.2 Pipette 50 mL of the 5.0 g Pb/U.S. gallon standard into a 250-mL volumetric flask, dilute to volume with the 1 percent Aliquot 336 in MIBK solution, and store in a brown polyethylene bottle.

This is the 1.0 g Pb/U.S. gallon standard.

7.10.3 Pipette 2, 5, and 10 mL of the 1.0 g Pb/U.S. gallon standard into 100-mL volumetric flasks.

Add 5 mL of 1 percent Aliquot 336 in MIBK solution to each flask, dilute to volume with MIBK, and store in a brown polyethylene bottle.

These are the 0.02, 0.05, and 0.10 g Pb/U.S. gallon standards.

7.11 Prepare the three calibration standards and one blank.

To each of four 50-mL volumetric flasks, add approximately 30 mL of MIBK with the auto dispenser.

With a class A volumetric pipette, add 5 mL of the 0.02, 0.05, and 0.10 g Pb/U.S. gallon to three separate 50-mL volumetric flasks.

Add 5 mL of the lead-free gasoline into each of the three flasks. For the blank, add only 5 mL of the lead-free gasoline.

Mix well. Add 100 µL of the iodine/toluene solution and mix well by shaking for a minimum of 1 minute.

Add 5 mL of the 1 percent Aliquot 336 in MIBK solution and mix well.

Dilute to volume with MIBK and mix well.

7.12 Obtain and inspect samples as described in the FCAB Enforcement Sample Chain-of-Custody Procedure (Attachment E).

7.13 Prepare the samples and Quality Control Standards.

In a 50-mL volumetric flask, add approximately 30 mL of MIBK with the auto-dispenser.

With a class A volumetric pipette, add 5 mL of the sample or standard into the flask and mix well.

Add 100 µL of the iodine/toluene solution and mix well by shaking for a minimum of 1 minute.

Add 5 mL of the 1 percent Aliquot 336 in MIBK solution and mix well.

Dilute to volume with MIBK and mix well.

7.14 Plan the analysis run.

7.14.1 Provide for at least one laboratory duplicate.

If the number of samples exceeds 20, then there must be at least one laboratory duplicate analyzed after every 20 samples.

7.14.2 The 0.100 g Pb/U.S. gallon calibration standard must be analyzed after every 10 analyses.

7.14.3 At least one Quality Control Standard must be analyzed for each calibration.

8. Test Procedure

100 Turn on the computer and verify that its clock settings for date and time are accurate.
Install the floppy disk named “TP109” from the disk file in Room 405.

Open the file named “TP 109 Data Base.” The input format (Attachment F) will automatically come up on the screen.

Reset the automatic data input for the field “Analyst” to the analyst’s initials.

101 Light the AA burner.

Warning: Always turn on the air first, then fuel, before lighting.

With the instrument prepared for use as described in Steps 7.3 through 7.5, adjust the burner fuel and air flow rates while aspirating blank solution until an oxidizing, blue flame is produced.

While the blank is being aspirated, hold down the "AUTO ZERO" button until the readout displays zero.

102 Aspirate the 0.100 g Pb/U.S. gallon standard.

If it has not been done, align the burner head with the vertical, horizontal, and lateral adjustment screws until a maximum response is achieved.

Adjust the nebulizer sample intake until maximum response is achieved.

The response must be greater than 0.050 absorbance units.

If this minimum is not achieved, realign the burner head or check the aspirator tube for blockage.

If these inspections fail to identify the problem, replace the hollow cathode lamp with a spare.

If that does not increase the response, refer to the instrument manual.

103 Re-aspirate a blank and hold down the "AUTO ZERO" button until the readout displays zero.

104 Turn the Mode control to "CONC" and adjust the readout to 0.100 with the "Concentration" and "Decimal Point" dial.

Lock the concentration control setting by turning the locking ring to the right.

Reset the Signal to "INT10" and turn the Mode to "AUTO CONC."

105 Calibrate the instrument with the 0.100 g Pb/U.S. gallon standard.

While aspirating the standard, press the "AUTO CONC" button and hold until the "READ" button lights again.

Check the zero by aspirating a blank and pressing the "READ" button.

Read a blank after each analysis. All blanks must be within 0.002 g Pb/U.S. gallon of zero. Blank measurement results are not entered into the TP 109 Data Base.

After reading the blank, the operator may re-zero the instrument using the "AUTO ZERO" button.

106 Check the linearity of the calibration by analyzing the 0.100, 0.050, and 0.020 g Pb/U.S. gallon calibration standards.

Enter the sample ID number and the instrument reading into the data base.

All calibration standards must be within 0.002 g Pb/U.S. gallon of their nominal values.

If they are not within that limit, determine if the error is caused by cross-contamination, an improperly prepared standard, or changes in the operating condition of the AA.

Proceed when the calibration is acceptable.

107 Analyze the Quality Control Standard in the same manner as the calibration standards. In addition, enter the dilution ratio into the data base if the ratio is other than 1.

Record the date and the result on the Form 109-02 specific to that standard.

Calculate the moving range and plot both the result and the moving range values. Review the control chart for out-of-control indications.

Out-of-control indications require immediate corrective action. Investigate any shift in the average bias.

Record the results of the investigation on the control chart and in the data base.

108 Run each sample in the same manner as the Quality Control Standard.

109 Samples exceeding the legal limit for lead in unleaded gasoline of 0.050 g Pb/U.S. gallon must be analyzed as laboratory duplicates.

Record laboratory duplicate values, as they become available, on Form 109-01.

Calculate and plot the range.

Review the control chart for out-of-control indications. Out-of-control indications require immediate corrective action.

Investigate any causes of excessive variability. After investigation, duplicate the initial sample and another sample to reestablish process control.

Record the results of the investigation on the control chart and in the data base.

110 Samples reading over 0.100 g Pb/U.S. gallon must be diluted with lead sterile gasoline and reanalyzed.

A dilution ratio is obtained by starting with the initial reading. Multiply the reading by 10.
The result and two times the result will give a range of acceptable dilution ratios. Pick a dilution ratio in the range that can be done conveniently using available glassware.

The diluted concentration will be between 0.050 and 0.100 g Pb/U.S. gallon.

111 After running 10 samples, and as the last analysis of the session, run the 0.100 gPb/U.S. gallon standard.

The response must be within 0.003 g Pb/U.S. gallon of the nominal value of that standard.

If not, the analyzer will be calibrated by repeating Steps 105 through 107. Samples run since the last within-tolerance 0.100 standard reading must be reanalyzed.

112 Upon the completion of the final blank solution measurement review the data entered in the TP 109 Data Base for reasonableness, completeness, and conformance to the acceptance criteria. Take corrective action as needed.

113 Aspirate 50 mL of acetone and then 25 mL of de-ionized water.

114 Turn off the power to the hollow cathode tube.

115 Discontinue the aspiration of de-ionized water and turn off the acetylene source at the cylinder. After the acetylene flow comes to zero, close the acetylene source toggle switch on the Burner Control.

With air still flowing, clean the burner head slot of any deposits on its surface with a single-edge razor blade.

Turn the air off at the flow controller.

Turn off the power to the AA.

116 Quit the FileMaker application and eject the disk. Transfer the floppy disk to the "Chem Lab I" computer in Room 405.

Check the hard drive icon on the screen to verify the computer ID.

Copy the file to the hard drive, then open the file and print the report.

Quit the FileMaker application; eject the disk and return it to the disk file.

117 Transcribe the sample test results from the TP 109 Data Base Report to EPA Form 3500-5. Initial and date the form as close to the data entry as possible.

118 Sign and date the TP 109 Data Base Report.

9. Data Input

9.1 The test date, analyst's initials, time, and a dilution ratio of one are automatically entered when a new record is created.

The analyst enters the Sample ID, the dilution ratio (if greater than one), the instrument reading, and applicable comments into the TP 109 Data Base at the time of analysis.

9.2 Laboratory duplicate results are recorded on Form 109-01. Individual control charts (Form 109-02) are used to record Quality Control Standard results.

10. Data Analysis

10.1 The analyst reviews the TP 109 Data Base Report data for reasonableness, completeness, and conformance to the acceptance criteria.

10.2 A verifying technician compares EPA Form 3500-5 data with the corresponding TP 109 Data Base Report to identify any transcription errors.

10.3 The verifying technician reviews the TP 109 Data Base Report to confirm compliance with the acceptance criteria.

10.4 The verifying technician signs and dates the TP 109 Data Base Report, indicating the validation has been completed.

11. Data Output

The completed EPA Form 3500-5, the TP 109 Data Base Report, and Control Chart Forms 109-01 and 109-02 are filed according to Attachment E.

12. Acceptance Criteria

12.1 The 0.100 g Pb/U.S. gallon standard response must exceed 0.050 absorbance.

12.2 All blanks must read within 0.002 g Pb/U.S. gallon of zero.

12.3 All calibration standards (0.020, 0.050, and 0.100 g Pb/U.S. gallon) must be named within 0.002 g Pb/U.S. gallon of their nominal values.

12.4 Any sample that exceeds 0.050 g Pb/U.S. gallon must be analyzed in duplicate. However, it is not required that it be analyzed under the same calibration.

12.5 The 0.100 g Pb/U.S. gallon standard must be analyzed every 10 analyses and as the last analysis of the session. The response must be within 0.003 g Pb/U.S. gallon of the standard value.

If not, an investigation for cause must be conducted and documented on the control chart and in the data base. The analyzer must be calibrated and samples run since the last within-tolerance 0.100 standard reading must be reanalyzed.

12.6 A Quality Control Standard must be analyzed for every calibration. Individual control charts (Form 109-02) are used to record the results for each standard.

Out-of-control indications must be investigated for cause.

Investigation results must be documented on the control chart and in the data base.

12.7 All samples that exceed 0.100 g Pb/U.S. gallon must be diluted with lead sterile gasoline so that the diluted sample concentration is between 0.050 and 0.100 g Pb/ U.S. gallon.

12.8 Laboratory duplicate range values must be within the control limits or an investigation for cause must be performed and reported. The results of any investigation are documented on the control chart and the data base.

Reestablishment of process control must be demonstrated before further analyses can be performed

13. Quality Provisions

13.1 Laboratory correlation, precision, and accuracy statistics will be reported in an annual quality assurance report.

13.2 Repeatability for this procedure has been statistically determined using 1985 and 1986 FCAB test data.

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the normal and current operations of the test method, exceed the following values only in 1 case out of 20:

±0.002 g Pb/U.S. gallon

13.3 The accuracy of this method was determined with NIST standards analyzed by FCAB during 1985 and 1986. The accuracy expressed as a 95 percent probability interval for the percent recovery of an individual sample was found to be:

97.8 percent ±6.5 percent

13.4 The lower limit of detection has been calculated through the successive analyses of method-detection-limit standards by FCAB testing in 1985 and 1986 to be:

0.002 g Pb/U.S. gallon

13.5 The results of laboratory duplicates and quality control standard measurements are analyzed using statistical process control methods.

13.6 Validation of all analysis data is done by an independent technician.

13.7 Analysis samples sent to other facilities are sealed with a new Official Sample Seal, EPA Form 7500-2 (Attachment G), and are shipped with Form 109-03 to assure sample integrity.

EPA

U.S. Environmental Protection Agency
Washington, DC 20460

Fuels Field Inspection

Local RVP _____

Check here if QA sample taken ☐

Inspection Number 1033402

Inspection Date (mm-dd-yy)	Time In: (military)	Time Out:	Type of Inspection ☐ Random ☐ Directed	Inspector Code

Facility Type (mark only one): ☐ Refiner ☐ Importer ☐ Distributor ☐ Ethanol Blender ☐ Reseller ☐ Carrier ☐ Retailer ☐ Wholesale P-C

Equipment SIN	Nozzle Gauge	Lead Screening Kit	Volatility Screening Instrument	Field Tests Conducted By

1. Facility Information

Facility Name	Corporate, Trade or Brand Name	Owner's Telephone No. (inc. area code)

Street	City	State	ZIP

County	Facility Owner	Lessee (if leased)

Company Contact [title]	Escort [title]

2. Fuel Description and Use Information

Grade	Tank	Posted % ETOH	Posted Octane	Self Serve Price	per	Full Serve Price	per	Intended Use Begin Date	End Date	Areas in Use
		.	.	.	.	.	.	.	.	.
		.	.	.	.	.	.	.	.	.
		.	.	.	.	.	.	.	.	.
		.	.	.	.	.	.	.	.	.
		.	.	.	.	.	.	.	.	.
		.	.	.	.	.	.	.	.	.

3. Sample Information and Field Violations

Sample #	Grade	Tank #	Pump #	Sample Site	Sample Type	Fuel Volume	Pump, Tank or Vehicle SIN	Violation Code
1						.		
2						.		
3						.		
4						.		
5						.		
6						.		
7						.		
8						.		
9						.		
10						.		

4. Test Results

Sample #	Test Type	Test #	Test Result

Number of Nozzles _____ %EtOH Posted? Yes ☐ No ☐

Total Monthly Throughput (gallons) _____

5. Comments

SAMPLE

RVP STANDARD _____
CHAIN OF CUSTODY RECORD

Sample numbers: _____
Date shipped to lab: _____
Seals OK? ___ _____
Signature

Sample numbers: _____
Date received by lab: _____
Laboratory location: _____
Seals OK? ___ _____
Signature

Person Receiving Copy of Form	Inspector's Name (print)	Inspector's Signature

EPA Form 3500-5 (Rev. 3/92); Previous versions are usable.

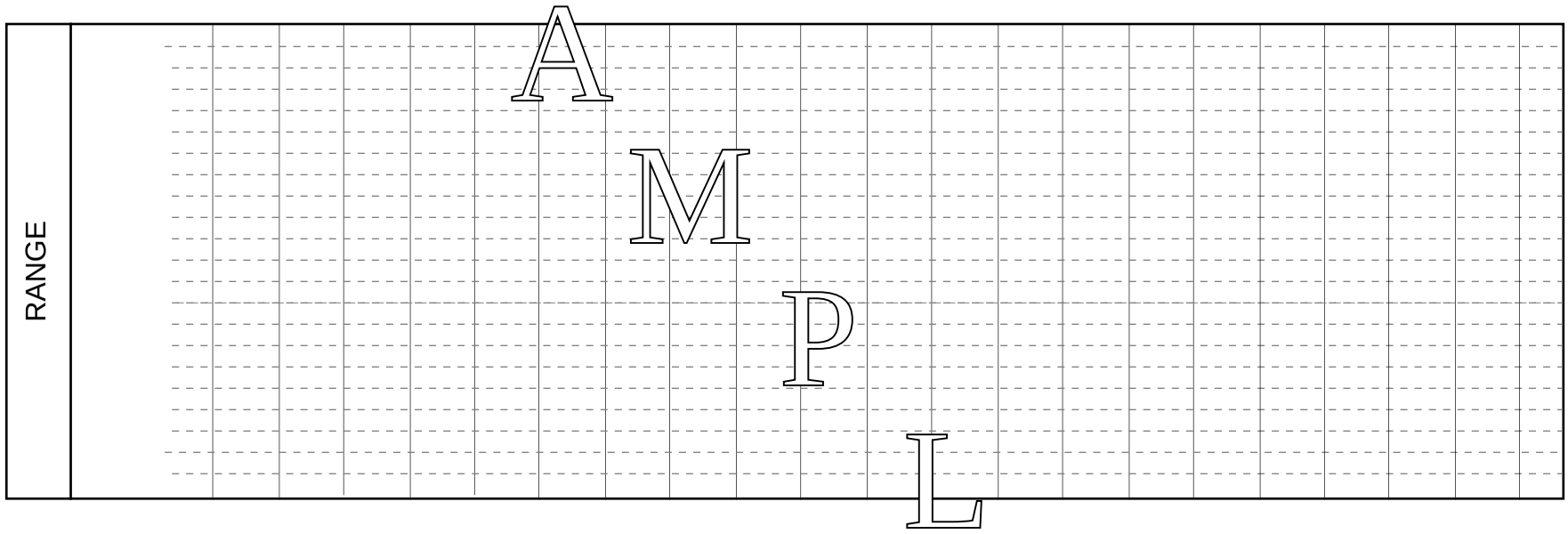
CONTROL CHART FOR THE RANGE OF DUPLICATE ANALYSES

ENGINEERING OPERATIONS DIVISION - FUELS AND CHEMICAL ANALYSIS BRANCH

OPERATION _____ INSTRUMENT _____

CHARACTERISTIC _____ COMMENTS _____

UNIT OF MEASUREMENT _____ BEGINNING DATE _____ ENDING DATE _____



DATE																				
READING 1																				
READING 2																				
RANGE (R)																				

RANGE = ABSOLUTE VALUE (READING 1 - READING 2)

UPPER CONTROL LIMIT FOR THE RANGE OF DUPLICATE MEASUREMENTS(UCLR) = 3.267 * AVERAGE RANGE

LOG ADDITIONAL NOTES ON BACK OF THIS SHEET

Form 109 - 01: 07-30-93

CONTROL CHART FOR INDIVIDUAL OBSERVATIONS

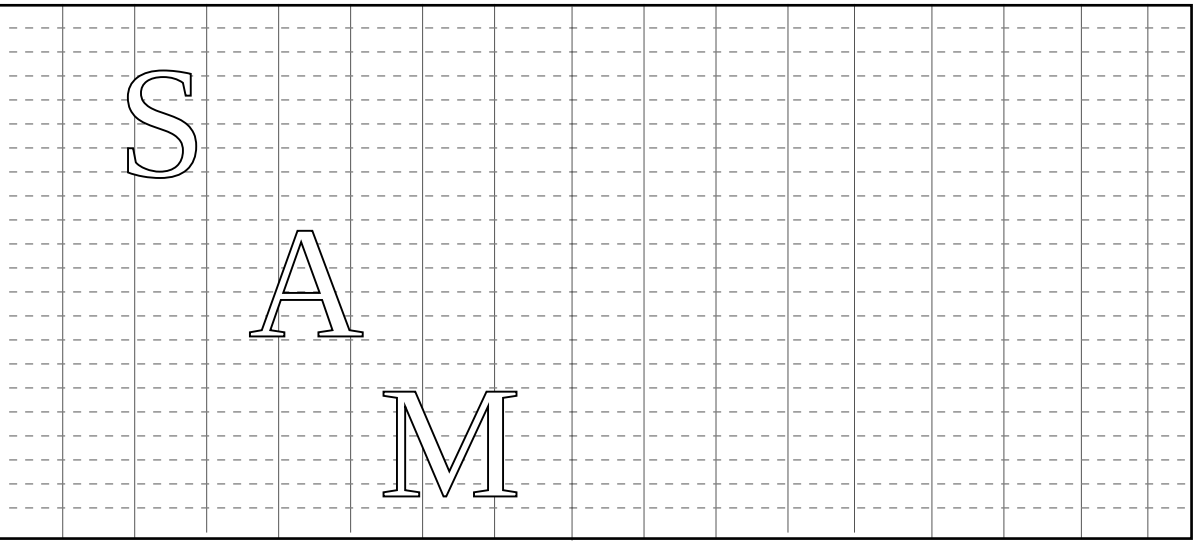
ENGINEERING OPERATIONS DIVISION - FUELS AND CHEMICAL ANALYSIS BRANCH

OPERATION _____ INSTRUMENT _____

CHARACTERISTIC _____ COMMENTS _____

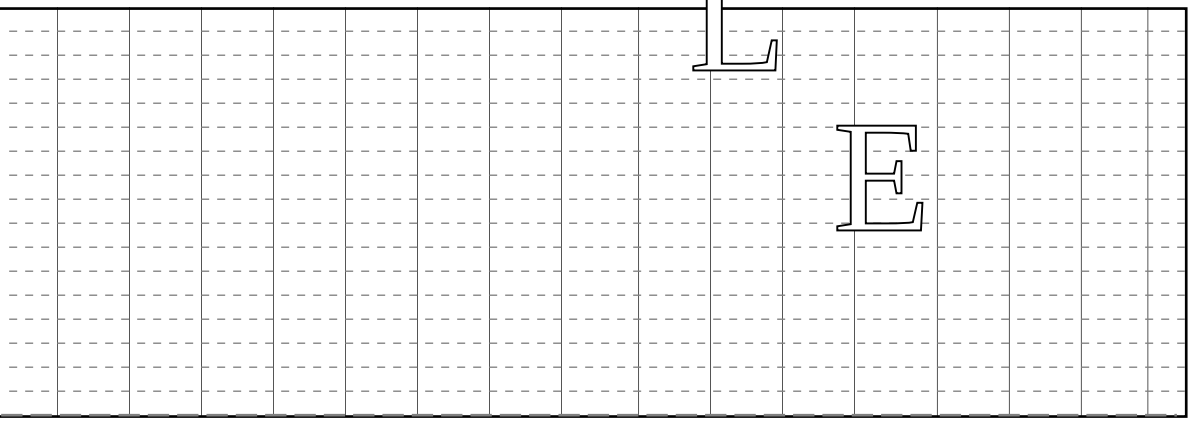
UNIT OF MEASUREMENT _____ BEGINNING DATE _____ ENDING DATE _____

INDIVIDUAL OBSERVATION



DATE															
READING															
MOVING RANGE															

MOVING RANGE



AVERAGE X = AVERAGE OF THE INDIVIDUAL OBSERVATIONS RBAR = AVERAGE MOVING RANGE

UCLX = AVERAGE X + 2.66 RBAR LCLX = AVERAGE X - 2.66 RBAR UCLR = 3.267 RBAR

LOG ADDITIONAL NOTES ON BACK OF THIS SHEET

Form 109-02: 07-30-93

CHAIN OF CUSTODY

U.S. Environmental Protection Agency
Engineering Operations Division

Fuels Field Inspection Form and Sample ID number: _____

Analyses requested: _____

_____

_____

_____

CUSTODY RECORDS

Transferred from:

Name: _____
Location: _____
Comments: _____
Seal OK? _____ Date: _____/_____/_____
Signature: _____

Received by:

Name: _____
Location: _____
Comments: _____
Seal OK? _____ Date: _____/_____/_____
Signature: _____

Name: _____
Location: _____
Comments: _____
Seal OK? _____ Date: _____/_____/_____
Signature: _____

Name: _____
Location: _____
Comments: _____
Seal OK? _____ Date: _____/_____/_____
Signature: _____

Name: _____
Location: _____
Comments: _____
Seal OK? _____ Date: _____/_____/_____
Signature: _____

Name: _____
Location: _____
Comments: _____
Seal OK? _____ Date: _____/_____/_____
Signature: _____

FCAB Enforcement Sample Chain-of-Custody Procedure

The objective of chain-of-custody procedures is to ensure the integrity and security of official samples and data. As such, this procedure is divided into two sections: sample handling and data handling. Chain-of-custody procedures are continually in effect for enforcement samples.

Samples and their paperwork are received either from Shipping and Receiving, a field inspector, or another Fuels and Chemical Analysis Branch (FCAB) analyst.

Sample Handling

Receiving Samples:

Samples received directly from Shipping and Receiving or a field inspector must be examined for broken seals, leaks, and the consistency of the recorded data found on the Official Sample Seal, EPA Form 7500-2.

If the seal is broken, it must be acknowledged as broken by answering "No" to the question "Seals OK?" in the area marked "Chain-of-Custody Record" on EPA Form 3500-5.

If EPA Form 3500-5 is missing from the shipment, the analyst will take an unnumbered blank form and fill it out from the information found on the bottle seal.

The analyst, in conjunction with his/her supervisor, will immediately contact the inspector's supervisor or, in the case of a contract inspector, the EPA Project Officer to obtain the missing form.

The analyses to be performed on such a sample will be determined by the inspector's supervisor or, in the case of a contract inspector, by the EPA Project Officer.

The Sample Number and the name of the Inspector on Form 3500-5 are compared with the data on the sample bottle seal(s), EPA Form 7500-2.

If a conflict exists between any of the above data found on Form 3500-5 and EPA Form 7500-2, notify the inspector's supervisor or EPA Project Officer immediately so that s/he can consult with the inspector and determine which form will be edited.

The analyst will edit the conflicting data by striking a single horizontal line in ink through the incorrect data and clearly printing the correct information, his/her initials, and the date as close to the incorrect data as possible.

The analyst must locate and identify all the samples associated with each Form 3500-5. S/he will enter the following information into the section labeled “Chain of Custody Record”: the sample ID numbers, date received, laboratory location, and her/his signature on the appropriate lines.

Samples received at a later date than the form will be identified in the same manner.

The Sample ID, the date received, the initials of the analyst receiving the sample, and the analyses requested will also be recorded into a log maintained by FCAB. This log may be electronic or physical.

Movement of Samples through the Laboratory:

Samples will be moved from the person receiving the samples to the analyst who will do the first analyses on those samples. The order in which tests shall be done on samples is specified by current Federal Regulations for volatility and must be followed.

For other tests, the order will be determined by the requirements of the test. The analyst who completes the analyses is responsible for transferring the sample to the next analyst or placing the samples in storage in Room 405.

In all cases, samples must be moved expeditiously to and from rooms specified by the Branch Chief as analytical areas. Those rooms may only hold samples that are awaiting analyses. When enforcement samples are being held, these rooms must be secure.

Only authorized personnel may be in the above rooms unattended with enforcement samples. A list of authorized personnel is kept by the Branch Chief.

All completed samples and those awaiting analyses will be stored in Room 405. Room 405 will be secured at all times.

Breaking sample seals:

Breaking of the sample seal will be done immediately prior to the initial analysis performed on the sample and not before.

The analyst breaking the sample bottle seal over the cap of the bottle, EPA Form 7500-2, will place his/her initials and the date in the appropriate space on the form around the circumference of the bottle or on the remainder of the seal across the bottle cap.

Movement of samples to other facilities:

Samples or portions of samples to be sent to other facilities will be sealed with an Official Sample Seal, EPA Form 7500-2. The form will be filled out in ink with the sample number, the date on which the new seal was attached, the sender's signature, and his/her name and title printed in the appropriate space.

The analyst will update the "Chain-of-Custody Record" on the original EPA Form 3500-5 to indicate that the sample was split on that date.

A new Chain of Custody Form 109-03 and a copy of this procedure will be sent along with the sample to the other facility. Fill in the sample number and analyses requested.

The analyst will record his/her name under "Transferred from," NVFEL as the location, the reason for the sample transfer on the "Comments" line, the correct response to the question "Seal OK?," the date, and his/her signature.

The form will be photocopied and the photocopy will be stored by FCAB in Room 405 with the original EPA Form 3500-5.

Sample Disposal:

Samples will be disposed of in an environmentally responsible manner. Samples will be disposed of if they comply with all Federal fuel and fuel additive regulations or upon approval of the Field Operations and Support Division (FOSD).

Samples will be recorded as "disposed" in Section 5 of EPA Form 3500-5, the Chain-of-Custody Record, along with the signature of the person acknowledging the disposal of the sample and the date signed.

Data Handling

Hand Completed Data Forms:

EPA Form 3500-5 is received with the samples from Shipping and Receiving or from a field inspector.

The sample custodian and the analyst are all responsible for filling out the appropriate areas on the forms and passing those documents, along with the sample, to the next analyst or to FCAB's data storage area in Room 405.

Once the sample has been analyzed for all the properties requested, the form is photocopied in duplicate.

One copy is sent out by overnight mail to Headquarters. The second copy is delivered to FOSD's data staff in Ann Arbor. The original copy is retained by FCAB and stored in Room 405.

They will be archived in accordance with Agency policy.

Other forms and control charts are filled out by the analyst as data are generated. Photocopies of any form or control chart may be requested at any time by EOD staff or FCAB customers.

Original control charts are kept with the instrument until they are no longer needed to monitor test quality. Then they and other forms are stored in Room 405 for at least 1 calendar year and archived in accordance with Agency policy.

Instrument-generated Hard Copies:

Hard copy sample test data generated by test instrumentation are reviewed and initialed by the analyst performing those tests. The analyst will transfer the appropriate sample test data from the hard copy to the EPA Form 3500-5 for that sample.

All instrument hard copy data will be stored in Room 405 for at least 1 calendar year. These reports will be archived in accordance with Agency policy.

Instrument-generated Electronic Copies:

When the run is completed, all instrument-generated electronic data files will be secured in Room 405 in the appropriate format.

If a data file must reside on an instrument system, it will be password protected if possible.

If that is not possible, an instrument-generated hard copy will be created and secured as described above.

FCAB Master Data Bases:

Any FCAB enforcement data base will be retained in Room 405.

TP 109 Data Base Report

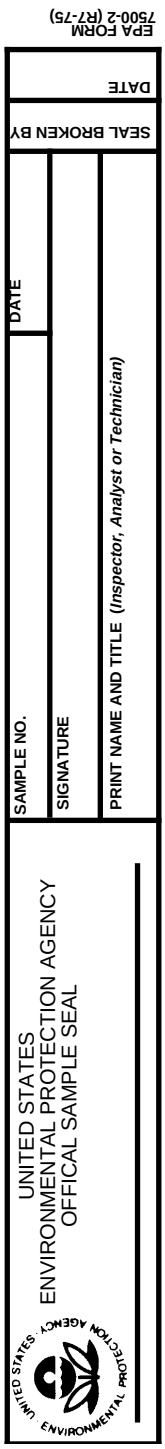
Date	Time	Analyst	Sample ID	Dilution Factor	Instrument Reading	Sample Concentration (g Pb/U.S. gal)
2/28/93	3:58:53 AM	CAS	0.100	1	0.100	0.100
Comments: Trouble with the new air supply purity.						
2/28/93	3:59:56 AM	CAS	0.050	1	0.050	0.050
2/28/93	4:01:34 AM	CAS	0.020	1	0.019	0.019
2/28/93	4:04:50 AM	CAS	0.000	1	0.000	0.002
2/28/93	4:05:35 AM	CAS	NBS-II	1	0.049	0.049
2/28/93	4:06:15 AM	CAS	Sample C	1	0.084	0.084
2/28/93	4:42:26 AM	CAS	Sample A	10	0.098	0.980
2/28/93	4:42:54 AM	CAS	NBS-II	1	0.048	0.048
2/28/93	4:43:35 AM	CAS	Sample B	1	0.052	0.052
2/28/93	4:43:59 AM	CAS	Sample C	1	0.083	0.083
2/28/93	4:44:28 AM	CAS	Sample B	1	0.049	0.049

I have performed the steps in accordance with the requirements of Test Procedure 109

Analyst's Signature _____ Date _____

I have validated the data in accordance with the requirements of Test Procedure 109

Validator's Signature _____ Date _____


EPA FORM
7500-2 (R7-75)
DATE
SEAL BROKEN BY
DATE
SAMPLE NO.
SIGNATURE
PRINT NAME AND TITLE (Inspector, Analyst or Technician)
UNITED STATES
ENVIRONMENTAL PROTECTION AGENCY
OFFICAL SAMPLE SEAL
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY