United States
Environmental Protection Agency

Office of Transportation and Air Quality
National Vehicle and Fuel Emissions Laboratory
Ann Arbor, MI 48105

Chain of Custody Procedure for Fuels Analysis Requested by OECA

This procedure is written for the Environmental Protection Agency, National Vehicle and Fuel Emissions Laboratory (NVFEL) internal use. The use of specific brand names by NVFEL in this procedure are for reference only and are not an endorsement of those products. This document may be used for guidance by other laboratories.

NVFEL Reference Number

120

Implementation Approval

Original Procedure Authorized by EPCN # 331 on 01-31-2002

Revision Description

Table of Contents

Attachments

1. Purpose

The purpose of this procedure is to document the steps required to ensure the physical security of the sample and to document chain of custody requirements for samples undergoing analysis at National Vehicle and Fuels Emissions Laboratory (NVFEL) - Chemistry Laboratory (Chem Lab). This procedure is normally required by the Office of Enforcement and Compliance Division (OECA), however, parts of this procedure may be used by the Chem Lab for other customers.

Chain of Custody is a series of steps that identify, track, and document both sample and analysis data throughout the chemical testing process performed at the NVFEL.

There are two ways data is transmitted to NVFEL from OECA. The Field Fuels Inspection (FFI), EPA Form 3500-5 has carbon copies that the field inspector keeps. They send the original paper document to NVFEL. The other way is with a computer generated FFI (CGFFI), which is a two-page document. Page one is also labeled Field Fuels Inspection, however, there is no form number associated with it. Page two is a Chain of Custody form, which contains the sample numbers. This procedure documents the use of the CGFFI form. If the EPA Form 3500-5 is used, any data recorded will be in the corresponding area as described for the CGFFI.

2. Test Article Description

This procedure applies to all Office of Enforcement and Compliance Assurance (OECA) samples received at the NVFEL – Chem Lab.

A document accompanying the sample (traveler) for each analysis contains the official data.

The “Sample Transfer” tag is always completed, initialed and dated by the analyst as “Accepted” when the sample is taken for analysis, and always completed, initialed and dated as “Released” when the sample leaves the custody of the analyst.

3. References

3.1 National Environmental Laboratory Accreditation Conference, Quality Systems Manual.

3.2 Current NVFEL Quality Manual.

3.3 Current NVFEL Safety Policies.

4. Required Equipment

4.1 Computer
Equipment Used: PC with Windows NT4

4.2 Computer Database
Program Used: Access Application

4.3 "Official Sample Seal" Attachment A

4.4 Computer Generated Fuels Field Inspection Form, Attachment B

or

Fuels Field Inspection, EPA Form 3500-5, Attachment C.

4.5 EPA NVFEL Enforcement, 120-01: 01/01/2002, Attachment D (for gasoline)

4.6 EPA NVFEL Diesel Enforcement, 120-02: 01/01/2002, Attachment E

4.7 "Test Required" label, Attachment F.

4.8 "Sample Transfer" tag, Attachment G.

4.9 External Fuel Analysis Chain of Custody, Form 120-03: 01-01-2002. Attachment H.

5. Precautions

5.1 The Access database program is closed after use.

5.2 The sample containers must be handled carefully so they are not damaged.

5.3 The FTAG Number on the "Test Label" remains legible and is securely attached to each sample container throughout analysis.

5.4 Ensure that the auto-sample vials used for analysis at various instruments are identified with the original FTAG Number of the sample.

5.5 Ensure that any manual entry of sample identification numbers into analysis instrument sampling systems are correct for both sample order and sample identity.

6. Visual Inspection

The sample is delivered to the "Volatility Lab" by shipping and receiving.

If a sample does not meet the requirements of this section, record the reason in the comment section of CGFFI form. Record your initials and date in the CGFFI comment section.

6.1 Verify that the "Official Sample Seal" (Attachment A) has not been tampered with. Note the integrity of label in the comment section of the CGFFI.

6.2 Verify that the "Official Sample Seal" is attached to both the sample container cap and the container.

6.3 Verify that the sample container has not leaked fuel. Note the integrity of the container in the comment section of the CGFFI. See Attachment B.

6.4 Look at the fuel in the container. If it appears normal, write "OK' in the CGFFI comment section. If not, note the conditions in the CGFFI comment section.

6.5 Verify that the CGFFI has the Inspection Number, Analyses to be performed, and the name and signature of the field inspection officer.

6.6 Verify that the "Inspection Number" on the "Official Sample Seal" matches the "Inspection Number" on the CGFFI form. If not, then OECA is notified and the discrepancy is noted in the CGFFI comment section.

6.7 On bottom of the CGFFI, record the "Date received by lab", enter "NVFEL" as laboratory location and check off the "Seal OK" box, if the seal was OK. Then sign on "Signature" line.

6.8 Verify that adequate sample volume exists to perform the required analyses.

6.8.1 Place the container next to the volume gage.

6.8.2 If the fuel in the container is less than the 70% indicator but more than the 20% level, record fuel level in the CGFFI comment section.

6.8.3 If the fuel in the container is less than the 20% indicator, call OECA for instructions on how to proceed.

7. Test Article Preparation – N/A

8. Test Procedure

100 Go to the Chem Lab computer that has the database program.

101 Open the "Start Fuel Tests Appl." program.

102 Click on "Data Detail," which opens the "Field Fuels Inspection" form.

103 Click on the "New FFI" button.

104 Transfer the corresponding information for each sample from CGFFI form (or Form 3500-5) to the database.

105 In the "Sample Log" section, enter the sample number, fuel type, and check the corresponding boxes for the required analyses.

106 Once the data are entered, an unique FTAG number is assigned to the sample.

Note: Write the FTAG Number on Form 3500-5.

107 For gasoline, under "Report List," scroll down to "Enforcement Paper Work by FTAG."

or

For diesel, under "Report List," scroll down to "Diesel Enforcement Paper Work by FTAG."

108 Double-click on that heading.

109 Type in the FTAG Number for the sample you want printed on the form.

110 Click on "the "OK" button. This will automatically print either 120-01or Form 120-02, depending on which pull down menu selection was made in Step107.

111 Under "Report List" scroll down to "Label by FTAG."

112 Double-click on that heading.

113 Type in the FTAG number of the sample you want printed on the label.

114 Click on "the "OK" button. This will automatically print the label that will be attached to the sample container.

This is the "Tests Required" label. The "Test Required" label lists all of the required analyses.

115 Verify that the data transcribed from the CGFFI is correct on Form 120-01 (or Form 120-02) and the label. If not correct, go back to the Access database record, make corrections, and repeat Steps 107 through 115.

The CGFFI form is attached to Form 120-01 (or Form 120-02). Form 120-01 (or Form 120-02) becomes a traveler document that accompanies the sample(s) throughout the analyses process. If Form 3500-5 was submitted by the field inspector, it becomes the traveler document that accompanies the sample(s) during analysis.

116 Close the "Fuels Test " program to ensure data security.

117 Attach the "Tests Required" label to the sample container.

118 Attach a waterproof "Sample Transfer" tag with a wire to the sample

119 On the "Sample Transfer" tag, in waterproof ink, write the corresponding FTAG Number from the "Test Required" label for that sample. Verify that all FTAG numbers on tags and labels match each other.

120 The analyst initials and dates the top "Released" field of the transfer tag upon accepting the sample. They place an "X" under the transfer tag "Secure" heading and put the sample in secured location. The samples, traveler documents and tags remain in the secured area until they are needed for analysis. The "Official Sample Seal" must remain intact until the sample is ready for RVP analysis.

121 As the sample is transferred between analysis stations, the "Sample Transfer" tag must be initialed and dated by the person transferring the sample and by the person receiving the sample. If the sample is transferred into secured storage, the person transferring must place an "X" under the transfer tag "Secure" heading and put the sample in secured location.

122 As each analysis is completed; the results are entered into the appropriate field(s) of the traveler document, dated and initialed by the analyst. A qualified analyst, other than the one performing the analysis, must verify these values for correctness in the database and they must date and initial traveler document.

If any discrepancies are found between the traveler document and the Access database, the analyst is notified and is responsible for making the appropriate changes. If a typo or other manual data entry error is found, the data must be corrected, dated and initialed by the person that performed the analysis. A qualified analyst, other than the one performing the analysis, must verify these corrections.

123 Upon completion of all requested analyses, transfer the sample container into the secured storage area. Document this transfer on the "Sample Transfer" tag.

124 File the traveler document, containing the official analysis results, in the secured location.

125 To transfer monthly and annual official data to OECA:

Obtain a completed report and take it to the Chem Lab Manager, or his designated representative. They will randomly select data to validate a minimum of 2 results per analyst whose results are represented in the report.

This second level of validation consists of comparing instrument QC data with sample data on the Official Record, and with the value in the report. This ensures that the sample values are identical and within the QC guidelines of the method. See Attachment I for the validation wording that will be on each report.

Sign the report cover page.

126 Each completed sample is held in secured storage until a written release for disposal is provided by OECA. Upon disposal, the "Sample Transfer" tag is completed, initialed and dated as "Disposed of". The tag is then filed by the FTAG Number in the secured area with corresponding traveler documentation.

Section 200 - Splitting Samples for Analyses at Other Laboratories.

If a Confidential / Proprietary sample, or an aliquot is sent to another laboratory for analysis, it must be identified only by the FTAG number on both the sample and on any accompanying paperwork. Reference to the product name, number, or manufacturer is not to be included.

201 Transfer an aliquot of the fuel to a new clean sample container.

203 On the "Official Sample Seal," record the FFI sample number, the date, and sign your name.

202 Seal the container with an "Official Sample Seal"

204 In the comments section of the CGFFI (or Form 3500-5), record the sample as "Split" and where it was sent. Also write the date and your initials in this section.

205 On the "Split Sample Chain of Custody" form, record the FFI number, the sample number, and a list of requested analyses.

206 In the first "Transferred from" area, record your name, location, comments, "yes" for seal OK, the date, and your signature.

9. Data Input

9.1 The corresponding data are transferred for each sample from CGFFI form (or Form 3500-5) to the database.

9.2 When possible, sample and quality control data are transferred electronically from instruments back to the Chem Lab database after each analysis session. Data are transferred manually for instruments that do not have that capability.

10. Data Analysis

N/A

11. Data Output

11.1 Instrument raw data.

11.2 A completed traveler document containing the official analysis data.

11.3 A completed "Sample Transfer" tag.

11.4 Monthly and annual reports to OECA.

12. Acceptance Criteria

NVFEL must accept all enforcement samples, unless a rejection decision is made by OECA and documentation is provided. The following conditions must be met. If not, they are documented in the comment section of the traveler document.

12.1 The sample must be of adequate volume for the analyses requested.

12.2 The "Official Sample" seal must not be broken or removed prior to the RVP test. It must also have complete identifying documentation.

12.3 All FFI forms, Chain of Custody forms, and Official Sample seals accompanying the samples must be signed by the field inspector.

12.4 Any discrepant samples must be segregated until a decision is made on disposition. Sample disposition must be documented, along with date and initials, in the comment section of the CGFFI form or on the documentation sent by OECA in the absence of the CGFFI form.

12.5 The “Sample Transfer” tag must be completely filled out by the appropriate personnel to reflect the movements necessary to accomplish the analyses performed on the sample.

12.6 Only Chemistry Laboratory personnel are permitted to be sample custodians.

12.7 Only Chemistry Laboratory personnel are permitted to verify Chain of Custody and analysis data.

13. Quality Provisions

13.1 Test results held digitally within the analyzer systems must be checked by the analyst for completeness and correctness per the requirements of the particular test method before transfer is made to the Chemistry Laboratory database. Analyst specific passwords must be used to permit electronic transfer.

13.2 Test data held on the Chemistry Laboratory database is backed up weekly to permanent media.

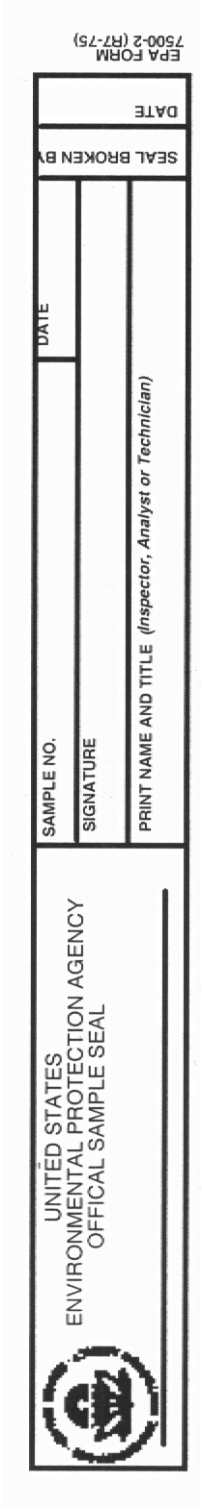
13.3 Test results written on the traveler document must be verified by the analyst.

13.4 Another analyst familiar with the analysis must verify data transcription of each analysis result from on the traveler document to the database.

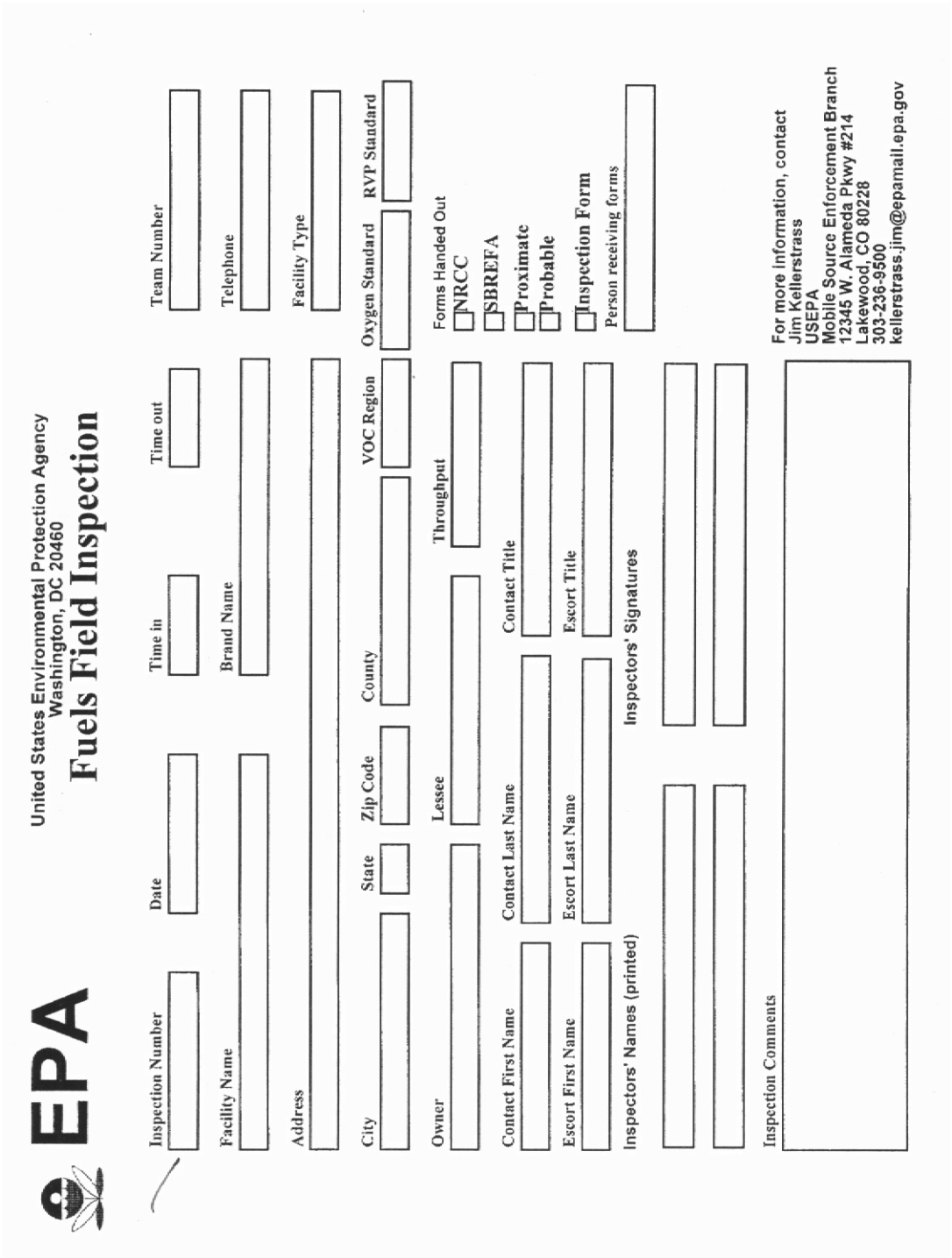
13.5 The file containing the official data entered on the traveler document and the computer containing the Chem Lab database is physically secured in a locked area.

13.2 Computer programs used to transfer data to and from each instrument have been verified to perform correctly prior to implementation of the computer program.

Attachment A



Attachment B



Attachment B Continued

Chain of Custody

Inspection Number | Inspection Date | Facility Name

Address | City | State | Zip Code

County | VOC Region | Oxygen Standard | RVP Standard

Sample #	Sample Type	Grade	Begin Date	End Date	Area In Use	Test Type

Field

Date shipped to lab: _____

Seals Ok? ☐

Inspectors' Names (print) _____ _____

Inspectors' Signatures _____ _____

Lab

Date received by Lab _____

Laboratory locations: _____

Seals Ok? ☐ Signature _____

Attachment C

EPA U.S. Environmental Protection Agency, Washington, DC 20460

Fuels Field Inspection

Local RVP: _____ | Check here if QA sample taken: ☐ | Inspection Number: _____

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 Contract [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	Annual In Use

3. Sample Information and Field Violations

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

4. Test Results

Sump #	Test Type	Test #	Test Result

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

Total Monthly Throughput (gallons)

5. Comments

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.

Attachment D

FFI # **EPA NVFEL Enforcement** **FTAG ID** Counter #

Facility Name
Owner
Address
City, Sta. Zip
Phone number FAX:

Comments

Inspector Name
Inspection Date Date
Log in Date Date

RVP VOC Season: Fuel_Code Log in Person

Item	Method	Units		Result	Result	Analyst	Date	Val.by	Date
Elemental Composition				XXX					
Sulfur (TP 116)	ASTM D 2622	ppm	X						
Molecular Composition				X . XX					
Benzene	ASTM D 3606	vol %	X						
FIA (TP112)			X	XX . X					
Aromatics	ASTM D 1319	vol %							
Olefins	ASTM D 1319	vol %							
Saturates	ASTM D 1319	vol %							
Aromatics (TP 117)	MSD		X	XX . X					
Total Aromatics	MSD	vol %							
Benzene	MSD	vol %							
Oxygen	MSD	wt %							
Total Oxygenates	MSD	vol %							
	MSD	vol %							
	MSD	vol %							
Oxygenates (OFID)			X	X . XX					
DIPE	O-FID	vol %							
Methanol	O-FID	vol %							
Ethanol	O-FID	vol %				CPU	Results		
t-Butanol	O-FID	vol %				VOC			
Methyl-t-butyl ether	O-FID	vol %				NOx			
Ethyl-t-butyl ether	O-FID	vol %				Toxics			
MTBE	O-FID Oxy	wt%					XX . X		
ETBE	O-FID Oxy	wt%							
TAME	O-FID Oxy	wt%							
EtOH	O-FID Oxy	wt%							
TAME	O-FID	vol %							
Total Oxygen	O-FID	wt %							
Physical Properties									
Distillation (TP 115)	ASTM D 86		X	XXX . X					
Initial Boiling Point		°F							
10% Evap. Point		°F							
50% Evap. Point		°F							
90% Evap. Point		°F							
End Point		°F							
Residue		vol %							
Recovery		vol %							
Loss		vol %							
E 200		vol %							
E 300		vol %							
Gravity (TP113)	ASTM D 4052	Density	X		X . XXXX				
	ASTM D 4052	API°			XX . X				
	ASTM D 4052	Spec.Grav			X . XXXX				
				XX . XX					
Vapor Pressure (TP104)	CFR Method 3	Ptot	X						
		Pabs							
		RVPE							

Form 120-01; 01/01/2002

Attachment E

Field Fuel Inspection # **EPA NVFEL** **Diesel Enforcement** **FTAG ID** Counter

Facility Name
Owner Comments
Address
City, State Zipcode
Phone # FAX: # RVP

Inspector Name
Inspection Date Date
Log in Date Date

VOC Season: Fuel_Code Log in Person Initial

Item	Method	Units		VOC Result	Season: Result	Fuel_Code Analyst	Fuel_Code Date	Log in Person Analyst	Log in Person Date
Elemental Composition									
Sulfur (TP 116)	ASTM D 2622	Wt. %	X						
Molecular Composition									
FIA (TP112)			X						
Aromatics	ASTM D 1319	vol %							
Olefins	ASTM D 1319	vol %							
Saturates	ASTM D 1319	vol %							
Paragon									
Kinematic Viscosity		Centistrokes							
Cetane Number		Cetane #							
Flash Point	D 93	Deg F							
Aromatics SFC	ASTM D 5186	vol%							
Physical Properties									
Distillation (TP 115)	ASTM D 86		X						
Initial Boiling Point		°F							
10% Evap. Point		°F							
50% Evap. Point		°F							
90% Evap. Point		°F							
End Point		°F							
Residue		vol %							
Recovery		vol %							
Loss		vol %							
Cetane Index (TP 114)	D 976		X						
API Gravity (TP 113)	ASTM D 4052	API°	X						
		Density							
		Spec.Grav							

Form 120-02; 01/01/2002

Attachment F

FTAG: Counter # **Log in Date**

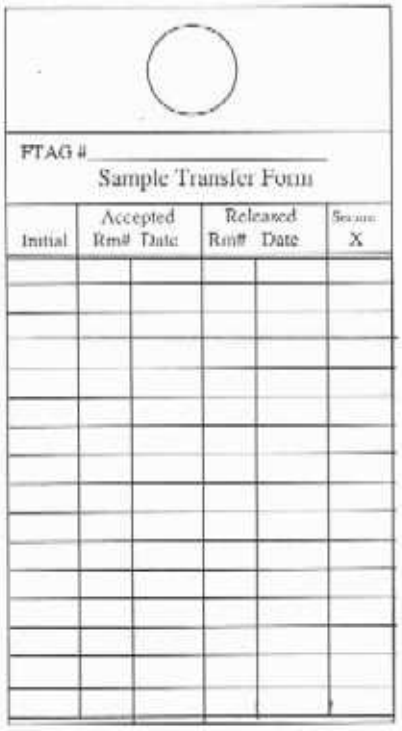
Sample ID:FFI # +sample # Cold Rm.Tray #

Physical RVP D 86 Gravity

Molecular D 1319 MSD D 3606 OFID VOC

Elements: Sulfur

Attachment G



FTAG #_____

Sample Transfer Form

Initial	Accepted Rm#	Accepted Date	Released Rm#	Released Date	Secure X

Attachment H

Sample and / or Official Data Chain of Custody

U.S. Environmental Protection Agency
National Fuels and Emissions Laboratory
Ann Arbor, MI. 48105

Fuels Field Inspection Form and Sample ID number: ______________________________

Analyses requested: ____

____

____

____

CUSTODY RECORDS

Transferred from:	Received by:
Name: ____________________	Name: ____________________
Location: ____________________	Location: ____________________
Comments: ____________________	Comments: ____________________
Seal OK? ________ Date: ______/______/______	Seal OK? ________ Date: ______/______/______
Signature: ____________________	Signature: ____________________
Name: ____________________	Name: ____________________
Location: ____________________	Location: ____________________
Comments: ____________________	Comments: ____________________
Seal OK? ________ Date: ______/______/______	Seal OK? ________ Date: ______/______/______
Signature: ____________________	Signature: ____________________
Name: ____________________	Name: ____________________
Location: ____________________	Location: ____________________
Comments: ____________________	Comments: ____________________
Seal OK? ________ Date: ______/______/______	Seal OK? ________ Date: ______/______/______
Signature: ____________________	Signature: ____________________

Form 120-03: 01-01-2002

Attachment I

Statement on Monthly and Annual Reports

These data were validated by the undersigned through a random sampling of results from each analyst. The entire body of analysis data has not been subject to this second level of validation. However, in any requested enforcement case, this second level of data validation will be provided upon request by OECA.