

Part VIII. Test Procedures, Methods and Reporting Requirements for the Information Collection Request for Petroleum Refineries

This document provides an overview of approved methods, target pollutants and units of measure, and reporting requirements for petroleum refineries. All recipients must complete and submit test results by August 31, 2011. This document will be referred to as the 'petroleum refinery test program.' The document is organized as follows:

- 1.0 Testing Procedures and Methods**
- 2.0 How to Report Data**
- 3.0 How to Submit Data**
- 4.0 Contact Information for Questions on Test Plan and Reporting**

1.0 Testing Procedures and Methods

The EPA petroleum refinery test program requires emissions and other test data for several pollutants, including specific hazardous air pollutants (HAP), criteria pollutants, and potential surrogate groups. If EPA is requesting that you complete emissions testing, the ICR website (<https://refineryicr.rti.org>) will include a list of emissions sources selected for testing. For each petroleum refinery emissions source listed on the ICR website, you must perform an emissions test for some combination of pollutants (*i.e.*, simultaneous or overlapping measurements per group) and diluents according to the test protocols presented in Sections 1.1 through 1.4 of this section.

Please refer to the ICR website (<https://refineryicr.rti.org>) for additional testing information. As noted previously, the ICR website includes the specific sources for which we are requesting that you perform emissions testing. Please note that you do not have to submit a test plan to EPA for approval prior to testing; however, we recommend that you prepare a test plan for your own use to assist in the planning and to ensure that you address all of the testing and reporting requirements. Please note that you also must report your process and emissions testing data using the EPA Electronic Reporting Tool (ERT), where applicable. The ERT requires you to report most elements of a test plan. You are directed to the ERT (http://www.epa.gov/ttn/chief/ert/ert_tool.html) for a more complete and interactive description of these test plan elements.

You must follow all of the procedures as specified in the test methods, including the quality assurance and quality control measures, and document the results in the ERT and in any test report provided to EPA. For this program, you need not obtain audit materials from your state or local agency or from EPA. You may apply any third party audit materials you have on hand and document the results, but you are not required to do so.

You may have submitted test data for some of these pollutants already. If you have conducted any of the requested groups of tests (*i.e.*, any complete set of tests required to be conducted

simultaneously) in the past 5 years and reported the data in Part IV of this ICR, you are not required to do additional testing for those pollutants.

1.1 Stack Test Methods

The following emissions sources must follow the stack test method procedures described in this section:

- Fluid catalytic cracking unit
- Thermal catalytic cracking unit
- Fluid coking unit
- Delayed coking unit
- Catalytic hydrocracking unit
- Hydrogen production unit
- Asphalt blowing still
- Sulfur recovery unit

During a 30-day period that includes emissions testing and fuel analysis, you should also collect and report relevant process data that is available from installed process monitors or recordkeeping, such as: total heat input; material description, composition, and feed rate; coke burn-off rate; emissions control devices in use during the test; control device operating or monitoring parameters (including, as appropriate to the control device, flue gas flow rate, pressure drop, scrubber liquor pH, scrubber liquor flow rate, sorbent type and sorbent injection rate, primary and secondary voltage and current, temperature); process parameters (such as type of crude, oxygen concentration, temperature); and any pollution prevention or other hazardous air pollutant emission reduction approaches being implemented during the test program. You should report the above process data as daily averages in the Process Data section of the Testing Tool; you can download this spreadsheet from the ICR website (<https://refineryicr.rti.org>). For process data that you record during testing, make clear, using the Notes section of the ERT or spreadsheet, the correlation between emissions measurements and process data (*e.g.*, identify Method 23, run 1 for the associated process data on the process data details tab of the ERT).

The owner/operator of the petroleum refinery must certify that the unit tested was operating in a normal and representative manner during the performance test. The owner/operator must also certify that it operated the pollution control equipment, if any, on the unit tested in accordance with manufacturers' specifications and requirements for proper operation during the emissions testing.

1.1.1 Sample Location and Gas Composition Analysis Methods

You must use U.S. EPA Method 1 of Appendix A-1 to 40 CFR Part 60 to select the locations and number of traverse points for sampling for the tests in this section. See <http://www.epa.gov/ttn/emc/methods/method1.html> for a copy of the method and guidance information for sampling situations not meeting Method 1 criteria.

For analysis of flue gas composition, including oxygen concentration, you must use U.S. EPA Method 3A or 3B of Appendix A-2 to 40 CFR Part 60. See

<http://www.epa.gov/ttn/emc/methods/method3a.html> for Method 3A or
<http://www.epa.gov/ttn/emc/methods/method3b.html> for Method 3B information.

1.1.2 Emissions Measurement Methods

Table 1.1 summarizes the testing required to be performed for each type of process unit. Where indicated in the tables, you must conduct the tests for the indicated pollutants simultaneously for your process unit. If the table does not indicate simultaneous sampling is required for multiple pollutants, you may still choose to do so. For example, if you are asked to test your fluid catalytic cracking unit, you may conduct four separate sets of tests, one set that includes all pollutants indicated with an “a”, another set that includes all pollutants marked with a “b,” etc., or you may choose to conduct all the testing at the same time (if possible). For situations where simultaneous sampling may not be possible due to sampling port or duct diameter constraints, contact the testing representative identified in Section 4.0 of this part (Part VIII) with your recommended solution for approval.

Table 1.2 presents a list of the test methods to use for completing the tests required in Table 1.1. Tables 1.3 through 1.5 provide listings of target analytes for volatile and semi-volatile organic HAP and aldehyde test methods. Table 1.6 provides a listing of target analytes for C1 through C5 hydrocarbons. For copies of the U.S. EPA test methods, continuous emissions monitoring systems (CEMS) performance specifications, and additional information, please refer to EPA’s Emission Measurement Center website, <http://www.epa.gov/ttn/emc/>, and EPA’s Hazardous Waste website, <http://www.epa.gov/waste/hazard/testmethods/sw846/online/index.htm>, as appropriate. If you would like to use a method not on this list, please contact EPA for approval of an alternative method. Please see Section 4.0 of this document for a list of persons to contact with questions regarding test methods and reporting.

Report all pollutant emission data as indicated in Table 1.2. Report the results of your emissions tests according to the directions provided in Section 2.0 of this enclosure.

Unless otherwise specified, each pollutant emissions test should consist of at least three test runs for the sampling duration and/or volume indicated for each specified unit or operation. You should collect emissions samples for the identified pollutants downstream of the last relevant emissions control device (*i.e.*, stack or other point representing the composition of the flue gases at the exit to the atmosphere), unless otherwise indicated.

You must keep the following records for 3 years:

- Documentation that each emissions test was conducted in accordance with the enclosed sampling protocol; and
- The results of each emissions test.

Table 1.1: Required Stack Testing Matrix for Process Units by Process Type

Pollutant ¹	Process Unit						
	Delayed Coking ²	Fluid Catalytic Cracking, Thermal Catalytic Cracking, Fluid Coking, Catalytic Hydrocracking ²	Catalytic Reforming		Hydrogen Production ²	Asphalt Blowing ²	Sulfur Recovery ²
			Purge Vent(s) ^{2,3}	Coke Burn Vent ^{2,3}			
Speciated Volatile Organic HAP	a	a	a	a	a	a	a
Speciated Semi-volatile Organic HAP	a	a	a		a	a	a
Aldehydes	a	a	a		a	a	a
Total hydrocarbons (THC)	a	a	a		a	a	a
Methane and ethane	a	a	a		a		a
C1 through C5 hydrocarbons						a	
Carbon Monoxide (CO)	a	a	a		a	a	a
Dioxins and Furans (D/F), Polychlorinated biphenyls (PCB)	b	b		b			
Hydrogen chloride (HCl), chlorine gas (Cl ₂), hydrogen fluoride (HF), and hydrogen cyanide (HCN)	c	e		e			
Hydrogen sulfide (H ₂ S), carbonyl sulfide (COS), and carbon disulfide (CS ₂)	c					f	f
Total reduced sulfur compounds (TRS)	c					f	f
Mercury (Hg)	d	d		d			
Hexavalent chromium (Cr ⁺⁶)		d ⁴					
Other Metals	d	d		d			
PM/PM _{2.5} (filterable)	d	d		d			
PM _{2.5} (condensable)	d	d		d			
Ammonia (NH ₃)		d ⁴					
Oxides of nitrogen (NO _x)	d	d		d	g	g	
Sulfur dioxide (SO ₂)	d	d		d	g	g	
Gas flow rate	a, b, c, d	a, b, d, e		a, b, d, e	a, g	a, f, g	a, f
Oxygen (O ₂) and Carbon dioxide (CO ₂)	a, b, c, d	a, b, d, e		a, b, d, e	a, g	a, f, g	a, f
Moisture	a, b, c, d	a, b, d, e		a, b, d, e	a, g	a, f, g	a, f

¹ Three runs per test, unless otherwise noted

² For each process unit, simultaneous sampling for pollutants marked with same letter

³ Catalytic reforming purge vents include all atmospheric vents associated with catalyst regeneration other than the coke burn-off vent during the coke burn-off cycle. Catalytic reforming purge vents do not include gas streams directed to a fuel gas system, a process heater or boiler, or a flare. Catalytic reforming purge vents include the atmospheric vents associated with initial depressurization and purging (including vacuum system exhausts, if used), catalyst rejuvenation, and reduction or activation purging. These vents differ between different reforming units and may use the same discharge vent used during the coke burn-off cycle, in which case this vent would also require testing for organics. If you have additional questions regarding what is or is not considered a purge vent, contact Brenda Shine (contact information provided in Section 4.0 of this Part VIII).

⁴ For fluid catalytic cracking units and fluid coking units only

Table 1.2: Summary of Test Methods and Alternative Methods

Pollutant	Recommended Method	Alternative Method	Target Reported Units of Measure¹
Speciated Volatile Organic HAP ²	SW-846 Method 0031 with SW-846 Method 8260B. Collect and analyze a minimum of three sets of adsorbent traps over a minimum 1-hour period per test run (total of three test runs).	U.S. EPA Method 18. Use a minimum sample time of 1 hour per run.	lb/hr and µg/dscm
Speciated Semi-volatile Organic HAP ³	SW-846 Method 0010. Collect at least 4 cubic meters per each of three test runs. Use SW-846 Method 8270C or D for the analytical finish.		lb/hr and µg/dscm
Aldehydes ⁴	SW-846 Method 0011 with SW-846 Method 8315A. Collect a minimum volume of 1.3 cubic meters	U.S. EPA Method 320 only if concentrations are known to be 1 ppmvd or greater. Use a minimum test run time of 2 hours. All compounds must be validated according to Section 13.0.	lb/hr and µg/dscm
THC	U.S. EPA Method 25A. Use a minimum sampling time of 1 hour per run. Calibrate the measuring instrument with propane.	You may calibrate the measuring instrument with a mixture representing the organic compounds being emitted. Provide the chemical make-up and response factors for the alternative standards; report the results as the alternative organic mixture and as propane equivalent concentrations.	lb/hr and ppmvd as propane
Methane and ethane	U.S. EPA Method 18. Use a minimum sample time of 1 hour per run.	U.S. EPA Method 320. All compounds must be validated according to Section 13.0.	lb/hr and ppmvd
C1 through C5 hydrocarbons ⁵	U.S. EPA Method 18. Use a minimum sample time of 1 hour per run.	U.S. EPA Method 320. All compounds must be validated according to Section 13.0.	lb/hr and ppmvd
CO ⁶	U.S. EPA Method 10 or 10B. Collect a minimum volume of 60 liters over a minimum sample time of 1 hour per run.	None	lb/hr and ppmvd
D/F, PCB ⁷	U.S. EPA Method 23. Collect a minimum volume of 3 cubic meters. Use high resolution GC/MS for the analytical finish.	None	lb/hr and pg/dscm
HCl, Cl ₂ , and HF	U.S. EPA Method 26A. Collect a minimum volume of 2 cubic meters.	U.S. EPA Method 26 or U.S. EPA Method 320 (excluding Cl ₂) if there are no entrained water droplets in the emissions stream. All compounds must be validated according to Section 13.0.	lb/hr and mg/dscm
HCN	U.S. EPA Other Test Method 29. Collect a minimum volume of 0.9 cubic meters.	U.S. EPA Method 320 if there are no entrained water droplets in the emissions stream. All compounds must be validated according to Section 13.0.	lb/hr and µg/dscm

Table 1.2: Summary of Test Methods and Alternative Methods (cont.)

Pollutant	Recommended Method	Alternative Method	Target Reported Units of Measure¹
Hg	ASTM D6784-02 (Ontario-hydro method). Collect a minimum volume of 3 cubic meters.	U.S. EPA Method 30B only if you have the knowledge and capability to speciate mercury using this method.	lb/hr and µg/dscm
Hexavalent Chromium (Cr ⁺⁶)	SW-846 Method 0061. Collect a minimum sample volume of 3 cubic meters per run (or the maximum sample volume possible while maintaining the pH of first impinger solution above 8.5 if less than 3 cubic meters can be collected) and use a sample preconcentrator according to §4.4 and 4.5.	None	lb/hr and µg/dscm
Metals ⁸	U.S. EPA Method 29. Collect a minimum volume of 3 cubic meters per run. Use ICAP/MS or GFAAS for the analytical finish. For stacks without entrained water droplets, determine filterable PM emissions according to §8.3.1.1 with a filter temperature of 320°F +/- 25°F. For stacks with entrained water droplets, do not determine filterable PM with Method 29 (see PM/PM _{2.5} below).	For stacks without entrained water droplets, you may opt to conduct a separate EPA Method 5 or 5F test with a filter temperature of 320°F +/- 25°F in lieu of measuring PM with the Method 29 train (see PM/PM _{2.5} below).	lb/hr and mg/dscm
PM/PM _{2.5} (filterable plus condensable) ⁹ from stacks without entrained water droplets (e.g., not from units with wet scrubbers) ⁹	U.S. EPA Method 201A and Method 202. Collect a minimum volume of 2 cubic meters per run.	None	lb/hr and gr/dscf lb/hr and gr/dscf (if use Method 5F, report both total filterable catch and non-sulfate PM)
PM/PM _{2.5} (filterable plus condensable) ⁹ from stacks with entrained water droplets AND Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) from wet scrubber recirculation liquid ⁹	U.S. EPA Method 5 with a filter temperature of 320°F +/- 25°F and Method 202. Collect a minimum volume of 2 cubic meters per run. AND ASTM D5907	None For TDS and TSS, Standard Methods of the Examination of Water and Wastewater Method 2540B for solids in scrubber recirculation liquid (as fed to the wet scrubber)	lb/hr and gr/dscf mg solids per liter of scrubber recirculation liquid ¹⁰

Table 1.2: Summary of Test Methods and Alternative Methods (cont.)

Pollutant	Recommended Method	Alternative Method	Target Reported Units of Measure¹
NH ₃	South Coast AQMD Method 207.1	U.S. EPA Conditional Test Method 027 (CTM-027) or U.S. EPA Method 320	lb/hr and ppmvd
NO _x ¹¹	U.S. EPA Method 7E; 2-hr sample time	U.S. EPA Method 7	lb/hr and ppmvd
SO ₂ ¹¹	U.S. EPA Method 6C; 2-hr sample time	U.S. EPA Method 6	lb/hr and ppmvd
H ₂ S, COS, CS ₂	U.S. EPA Method 15. Per method, a minimum of 16 individual samples during a period of 3 to 6 hours, per run	None	lb/hr and ppmvd
TRS ¹²	U.S. EPA Method 15A or 16A; 3-hr sample time per run; (one 3-hr sample per run or three 1-hr samples per run)	U.S. EPA Method 16B combined with the analysis procedures of U.S. EPA Method 15.	ppmvd
Gas flow rate	U.S. EPA Method 2	U.S. EPA Method 2A, 2B, 2C, 2D, 2F, or 2G, as appropriate	acfm, scfm, and dscfm
O ₂ /CO ₂	U.S. EPA Method 3A	U.S. EPA Method 3B	percent volume, dry
Moisture	U.S. EPA Method 4	None	percent volume

- ¹ lb/hr = pounds per hour; µg/dscm = micrograms per dry standard cubic meter; ppmvd = parts per million by volume, dry basis; pg/dscm = picograms per dry standard cubic meter; mg/dscm = milligrams per dry standard cubic meter; gr/dscf = grains per dry standard cubic foot; acfm = actual cubic feet per minute; scfm = standard cubic feet per minute; dscfm = dry standard cubic feet per minute.
- ² Target analytes are given in Table 1.3; test runs must occur during the test runs for speciated semi-volatile organic HAP (*i.e.*, one volatile organic HAP run conducted per each semi-volatile organic HAP run). Method 0031 allows flexibility in the sample rate and sample time per trap pair to address sampling conditions; *e.g.*, lower sample rate and total volume collected per trap pair under conditions when less than maximum detection ability is needed. Additional trap pairs may be taken (*e.g.*, fourth pair) and retained as back/up archive set in case of breakage or other loss.
- ³ Target analytes are given in Table 1.4; use either SW-846 Method 8270C or 8270D for the analysis of Method 0010 samples. Follow SW-846 Method 3542 for Method 0010 sample train components extraction and preparation for analyses. This procedure will result in three separate sample fractions for analyses – the front half (filter and front half rinse), the back half (XAD-2 and back half rinse), and condensate (first impinger and impinger rinse). You may combine the three analytical fractions collected in the Method 0010 sampling train and analyze as one sample. If you choose this option rather than analyzing the fractions separately, you must track the various fractional extraction and condensate volumes carefully in order to calculate the relative in-stack concentrations correctly. You may use acetone followed by MeCl for the solvent rinse in lieu of the 50/50 MeCl/MeOH rinse (*i.e.*, no liquid/liquid extraction). In addition, the analytical finish for either method should also employ high resolution gas chromatography and either low resolution or high resolution mass spectrometry with selective ion monitoring (SIM) specifically for targeted polycyclic aromatic hydrocarbons (PAH).
- ⁴ Target analytes are given in Table 1.5; test runs must occur during the test runs for speciated volatile and speciated semi-volatile organic HAP.
- ⁵ Target analytes for C1 through C5 hydrocarbons are given in Table 1.6; test runs must occur during the test runs for speciated volatile and speciated semi-volatile organic HAP.
- ⁶ If a unit has a certified CEMS installed for CO, you may report hourly averages from 30 days of CEMS data for the unit in lieu of conducting a CO stack test (same 30 days for which process data are provided). In order to correlate these emissions with other stack test emissions, a portion of the CEMS data submitted should contain the emissions data collected during performance of the other requested stack tests and hourly O₂ concentrations should also be provided, if continuously monitored. The CEMS must meet the requirements of Performance Specification 4 and 40 CFR 60.13 or the CEMS accuracy and ongoing QA/QC requirements of 40 CFR part 60, subpart J or Ja. Also provide the data of the most recent CEMS accuracy certification.
- ⁷ Just the 12 “dioxin-like” PCB congeners (IUPAC Numbers PCB-77, -81, -105, -114, -118, -123, -126, -156, -157, -167, -169, and -189)
- ⁸ Metals to be tested include antimony (Sb), arsenic (As), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), lead (Pb), manganese (Mn), nickel (Ni), and selenium (Se).
- ⁹ Conduct either the test for PM/PM_{2.5} (filterable) from stacks without entrained water droplets or the PM/PM_{2.5} (filterable) from stacks with entrained water droplets (with the TDS and TSS analysis), whichever is appropriate for your stack.
- ¹⁰ Also report scrubber recirculation liquid flow rate in liters/min.
- ¹¹ If a unit has a certified CEMS installed for NO_x and/or SO₂, you may report hourly averages from 30 days of CEMS data for the unit in lieu of conducting a NO_x and/or SO₂ stack test. Also provide hourly O₂ concentrations for the 30 days of CEMS data. In order to correlate these emissions with other stack test emissions, a portion of the CEMS data submitted should contain the emissions data collected during performance of the other requested stack tests. The CEMS must meet the requirements of Performance Specification 2 and 40 CFR 60.13 or the CEMS accuracy and ongoing QA/QC requirements of 40 CFR part 60, subpart J or Ja. Also provide the data of the most recent CEMS accuracy certification.
- ¹² If a unit has a certified CEMS installed for TRS, you may report hourly averages from 30 days of CEMS data for the unit in lieu of conducting a TRS stack test. In order to correlate these emissions with other stack test emissions, a portion of the CEMS data submitted should contain the emissions data collected during performance of the other requested stack tests. The CEMS must meet the requirements of Performance Specification 5 and 40 CFR 60.13 or the CEMS accuracy and ongoing QA/QC requirements of 40 CFR part 60, subpart J or Ja. Also provide the data of the most recent CEMS accuracy certification.

NOTE: Special criteria apply for simultaneous sampling, sampling run time, and sample volumes for the semi-continuous and batch processes related to the delayed coking unit and cyclic and semi-regenerative catalytic reforming units. Sampling should be conducted over the duration of the venting cycle. For vent cycles lasting less than 6 hours, testing over a vent cycle constitutes a single test run. For venting cycles shorter than the specified sampling time (or insufficient to allow collection of the required sample volume), sampling over the duration of the vent cycle

shall constitute an acceptable test run (not including continuous catalytic reforming purge vents). For semi-regenerative catalytic reforming units (only one cycle), the anticipated venting cycle should be divided into three roughly equivalent periods, and testing over each period would constitute a single run so that the three test runs would cover the duration of the entire venting event. Generally, you will need to apply U.S. EPA Method 1A in sampling these locations. If the venting cycles for specific catalytic reforming purge vents are very short, contact Brenda Shine (see Section 4.0 of this Part VIII) to see if performing a single test run is acceptable or if, for your unit, this purge vent does not need to be tested. For continuous catalytic reforming units, only small aliquots of catalyst are separated and purged prior to transfer to the regenerator portion at a given time, so these purges may be very short. For continuous catalytic reforming units, you should sample over numerous catalyst transfer aliquots to obtain a sample for 1 hour of active atmospheric purging as a single test run. Contact the appropriate EPA representatives listed in Section 4.0 of this Part VIII to request a variance to 1 hour sampling requirement if the catalyst transfer cycle is such that 1 hour of active atmospheric purging would take longer than 8 hours to obtain.

Table 1.3: Target Volatile Analytes

Compound	CAS Number	Compound	CAS Number
Acetone ¹	67-64-1	Methyl t-butyl ether ²	91-20-3
Acetonitrile	75-05-8	Methylene chloride	75-09-2
Acrolein ¹	107-05-8	Naphthalene	91-20-3
Acrylonitrile	107-13-1	Nitrobenzene	98-95-3
Benzene	71-43-2	2-Nitropropane	79-46-9
1,3-Butadiene ²	106-99-0	Pentane ²	109-66-0
Carbon disulfide	75-15-0	Styrene	100-42-5
Chlorobenzene	108-90-7	Tetrachloroethene	127-18-4
Cumene (isopropylbenzene)	98-82-8	Toluene	108-88-3
1,2-Dibromoethane	106-93-4	Trichloroethene	79-01-6
Ethylbenzene	100-41-4	Triethylamine	121-44-8
Hexane ²	110-54-3	2,2,4 Trimethylpentane ²	540-84-1
Methanol ¹	67-56-1	Xylenes (mixed isomers)	1330-20-7
Methyl isobutyl ketone	108-10-1		

¹ Analyte is not applicable to SW-846 Method 0031 sampling procedure. Collection of samples for these compounds is required only for hydrogen plant vent and requires collection in tedlar bags or evacuated canisters.

² Not specifically listed as an analyte for SW-846 Method 8260B.

Table 1.4: Target Semi-volatile Analytes

Compound	CAS Number	Compound	CAS Number
Acenaphthene	83-32-9	Dimethylaminobenzene	60-11-7
Acenaphthylene	208-96-8	7,12-Dimethylbenz(a)anthracene	57-97-6
Aniline	62-53-3	3,3'-Dimethylbenzidine	119-93-7
Anthracene	120-12-7	α,α -Dimethylphenethylamine	122-09-8
Benzidine ¹	92-87-5	2,4-Dimethylphenol	105-67-9
Benz[a]anthracene	56-55-3	Fluoranthene	206-44-0
Benzo[b]fluoranthene	205-99-2	Fluorene	86-73-7
Benzo[k]fluoranthene	207-08-9	Indeno(1,2,3-cd)pyrene	193-39-5
Benzo[g,h,i]perylene	191-24-2	Isophorone	78-59-1
Benzo[a]pyrene	50-32-8	3-Methylcholanthrene	56-49-5
Benzo[e]pyrene ²	192-97-2	2-Methylnaphthalene	91-57-6
Biphenyl ²	92-52-4	Naphthalene	91-20-3
Cresol (mixed isomers)	1319-77-3	Perylene ²	198-55-0
Chrysene	218-01-9	Phenanthrene	85-01-8
Dibenz[a,h]anthracene	53-70-3	Phenol	108-95-2
Dibenzofuran	132-64-9	1,4-Phenylenediamine	106-50-3
Dibenzo(a,e)pyrene	192-65-4	Pyrene	129-00-0
3,3'-Dimethoxybenzidine	119-90-4	o-Toluidine	95-53-4

¹ Notes non-reproducible chromatographic performance in SW-846 Method 8270D

² Not specifically listed as an analyte for SW-846 Method 8270D

Table 1.5: Aldehydes

Compound	CAS Number
Formaldehyde	100-41-4
Acetaldehyde	106-99-0
Propanal	123-38-6

Table 1.6: C1 through C5 Hydrocarbons

Description	Compound	CAS No.
C1 Alkanes	Methane	74-82-8
C2 Alkanes	Ethane	74-84-0
C3 Alkanes	Propane	74-98-6
C4 Alkanes	n-Butane	106-97-8
	Isobutane	75-28-5
C5 Alkanes	n-Pentane	109-66-0
	2-Methylbutane	78-78-4
	Cyclopentane	287-92-3
C2 Olefins	Ethylene	74-85-1
C2 Alkynes	Acetylene	74-86-2
C3 Olefins	Propylene	115-07-1
C4 Olefins	1-Butene	106-98-9
	2-Butene	107-01-7
	Isobutene	115-11-7
C5 Olefins	1-Pentene	109-67-1
	Cis-2-pentene	627-20-3
	Trans-2-pentene	646-04-8
	2-Methyl-1-butene	563-46-2
	3-Methyl-1-butene	563-45-1
	2-Methyl-2-butene	513-35-9
	Cyclopentene	142-29-0
C3 Alkadienes	Propadiene	463-49-0
C4 Alkadienes	1,2-Butadiene	590-19-2
	1,3-Butadiene	106-99-0
C5 Alkadienes	1,2-Pentadiene	591-95-7
	1-cis-3-Pentadiene	1574-41-0
	1-trans-3-Pentadiene	2004-70-8
	1,4-Pentadiene	591-93-5
	2,3-Pentadiene	591-96-8
	3-Methyl-1,2-butadiene	598-25-4
	2-Methyl-1,3-butadiene	78-79-5
	Cyclopentadiene	542-92-7

1.2 Fuel Gas Analysis Methods

Table 1.7 describes the tests and procedures required for the analysis of your fuel gas. Triplicate grab samples are to be taken for each mix drum associated with each fuel gas system. In addition to providing the results of the tests indicated in this section, you should also indicate whether each fuel gas system you tested includes gas from a coking unit.

You will find copies of EPA methods at <http://www.epa.gov/ttn/emc/tmethods.html>. You can find instructions on how to obtain ASTM methods at <http://www.astm.org/>.

Table 1.7: Summary of Test Methods and Alternative Methods for Organic Compounds, Reduced Sulfur, and Other Parameters for Refinery Fuel Gas

Pollutant	Recommended Method	Alternative Method	Target Reported Units of Measure¹
C1 through C5 hydrocarbons plus benzene ²	ASTM D1945-03. Use a minimum sample time of 2 hours per run.	U.S. EPA Method 18; U.S. EPA Method 320; ASTM D1946-90; GPA 2261-00; ASTM/UOP539-97	lb/hr and ppmvd
H ₂ S, COS, CS ₂ ³	U.S. EPA Method 15. Per method, a minimum of 16 individual samples during a period of 3 to 6 hours, per run.		lb/hr and ppmvd
TRS	U.S. EPA Method 15A or 16A; 3-hr sample time per run; (one 2-hr sample per run or three 40-minute samples per run)	U.S. EPA Method 16B combined with the analysis procedures U.S. EPA Method 15.	ppmvd
Inorganics/ other ⁴	ASTM D1945-03	ASTM D1946-90; GPA 2261-00; ASTM/UOP 539-97	
Higher Heating Value	<p>Any of the following methods, as appropriate:</p> <p>ASTM D240-02 (Reapproved 2007), Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter;</p> <p>ASTM D1826-94 (Reapproved 2003), Standard Test Method for Calorific (Heating) Value of Gases in Natural Gas Range by Continuous Recording Calorimeter;</p> <p>ASTM D3588-98 (Reapproved 2003), Standard Practice for Calculating Heat Value, Compressibility Factor, and Relative Density of Gaseous Fuels</p> <p>ASTM D4809-06, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method)</p> <p>ASTM D4891-89 (Reapproved 2006), Standard Test Method for Heating Value of Gases in Natural Gas Range by Stoichiometric Combustion</p> <p>Gas Processors Association Standard 2172-09, Calculation of Gross Heating Value, Relative Density, Compressibility and Theoretical Hydrocarbon Liquid Content for Natural Gas Mixtures for Custody Transfer</p> <p>Calculated based on detailed gas compositional analysis using: ASTM D1945-03, Standard Method for Analysis of Natural Gas by Gas Chromatography; ASTM D1946-90 (Reapproved 2006), Standard Method for Analysis of Reformed Gas by Gas Chromatography; GPA 2261-00, Analysis for Natural Gas and Similar Gaseous Mixtures by Gas Chromatography; or ASTM/UOP 539-97, Refinery Gas Analysis by Gas Chromatography</p> <p>Any of the methods specified in section 2.2.7 of Appendix D to part 75</p>		MMBtu/scf
Gas flow rate	ASME MFC-3M, -4M, -6M, -7M, -11M, -14M, or -18M; or AGA Report No. 11 (Measurement of Natural Gas by Coriolis Meter)	U.S. EPA Method 2, 2A, 2B, 2C, 2D, 2F, or 2G, as appropriate	acfm, scfm, and dscfm

Pollutant	Recommended Method	Alternative Method	Target Reported Units of Measure ¹
Moisture	U.S. EPA Method 4	None	percent volume

¹ lb/hr = pounds per hour; ppmvd = parts per million by volume, dry basis; MMBtu/scf = million British thermal units per standard cubic foot; acfm = actual cubic feet per minute; scfm = standard cubic feet per minute; dscfm = dry standard cubic feet per minute.

² Target analytes are given in Table 1.6, but include benzene (CAS No. 71-43-02) as a specific analyte.

³ If your fuel gas system has a certified continuous monitoring system (CMS) installed to measure H₂S concentrations, you may report daily averages from 30 days of data from that CMS in lieu of conducting a H₂S test. In order to correlate these emissions with other emissions tests, a portion of the monitoring data submitted should contain the data collected during performance of the other requested tests. The CMS must meet the requirements of Performance Specification 7 or the accuracy and ongoing QA/QC requirements of 40 CFR part 60, subpart J or Ja.

⁴ Target analytes are given in Table 1.8.

Table 1.8: Analytes for “Inorganics/Other” in Fuel or Flare Gas

Compound	CAS No.
Hydrogen	1333-74-0
Carbon monoxide	630-08-0
Carbon dioxide	124-38-9
Nitrogen	7727-37-9
Oxygen	7782-44-7

1.3 Sampling and Analysis Methods for Cooling Water Systems

Collect a sample from each cooling water system return line prior to exposure to air using “Air Stripping Method (Modified El Paso Method) for Determination of Volatile Organic Compound Emissions from Water Sources.”^a

Table 1.9 describes the tests and procedures required for the analysis of your cooling water system sample. You will find copies of EPA methods at <http://www.epa.gov/ttn/emc/tmethods.html>. You can find instructions on how to obtain Standard Methods for the Examination of Water and Wastewater at <http://www.standardmethods.org/>.

Table 1.9: Summary of Test Methods for Analysis via Texas El Paso Stripping Column

Pollutant	Recommended Method	Alternative Method	Target Reported Units of Measure ¹
Speciated Volatile Organic HAP ²	Standard Methods 6200 for water U.S. EPA Method TO-15 for stripping air	U.S. EPA Method TO-14A for stripping air	ppmvd in air (as measured) and ppmw in cooling water
Speciated Semi-volatile Organic HAP ³	Standard Methods 6410 for water U.S. EPA Method TO-13A for stripping air	None	ppmvd in air (as measured) and ppmw in cooling water
Formaldehyde	U.S. EPA Method TO-5 for stripping air.	U.S. EPA Method TO-11A for stripping air	ppmvd in air (as measured) and ppmw in cooling water (back-calculated)
THC	U.S. EPA Method TO-12 for stripping air.	None	ppmvd in air (as measured) and ppmw in cooling water (back-calculated)
CH ₄	U.S. EPA Method 18.	U.S. EPA Method 320	ppmvd in air (as measured) and ppmw in cooling water (back-calculated)
HCl, Cl ₂	U.S. EPA Method 26	U.S. EPA Method 26A	ppmvd in air (as measured) and ppmw in cooling water (back-calculated)

¹ ppmvd = parts per million by volume, dry basis; ppmw = parts per million by weight

² Target analytes are given in Table 1.3.

³ Target analytes are given in Table 1.4.

^a “Air Stripping Method (Modified El Paso Method) for Determination of Volatile Organic Compound Emissions from Water Sources” Revision Number One, dated January 2003, Sampling Procedures Manual, Appendix P: Cooling Tower Monitoring, prepared by Texas Commission on Environmental Quality, January 31, 2003.

1.4 Sampling and Analysis Methods for Enhanced Biodegradation Units (EBU)

Table 1.10 lists the procedures required for the analysis of your EBU(s). You will find copies of EPA methods at <http://www.epa.gov/ttn/emc/tmethods.html> and copies of EPA SW-846 methods at <http://www.epa.gov/waste/hazard/testmethods/sw846/online/index.htm>. You can find instructions on how to obtain Standard Methods for the Examination of Water and Wastewater at <http://www.standardmethods.org/>.

1.4.1 Sampling Methods

You should follow the sampling methods described in the EPA document “Handbook for Sampling and Sample Preservation of Water and Wastewater” (EPA-600/4-82-029). Additionally, you should conduct sample collection and preservation in accordance to 40 CFR Part 136, which can be accessed at: http://www.epa.gov/region9/qa/pdfs/40cfr136_03.pdf.

The sample chain-of-custody should also include the volumetric flow (m^3/s), pH, and temperature of each sample. You should include this information with the results of this study.

1.4.2 Sampling Location

Influent: One (1) grab sample of each influent stream to the EBU

EBU: One (1) grab sample within ten (10) feet of each influent location

Effluent: One (1) grab sample of each effluent stream of the EBU

1.4.3 Sampling Times

The sampling and testing events (*e.g.*, well-mixed assessment and F_{bio} determination) should occur at the same time and during normal operating conditions.

Table 1.10: Summary of Analytical Methods and Alternative Methods for Wastewater Treatment Systems

Pollutant	Recommended Method	Alternative Method	Target Reported Units of Measure ¹
Speciated Volatile Organic HAP ²	SW-846 Method 8260B	Standard Methods 6200	µg/L
Speciated Semi-volatile Organic HAP ³	SW-846 Method 8270C or D	Standard Methods 6410	µg/L
Aldehydes ⁴	SW-846 Method 8315A	Standard Methods 6252	µg/L
Sulfide (as S)	EPA Method 376.1	Standard Methods 4500-S ²⁻	µg/L
BOD ₅ ⁵	EPA Method 405.1	Standard Methods 5210	mg/L
COD ⁵	EPA Method 410.1	Standard Methods 5220	mg/L
Well-mixed assessment	Technical Support Document for Evaluation of Thoroughly Mixed Biological Treatment Units, 1998. ⁶	None	N/A
Site-specific biodegradation rates	Appendix C to 40 CFR Part 63 – Aerated reactor batch test with oxygen addition (BOX test). Do one test using benzene as the sole substrate and perform a second test using benzene, toluene, xylene, and naphthalene at equal concentrations.	Technical Support Document for the Evaluation of Aerobic Biological Treatment Units with Multiple Mixing Zones. ⁷	(F _{bio})

¹ µg/L = micrograms per liter; mg/L = milligrams per liter; F_{bio} = site-specific fraction of organic compounds biodegraded

² Target analytes are given in Table 1.3.

³ Target analytes are given in Table 1.4.

⁴ Target analytes are given in Table 1.5.

⁵BOD₅ = 5-day biological oxygen demand; COD = chemical oxygen demand.

⁶ A copy of this document is located at <http://www.epa.gov/ttncaaa1/t3/reports/guidfn.pdf>.

⁷ A copy of this document is located at <http://www.epa.gov/ttn/caaa/t3/reports/mulmix5.pdf>.

2.0 How to Report Data

The method for reporting the results of any testing and monitoring requests depend on the type of tests and the type of methods used to complete the test requirements. This section discusses the requirements for reporting the data.

2.1 Reporting stack test data

If you conducted testing using one of the methods listed in Table 2.1 you must report your data using the EPA Electronic Reporting Tool (ERT) Version 3. ERT is a Microsoft® Access database application. Two versions of the ERT application are available. If you are not a registered owner of Microsoft® Access, you can install the runtime version of the ERT Application. Both versions of the ERT are available at http://www.epa.gov/ttn/chief/ert/ert_tool.html. The ERT supports a Microsoft® Excel

spreadsheet application (which is included in the files downloaded with the ERT) to document the collection of the field sampling data.

** After completing the ERT, you will also need to attach an electronic copy of the emission test report (PDF format preferred) to the Attachments module of the ERT.

Table 2.1: List of Test Methods Supported by ERT

EPA Test Methods (40 CFR parts 60 and 61; EMC website, http://www.epa.gov/ttn/emc/tmethods.html; and SW 846, http://www.epa.gov/waste/hazard/testmethods/sw846/online/index.htm)
Methods 1 through 4 (testing locations, velocity, moisture, dilution gases)
Method 3A (O ₂ and CO ₂)
Method 5 (filterable PM)
Method 6C (SO ₂)
Method 7E (NO _x)
Method 10 (CO)
Method 17 (in-stack filterable PM)
Method 23 (PCDD and PCDF)
Method 25A (THC)
Method 26A (HCl and HF)
Method 29 (multiple metals including Hg)
Method 101 (Hg)
Method 101A (Hg)
Method 201A (formerly other test method (OTM) 27, conditional test method (CTM) 40) (filterable PM _{2.5})
Method 202 (formerly other test method (OTM) 28, conditional test method (CTM) 39) (condensable PM)
CT Method 39
CT Method 40

At present, only the methods shown in Table 2.1 are supported by the ERT. If you conducted testing using a method not currently supported by the ERT, you must report the results of this test in the Refinery Testing Supplement. The Refinery Testing Supplement can be downloaded from the ICR website (<https://refineryicr.rti.org>). You must report the results of each test on the appropriately labeled form corresponding to the specific tests requested at your emissions source. If you conducted testing at more than one source at your refinery using methods not currently supported by the ERT, follow the Refinery Testing Supplement instructions on the ICR website (<https://refineryicr.rti.org>). After completing the Refinery Testing Supplement, you must also submit an electronic copy of the emission test report (PDF format preferred).

As noted in Section 1 of this document, if you have installed a CEMS or CMS on a unit that this ICR asks you to test, you may submit CEMS or CMS data to meet the testing requirement for that specific pollutant. If you have a CO Continuous Emissions Monitoring System (CEMS) that meets Performance Specification 4 (or other specified procedures) or a SO₂ and/or NO_x CEMS that meets Performance Specification 2 (or other specified procedures) installed on the exhaust of your emissions unit, and you used CEMS data to meet CO, SO₂ and/or NO_x test requirements as noted in the footnotes of specific tables, you must report 30 days of daily averages of qualified CEMS data in a Microsoft® Excel CEMS Template. Similarly, if you have an H₂S continuous

monitoring system (CMS) that meets Performance Specification 7 or a TRS continuous monitoring system (CMS) that meets Performance Specification 5, and you used CMS data to meet the H₂S or TRS testing requirements as noted in the footnotes of specific tables, you must report 30 days of daily averages of qualified CMS data in a Microsoft® Excel CMS Template. The 30 operating days for which you provide data can be any 30 consecutive days with at least one hour of qualified CEMS or CMS data between March 1 and August 31, 2011, that includes the days during which you conducted the testing for the other pollutants requested for that process unit by this ICR. The Excel spreadsheet templates are specific to each pollutant and type of emissions source, and they can be downloaded from the ICR website (<https://refineryicr.rti.org>).

2.2 Required Fields for ERT Reporting

The list of fields within the ERT with notes explaining whether or not the field is required or optional can be found on the ICR website (<https://refineryicr.rti.org>).

2.3 Calculating and Reporting Values Measured Below Detection Levels

See Appendix A to this Component 4 for guidance on calculating and reporting measurements less than detection levels for emissions data collection (ICR) programs.

3.0 How to Submit Data

As explained in the previous section, where applicable, you must report your data using the ERT. If the ERT does not support a particular pollutant or method, you must report your data using the data reporting tools we provide. When you are ready to submit your data to EPA, use one of three transmittal ways listed below. In order to avoid duplicate data and keep all data for a particular facility together, we request that you submit all of the data requested from your facility in the same way.* To submit your data:

- Use the ICR website to upload your files. Detailed directions for uploading your files are provided on the ICR website (<https://refineryicr.rti.org>).
- E-mail an electronic copy of all requested files to refineryicr@epa.gov.
- Mail a CD or DVD containing an electronic copy of all requested files to the EPA address shown in your Section 114 letter. If no electronic copy is available, mail a hard copy of all requested files to the EPA address shown in your Section 114 letter.

* **Please Note:** If you are submitting Confidential Business Information (CBI), **DO NOT** use one of the methods listed above to submit your CBI. You must mail a separate CD, DVD, or hard copy package containing only the CBI portion of your data to the EPA address shown in your Section 114 letter.

4.0 Contact Information for Questions on Test Plan and Reporting

For questions on how to report data using the ERT, contact:

Ron Myers
U.S. EPA
(919) 541-5407
myers.ron@epa.gov

OR

Barrett Parker
U.S. EPA
(919) 541-5635
parker.barrett@epa.gov

For questions on the test methods, including the use of alternative methods, contact:

Peter Westlin
U.S. EPA
(919) 541-1058
westlin.peter@epa.gov

OR

Robin Segall
U.S. EPA
(919) 541-0893
segall.robin@epa.gov

OR

Jason DeWees
U.S. EPA
(919) 541-9724
deweese.jason@epa.gov

For questions on the petroleum refinery test plan, including emissions sources selected for testing and reporting mechanisms other than the ERT, contact:

Brenda Shine
U.S. EPA
(919) 541-3608
shine.brenda@epa.gov

For questions on uploading files, please visit the ICR website (<https://refineryicr.rti.org>) and use the toll free technical support hotline or technical support email address.

Appendix A

Guidance For Calculating And Reporting Measurements Less Than In-Stack Method Detection Levels For Emissions Data Submitted in Response to Information Collection Request (ICR) Programs

Identify the status of measured values relative to detection levels on the spreadsheet or in the ERT using the following descriptions:

- **BDL** (below detection level) – all analytical values used to calculate and report an in-stack emissions value are less than the laboratory’s reported detection level(s);
- **DLL** (detection level limited) – at least one but not all values used to calculate and report an in-stack emissions value are less than the laboratory’s reported detection level(s); or
- **ADL** (above detection level) – all analytical values used to calculate and report an in-stack emissions value are greater than the laboratory’s reported detection level(s).

For each reported emissions value, insert the appropriate flag (BDL, DLL, or ADL) in the **Note** line of the Microsoft® Excel emission test spreadsheet template or in the **Comments** line of the Electronic Reporting Tool (ERT).

When reporting and calculating individual test run data:

- For analytical data reported from the lab as “nondetect” or “below detection level”:
 - Include a brief description of the procedures used to determine the analytical detection and in-stack detection levels:
 - o In the **Note** line of Excel emission test spreadsheet template; or
 - o In the **Comments** line of Lab Data tab in the Run Data Details in the **ERT**.
 - Describe these procedures completely in a separate attachment including the measurements made, the standards used, and the statistical procedures applied.
 - Calculate in-stack emissions rate for any analytical measurement below detection level using the relevant detection level as the “reported” value.
 - Report the calculated emissions concentration or rate result:
 - o As a bracketed “less than” detection level value (*e.g.*, [<0.0105]) in the Excel emission test spreadsheet template and include the appropriate flag in the **Note** line; or
 - o As a numerical value in the **ERT** with the appropriate general flag in the **Flag** column and as a bracketed “less than” detection level value (*e.g.*, [<0.0105]) followed by a space and any specific flag identifier in the **Comments** column. The following specific flag identifiers are commonly used by laboratories: B, E, J, Int, R, DPE.
 - Report as numerical values (*i.e.*, no brackets or $<$ symbol) any analytical data measured above the detection level, including any data between the analytical detection level and a laboratory-specific reporting or quantification level (*i.e.*, flag as ADL).
 - Apply these reporting and calculation procedures to measurements made with **Method 23**:

- Report data in the Excel emission test spreadsheet template for each of the D/F congeners measured with Method 23 below the detection level as [$<$ detection level]
 - Do **not report emissions as zero** as described in the method
- For pollutant measurements composed of multiple components or fractions (*e.g.*, Hg and other metals sampling trains) when the result for the value for any component is measured below the analytical detection level;
 - Calculate in-stack emissions rate or concentrations as outlined above for each component or fraction;
 - Sum the measured and detection level values as outlined above using the in-stack emissions rate or concentrations for all of the components or fractions; and
 - Report the sum of all components or fractions:
 - As a bracketed “less than” detection level value (*e.g.*, [<0.0105]) in the Excel emission test spreadsheet template and include the appropriate flag in the **Note** line; or
 - As a numerical value in the **ERT** with the appropriate flag in the **Comments** line.
 - Report also the individual component or fraction values for each run if the Excel emission test spreadsheet template or ERT format allows; if not (*i.e.*, the format allows reporting only a single sum value):
 - For the Excel emission test spreadsheet template, next to the sum reported as above, report in the **Notes** line the appropriate flag along with the values for the measured or detection level value for each component or fraction as used in the calculations (*e.g.*, 0.036, [<0.069], 1.239, [<0.945] for a four fraction sample)
 - For the **ERT**, next to the sum reported as above, report in the **Flag** column the appropriate general flag and in the **Comments** column the measured or detection level value for each component or fraction as used in the calculations followed by a space and any specific flag identifier provided by the laboratory (*e.g.*, 0.036, [<0.069] J, 1.239, [<0.945] B for a four fraction sample).
- For measurements conducted using instrumental test methods (*e.g.*, Methods 3A, 6C, 7E, 10, 25A)
 - Record gaseous concentration values **as measured** including negative values and flag as ADL; do not report as BDL
 - Calculate and report in-stack emissions rates using these measured values
 - Include relevant information relative to calibration gas values or other technical qualifiers for measured values in **Comments** line in the **ERT**
- When reporting and calculating average emissions rate or concentration for a test when some results are reported as BDL
 - Sum all of the test run values including those indicated as BDL or DLL as “real” values
 - Calculate the average emissions rate or concentration (*e.g.*, divide the sum by 3 for a three-run test)

- Report the average emissions rate or concentration average:
 - o As a bracketed “less than” detection level value (*e.g.*, [<20.06]) in the Excel emission test spreadsheet template and include the appropriate flag in the **Note** line
 - o As a numerical value in the **ERT** and include the appropriate flag in the **Comments** line.