

Petroleum Refinery Emissions Information Collection

PART VII. SUMMARY OF TEST PROCEDURES, METHODS, AND REPORTING REQUIREMENTS FOR DISTILLATION FEED COMPOSITION ANALYSIS

The EPA petroleum refinery test program is requesting variability data for the feed to the distillation column. This part provides an overview of approved methods, target pollutants and units of measure, and reporting requirements for the analysis of the feed to your distillation column or first processing unit.¹ All recipients must complete and submit test results by August 31, 2011.

The distillation feed composition analyses requested include: mercury, chlorine, sulfur, antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, nickel, and selenium.

You will need to collect and analyze samples of the feed to your distillation column(s) three times, each time approximately 30 days from the last. Samples must be collected early enough in the testing period to ensure you can provide results by August 31, 2011. For example, you may elect to collect the first sample during April, the second during May, and the third during June. Once you collect samples, composite the samples, and then analyze and report each composited sample. Directions for collecting, preparing, and analyzing the distillation feed samples are outlined below.

For the purposes of this request, you may elect either to sample the crude oil (or other petroleum stream first processed) from the storage tank that supplies the distillation column or to take samples from the line feeding the distillation column. For atmospheric crude distillation units with desalters, you may elect to sample either before or after the desalter; you must indicate where the sample was taken.

If you have multiple distillation columns and the feed to each is similar, you must collect samples from each distillation column and composite them so that only one sample is analyzed per month. If you have multiple distillation columns that receive intentionally different feed streams, then you must collect and analyze separate samples for each distillation column receiving different feed streams. For example, if you have three distillation columns and two have the same feed, you would collect samples from the first two and composite those into one sample to analyze each month, and you would also collect samples from the third distillation column and analyze those samples separately.

¹ The terms “distillation feed” or “feed to a distillation column” is used generically in this part to refer to the feed to the first major processing unit at the refinery. For most refineries, the distillation feed will be the feed to the atmospheric crude distillation unit. For refineries that do not have atmospheric crude distillation units, the distillation feed will be the feed to the vacuum distillation unit (if one is used). For refineries that do not have either an atmospheric crude distillation unit or a vacuum distillation unit, samples of the feed used in the first major processing unit should be used (*e.g.*, a refinery that receives only unfinished oils that are processed in a catalytic cracking unit and then further processed to make finished motor gasoline would sample the feed to the catalytic cracking unit).

1. How to collect a distillation feed sample: Table 1 outlines a summary of how samples should be collected. When sampling from storage tanks, remove a minimum of three discrete, equal-volume grab samples from different locations in the tank and composite these samples for subsequent analysis. If you collect samples from the feed line, you must collect a minimum of three equal-volume samples with a minimum of 5 minutes between samples; these samples should then be composited for subsequent analysis. If you have multiple distillation columns with similar feed, collect an equal number of equal-volume feed samples from each distillation column and composite all of the samples for analysis. In other words, if there are two distillation columns with similar feed, collect a minimum of two equal-volume samples from each distillation column to prepare the composite. If there are three or more distillation columns with similar feed, collect a minimum of one equal-volume sample from each distillation column to prepare the composite.

Table 1. Summary of Sample Collection Procedures

Sampling Location	Sampling Procedures
<i>Site-Specific Sampling and Analysis</i>	
Manual Sampling	Follow collection methods outlined in ASTM D4057
Automatic Sampling	Follow collection methods outlined in ASTM D4177
<i>Crude Oil Supplier Analysis</i>	
Crude Oil Supplier	If you only process one type of crude oil and your crude oil supplier will provide all of the requested information, the crude oil supplier must collect the sample as specified above and prepare the sample according to methods specified in Steps 2 and 3 of this part.

2. Prepare sample for analysis: To prepare your samples for mercury and selected metal analyses, use SW-846-3020A or any SW-846 sample digestion procedures giving measures of total metal. For other analyses, use the guidance in ASTM D7455-08 for appropriate sample preparation.
3. Analyzing distillation feed sample: Table 2 outlines a list of approved methods for analyzing distillation feed samples. 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 Component 4 for a list of persons to contact with questions regarding test methods and reporting.

Table 2. List of Analytical Methods for Distillation Feed Analysis

Analyte	Method	Target Reported Units of Measure
Higher Heating Value	ASTM-5865-03a, ASTM D240, ASTM E711-87 (1996)	Btu/lb
Density/API Gravity	ASTM D287-92, ASTM D1217-993, ASTM D1481-93, ASTM D4052-96	lb/ft ³ AND API Gravity
Mercury Concentration	SW-846-7470A, EPA Method 1631E, SW-846-1631E, SW-846-1631, EPA 821-R-01-013, or equivalent	ppm
Chlorine Concentration	ASTM E776-87 (1996, 2004, or 2009), SW-846-9250, SW-846-5050, -9056, -9076, or -9250	ppm
Sulfur Concentration	ASTM D129-00 (2005), ASTM D1552-01 or -08, ASTM D2622-98 or -10	ppm
Total Selected Metals Concentration	SW-846-6020, -6020A, , SW-846-6010B, SW-846-7740 for Se, SW-846-7060 or -7060A for As	ppm

4. Reporting distillation feed analysis data: You must report the analysis results in Table 3. The electronic version of Table 3 is included in the electronic ICR that can be downloaded from the ICR website (<https://refineryicr.rti.org>). The template also asks you to note if the crude oil or intermediate slate that makes up the feed to the distillation column during the time of your sample is significantly different than the crude oil or intermediate slate you used during 2010.

You must keep the following records for 3 years:

- Documentation that distillation feed samples were obtained in accordance with the above sampling protocol;
- Documentation of the proper chain of custody for each distillation feed sample;
- Description of the QA/QC procedures followed in preparing each distillation feed sample for analysis and performing the required analysis; and
- The results of the analyses performed on each distillation feed sample.

