

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 triplicate samples of the feed to your distillation column 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 collect the first sample during early April, the second during early May, and the third during early 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.

Note: If you have multiple distillation columns and the feed to each is similar (*i.e.*, the feed to one distillation column is representative of the feed to all distillation columns at the refinery), you may collect samples from just one distillation column. If you have multiple distillation columns and the feed to all of them is not the same, you must sample the feed from those that have significantly different feed streams. For example, if you have three distillation columns and two have the same feed, you may sample from one of those two as well as the third and then note that the samples from the first are representative of the second.

1. How to collect a distillation feed sample: Table 1 outlines a summary of how samples should be collected.

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).

Table 1. Summary of Sample Collection Procedures

Sampling Location	Sampling Procedures
Site-Specific Sampling and Analysis	
Manual Sampling	Follow collection methods outlined in ASTM D4057
Automatic Sampling	Follow collection methods outlined in ASTM D4177
Crude Oil Supplier Analysis	
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.

Table 3. Distillation Feed Sample and Analysis Results

Sample Date	Unit ID for Distillation Column	Higher Heating Value (Btu/lb)	Density (lb/ft ³)	API Gravity	Concentrations (ppm)											Unit IDs for Other Distillation Column(s) with Feed	If the crude oil or intermediate slate being fed to the distillation column during the time of this sample is significantly different than the crude oil or intermediate slate used during 2010, briefly				
					Mercury	Chlorine	Sulfur	Antimony	Arsenic	Beryllium	Cadmium	Chromium	Cobalt	Lead	Manganese			Nickel	Selenium		