

## TIER 3 MOTOR VEHICLE FUEL STANDARDS FOR DENATURED FUEL ETHANOL



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### Background:

The U.S. Environmental Protection Agency (EPA) finalized the Tier 3 Vehicle Emission and Fuel Standards Program a rule designed to reduce air pollution from passenger cars and trucks. Starting in 2017, Tier 3 sets new vehicle emissions standards and lowers the sulfur content of gasoline, considering the vehicle and its fuel as an integrated system. The vehicle standards reduce both tailpipe and evaporative emissions from passenger cars, light-duty trucks, medium-duty passenger vehicles, and some heavy-duty vehicles. The gasoline sulfur standard will make emission control systems more effective for both existing and new vehicles, and will enable more stringent vehicle emissions standards since removing sulfur allows the vehicle's catalyst to work more efficiently. The Tier 3 standards are closely coordinated with California's Low Emission Vehicle (LEV III) standards as well as with EPA's and California's programs for GHG emissions from light-duty vehicles. EPA is setting these Tier 3 standards to address public health issues that exist currently and are projected to continue in the future. This document is intended to help ethanol producers comply with the new rules.

### A new definition and acronym:

**Denatured Fuel Ethanol (DFE)** means an alcohol of the chemical formula  $C_2H_6O$  which contains a denaturant to make it unfit for human consumption, that is produced or imported for use in motor gasoline

### Sampling methods for gasoline, diesel fuel, fuel additives, and renewable fuels

The sampling methods specified shall be used to collect samples;

**Manual sampling** of tanks and pipelines shall be performed according to the applicable procedures specified in ASTM D4057-12, Standard Practice for Manual Sampling of Petroleum and Petroleum Products, approved December 1, 2012.

**Automatic sampling** of petroleum products in pipelines shall be performed according to the applicable procedures specified in ASTM D4177-95 (Reapproved 2010), Standard Practice for Automatic Sampling of Petroleum and Petroleum Products, approved May 1, 2010.

**Composite samples** shall be prepared using the applicable procedures specified in ASTM D5854-96 (Reapproved 2010), Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products, approved May 1, 2010.

**Testing methods for sulfur** *other test methods listed if needed.*

**ASTM D5453-12**, Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence, approved November 1, 2012.

**ASTM D7039-13**, Standard Test Method for Sulfur in Gasoline, Diesel Fuel, Jet Fuel, Kerosene, Biodiesel, Biodiesel Blends, and Gasoline-Ethanol Blends by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry, approved September 15, 2013.

**Subpart O—Gasoline Sulfur**

**Beginning January 1, 2017**, producers and importers of DFE or other oxygenates designated for use in transportation fuel;

- ✓ Sulfur content must not be greater than 10 ppm
- ✓ DFE or other oxygenate must be composed solely of carbon, hydrogen, nitrogen, oxygen and sulfur
- ✓ Only previously certified gasoline (including previously certified BOBs), gasoline blendstocks, or natural gas liquids may be used as denaturants
- ✓ Concentration of all denaturants used in DFE is limited to a maximum of 3.0 volume percent

**Product Transfer Document (PTD)** Required on each occasion when any person transfers custody or title to any oxygenate upstream of any oxygenate blending facility, the transferor shall provide to the transferee product transfer documents include the following information:

- ✓ “Denatured fuel ethanol, maximum 10 ppm sulfur.”

**Batch Numbers** Every batch of oxygenate produced or imported shall be assigned a batch number consisting of the EPA-assigned oxygenate producer or importer registration number, the EPA facility registration number, the last two digits of the year in which the batch was produced, and a unique number for the batch, beginning with the number one for the first batch produced or imported each calendar year and each subsequent batch during the calendar year being assigned the next sequential number (e.g., 4321-54321-95-000001, 4321-54321-95-000002, etc.). An alternative batch numbering protocol may be used as approved by the Administrator.

**Annual Reports** Submit annual reports to EPA pursuant to the reporting requirements

## Sampling and testing requirements for producers and importers of denatured fuel ethanol and other oxygenates for use by oxygenate blenders

**Beginning January 1, 2017**, producers and importers of DFE and other oxygenates for use by oxygenate blenders must satisfy the sampling and testing requirements;

- ✓ Producers and importers of oxygenates for use by oxygenate blenders shall collect a representative sample from each batch of oxygenate produced or imported prior to the oxygenate leaving the facility, using the sampling methods specified
- ✓ Producers and importers of oxygenates must test each batch of oxygenate they produce or import to determine its sulfur content to the nearest ppm using a test method specified or, with respect to DFE may use the alternative means of determining the sulfur content

**Alternative means of determining the sulfur content of DFE** As an alternative to testing each batch of DFE, the sulfur content of batches of DFE produced using certified denaturant meeting the requirements may be determined as follows:

- ✓ The sulfur content of the batch of DFE shall be calculated by volume weighting the sulfur contribution from the denaturant), and the neat ethanol used
- ✓ The sulfur content of the neat (undenatured) ethanol used in the calculation may be assumed to be negligible or assumed to be some specific value for the purposes of calculating the sulfur content of the DFE batch provided that the DFE manufacturer or importer conducts production quality control which demonstrates that such an assumption is valid
- ✓ The sulfur content of the certified denaturant used in the calculation must be consistent with the PTD obtained from a registered certified ethanol denaturant producer or importer
- ✓ If the PTD from the certified ethanol denaturant states that the sulfur content is 330 ppm, then the sulfur content of the ethanol denaturant must be assumed to be 330 ppm
- ✓ A sample of each batch of DFE must be retained
- ✓ The sulfur content of each batch of DFE shall be reported to the nearest ppm

## Standards and requirements for certified ethanol denaturant

Producers and importers of ethanol denaturant that is suitable for the manufacture of DFE may designate the denaturant as certified ethanol denaturant if the following requirements are met;

- ✓ The sulfur content must not be greater than 330 ppm as determined in accordance with the test requirements
- ✓ If the denaturant manufacturer represents a batch of denaturant as having a sulfur content of less than 330 ppm in the PTD, then the actual sulfur content must be no greater than the stated value

**Test Requirements** Beginning January 1, 2017, or the first date that certified ethanol denaturant is introduced into commerce, whichever is earlier, sample and test each batch of gasoline and certified ethanol denaturant. Producers and importers of certified ethanol denaturant shall collect a representative sample from each batch of certified ethanol denaturant produced or imported and test each sample to determine its sulfur content for compliance with requirements.

**Registration** Unless registered under other programs the producer or importer of ethanol denaturant must register with EPA

**Product Transfer Document (PTD)** Required on each occasion when any person transfers custody or title to any ethanol denaturant designated as suitable for the production of DFE meeting federal quality requirements upstream of a DFE production or import facility, the transferor shall provide to the transferee product transfer documents which include all the following information;

- ✓ “Certified Ethanol Denaturant suitable for use in the manufacture of denatured fuel ethanol meeting EPA standards.”
- ✓ If the certified ethanol denaturant manufacturer represents that a batch of ethanol denaturant has sulfur content less than 330 ppm, then either the actual sulfur content of the denaturant must be clearly stated on the PTD, or the PTD must state the sulfur content is 330 ppm or less.

**Batch Numbers** Every batch of certified ethanol denaturant produced or imported shall be assigned a batch number consisting of the EPA-assigned oxygenate producer or importer registration number, the EPA facility registration number, the last two digits of the year in which the batch was produced, and a unique number for the batch, beginning with the number one for the first batch produced or imported each calendar year and each subsequent batch during the calendar year being assigned the next sequential number (e.g., 4321-54321-95-000001, 4321-54321-95-000002, etc.)

### Sample retention requirements for oxygenate producers and importers

Beginning January 1, 2017, any producer or importer of oxygenate shall do all the following:

- ✓ Retain a representative portion of each sample analyzed of at least 330 milliliters in volume
- ✓ Retain a representative sample of each batch of DFE for which the producer or importer used the alternative means of determining the sulfur contents of the DFE batch of at least 330 milliliters in volume
- ✓ Retain sample portions for the most recent 20 samples collected, or for each sample collected during the most recent 21 day period, whichever is greater, not to exceed 90 days for any given sample
- ✓ Comply with the DFE sample handling and storage procedures under each sample portion retained. Shall collect a representative sample from each batch of oxygenate produced or imported prior to the oxygenate leaving the oxygenate production or import facility
- ✓ Comply with any request by EPA to provide a retained sample portion to the Administrator's authorized representative; ship a retained sample portion to EPA, within two working days of the date of the request, by an overnight shipping service or comparable means, to the address and following procedures specified by EPA, and accompanied with the sulfur test result for the sample determined or the calculated sulfur content of the batch from which the sample was drawn

### Product transfer document requirements

**Ethanol denaturant** Each occasion when any person transfers custody or title to any ethanol denaturant designated as suitable for use in the manufacture of denatured fuel ethanol meeting federal quality requirements the transferor shall provide to the transferee documents which include all the following information:

- ✓ The name and address of the transferor
- ✓ The name and address of the transferee
- ✓ The volume of ethanol denaturant which is being transferred
- ✓ The location of the ethanol denaturant at the time of the transfer
- ✓ The date of the transfer
- ✓ A statement identifying the batch as "Ethanol denaturant suitable for the manufacture of denatured fuel ethanol meeting federal quality requirements"
- ✓ Information on the sulfur content of the ethanol denaturant

**DFE or other Oxygenate** Each occasion when any person transfers custody or title to any oxygenate, the transferor shall provide to the transferee documents which include all the following information:

- ✓ The name and address of the transferor
- ✓ The name and address of the transferee
- ✓ The volume of oxygenate which is being transferred
- ✓ The location of the oxygenate at the time of the transfer
- ✓ The date of the transfer
- ✓ For denatured fuel ethanol, a statement identifying the batch as “Denatured fuel ethanol, maximum 10 ppm sulfur”
- ✓ For oxygenates other than DFE, the name of the specific oxygenate must be identified on the PTD, followed by “maximum 10 ppm sulfur”

### Reporting requirements for gasoline refiners, gasoline importers, oxygenate producers, and oxygenate importers

**DFE or other Oxygenate producer or importer annual reports** any oxygenate producer, for each of its production facilities, and any importer for the oxygenate it imports, shall submit a report for each calendar year period that includes all the following information:

- ✓ The EPA oxygenate importer, or producer and producer facility registration numbers
- ✓ The total volume of oxygenate produced or imported, reported to the nearest whole number
- ✓ For each batch of oxygenate produced or imported during the calendar year, all the following:
  - The batch number assigned
  - The date the batch was produced
  - The volume of the batch, reported to the nearest whole number
  - The sulfur content of the batch, reported to two decimal places
  - For oxygenates other than denatured fuel ethanol, the identification of the test method used to determine the sulfur content of the batch
  - For denatured fuel ethanol, either the identification of the test method used to determine the sulfur content of the batch or the information used to calculate the sulfur content

**Report submission** Annual report required shall be signed and certified as meeting all of the applicable requirements of Subpart O by the owner or a responsible corporate officer of the refiner, gasoline importer, oxygenate producer, oxygenate importer, denaturant producer, or denaturant importer and submitted to EPA no later than the March 31 each year for the prior calendar year.

## Recordkeeping

**Records that producers and importers of denatured fuel ethanol and other oxygenates must keep** Beginning January 1, 2017 or the first date when DFE is introduced into commerce that is represented on the product transfer document as meeting the standards (whichever is earlier), records of all the following must be kept for each batch of oxygenate produced or imported by oxygenate producers and importers:

- ✓ The date the batch was produced
- ✓ The batch number
- ✓ The batch volume
- ✓ The product transfer document for the batch
- ✓ The sulfur content of the batch as determined
- ✓ The following records shall be kept if the sulfur content of the batch was determined by analytical testing:
  - The location, date, time, and storage tank or truck identification for each sample collected
  - The name and title of the person who collected the sample and the person who performed the test
  - The results of the test as originally printed by the testing apparatus, or where no printed result is produced, the results as originally recorded by the person who performed the test
  - Any record that contains a test result for the sample that is not identical to the result recorded
  - The test methodology used

For DFE, the following records shall be kept if the sulfur content of the batch was determined by the alternative means of demonstrating compliance with the sulfur requirements;

- ✓ The name and title of the person who calculated the sulfur content of the batch
- ✓ The date the calculation was performed
- ✓ The calculated sulfur content
- ✓ The sulfur content of the neat (undenatured) ethanol
- ✓ The date each batch of neat ethanol was produced
- ✓ The neat ethanol batch number

- ✓ The neat ethanol batch volume
- ✓ As applicable, the neat ethanol production quality control records, or the test results on the neat ethanol including:
  - The location, date, time, and storage tank or truck identification for each sample collected
  - The name and title of the person who collected the sample and the person who performed the test
  - The results of the test as originally printed by the testing apparatus, or where no printed result is produced, the results as originally recorded by the person who performed the test
  - Any record that contains a test result for the sample that is not identical to the result recorded
  - The test methodology used
- ✓ The sulfur content of the denaturant(s) used, and the volume percent at which the denaturant(s) were added to neat (undenatured) ethanol to produce denatured fuel ethanol
- ✓ Beginning January 1, 2017, all parties that take custody of denaturants designated as suitable for use in the manufacture of DFE must keep the following records:
  - The product transfer document for the denaturant.
  - As applicable, the volume percent at which the denaturant was added to neat ethanol.

### Performance-based Analytical Test Method Approach

*This statute is for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the sulfur standard but placed here for reference. It is not expected that DFE production facilities will invest in the equipment and personnel to perform analytical testing of sulfur.*

All sample handling, testing procedures, and tests must be conducted using these good laboratory practices;

Performance-based Analytical Test Method Approach means a measurement system based upon established performance criteria for accuracy and precision with use of analytical test methods. This is a measurement system used by laboratories to demonstrate that a particular analytical test method is acceptable for demonstrating compliance.

### **Precision and accuracy criteria for approval for the absolute fuel parameter of gasoline sulfur**

**Precision** Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the sulfur standard the maximum allowable standard deviation computed from the

results of a minimum of 20 tests made over 20 days (seven or fewer tests per week and two or fewer tests per day) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 1.5 times the repeatability “r” divided by 2.77, where “r” equals the ASTM repeatability of ASTM D7039 (Example: A 10 ppm sulfur gasoline sample: Maximum allowable standard deviation of 20 tests  $\leq 1.5 * (1.75 \text{ ppm} / 2.77) = 0.95 \text{ ppm}$ ). The 20 results must be a series of tests with a sequential record of analysis and no emissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

**Accuracy** Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline sulfur standard;

- ✓ The arithmetic average of a continuous series of at least 10 tests performed using good laboratory practices on a commercially available gravimetric sulfur standard in the range of 1-10 ppm shall not differ from the accepted reference value (ARV) of the standard by more than 0.71 ppm sulfur
- ✓ The arithmetic average of a continuous series of at least 10 tests performed using good laboratory practices on a commercially available gravimetric sulfur standard in the range of 10-20 ppm shall not differ from the ARV of the standard by more than 1.00 ppm sulfur

**Qualification criteria for Voluntary Consensus Standard Based (VCSB) Method-Defined Parameter Test Methods** such as ASTM, or for a commercially available industry crosscheck program, the summary statistics (mean and standard error = standard deviation/square root [number of results]) from the VCSB or commercially available inter-laboratory cross-check program (ILCP) data may be used as is without imposing the reference installations requirements, provided that the number of non-outlying results is greater than 16 for both the designated and alternative test methods.

- ✓ Beginning January 1, 2016, include full test method documentation by the Voluntary Consensus Standard Based (VCSB) organization, including a description of the technology and/or instrumentation that makes the method functional.
- ✓ Include information reported in the test method that demonstrates the test method meets the applicable precision information for the method-defined fuel parameter
- ✓ Include information reported in the test method that demonstrates the test method has been evaluated using ASTM D6708 and whether the comparison is a “null” result or whether a correlation equation needs to be applied that predicts designated test method results from the applicable method-defined alternative test method