

## **LABORATORY ANALYSIS FACT SHEET TPH-DRO and TPH-ORO by Method 8270C**

The Missouri DNR has adopted SW-846 method 8270C as the method of choice for the detection and quantitation of TPH-DRO and TPH-ORO. The following items represent exceptions or clarifications to the method as written.

### **Sample Preparation**

1. Samples are extracted by the appropriate matrix defined SW-846 extraction methods specified by Method 8270C.
2. Surrogates specified by method 8270C are added to water and soil samples prior to extraction. Only the Base Neutral surrogates need to be added.
3. Internal standards specified by method 8270C are added to sample extracts immediately prior to extract analysis. Only the internal standards specified by 8270C for the PAH compounds are required for analysis and must meet the acceptance criteria.

### **Sample Analysis**

1. The GC/MS system is tuned to DFTPP tune criteria listed in method 8270C, at the frequency specified in method 8270C. Alternative tuning criteria as specified in section 7.3.1.2 of method 8270C is also acceptable, as long as method performance is not adversely affected.
2. The DFTPP tuning standard **is not required** to contain 4,4'-DDT, pentachlorophenol, and benzidine to monitor injection port inertness and GC column performance.
3. The GC/MS operating conditions specified as guidance in Section 7.3 of Method 8270C are acceptable with the exception of mass range. Mass range should be changed from 35-500 amu to 35-550 amu. This is required in order to quantitate and detect C35.
4. A 5 point standard curve is used to quantitate TPH-DRO and TPH-ORO. Quantitation is by external standard.
5. The stock standard solution used to prepare calibration standards is a mixture of unleaded gasoline and Number 2 diesel fuel. The stock standard and all calibration standards use Methylene Chloride as the solvent. Each component should be at a concentration of 1000 mg/L in the stock standard.
6. The lowest calibration standard should be at the reporting limit for the method. The highest calibration standard must not exceed the linear range of the system.

7. Retention time windows are defined for TPH-DRO and TPH-ORO by analyzing a standard containing C10, C21, and C35. The retention time window for TPH-DRO is defined as beginning 0.1 minutes after C10 to 0.1 minutes after C21. The window for TPH-ORO is defined as beginning 0.1 minutes after C21 to 0.1 minutes after C35. The standard containing C10, C21, and C35 must be analyzed every day samples are analyzed in order to verify that the retention time windows are constant.
8. Because the retention time window will be several minutes wide for both TPH-DRO and TPH-ORO, the GC/MS data system may not accurately or appropriately establish the proper baseline for calibration or quantitation. The analyst **must** visually examine the computer-generated baseline for every analytical run, and manually adjust the baseline when needed. A properly drawn baseline must extend over the entire retention time window and include the area under the entire TPH-DRO or TPH-ORO series of peaks. It is **not appropriate** to draw the baseline “peak to peak.”
9. Individual quantitation ions are not appropriate for TPH-DRO and TPH-ORO calibration and quantitation. The Total Ion Chromatogram (TIC) must be used to calculate the area under the peak for TPH-DRO and TPH-ORO calibration and quantitation determinations over the entire retention time window.
10. It is highly likely that the calibration standards used for this method will have little presence in the C21 to C35 window. The response factor calculated for the C10 to C21 window must be used for the C21 to C35 window.
11. Area counts for the internal standards and surrogates added during sample preparation must be subtracted from the total area count for TPH-DRO and TPH-ORO. This is accomplished by subtracting the area count of the method blank from all subsequent calibration and analytical runs.
12. The %RSD for the calibration curve for TPH-DRO and TPH-ORO must be less than or equal to 20 percent, so that linearity through the origin can be assumed and an average calibration factor used for calculations.
13. A continuing calibration verification standard (CCV) must be analyzed every 12 hours of sample analysis. The CCV must contain all analytes reported by this method. The standard concentration should be at the mid-point of the calibration curve. If the percent difference (%D) for all CCV compounds is less than or equal to 20%, then the initial calibration is assumed to be valid. If the %D is greater than 20%, corrective action must be taken prior to sample analysis.
14. A method blank must be analyzed for every extraction group, with each extraction group not to exceed 20 samples. Additionally minimum quality control samples per extraction group include a Laboratory Control Sample (LCS) and Matrix Spike/Matrix Spike Duplicate (MS/MSD). Ideally, the spiking solution(s) for the LCS and MS/MSD would contain TPH-DRO, TPH-ORO, and the PAH's quantitated by this method. It may not be possible however to have every component in the

spiking solution(s) for the LCS and MS/MSD samples. It is acceptable to spike the LCS and MS/MSD with different components, as long as TPH-DRO, TPH-ORO, and the PAH's are present in either the LCS or the MS/MSD samples.

15. If a sample has a sufficient TPH-DRO or TPH-ORO concentration to require PAH analysis, the same analytical run can be processed against a standard 8270C method containing the PAH's of interest.
16. All calibration and quantitation data for PAH analysis is by internal standard, using all criteria specified in 8270C. This includes the RSD requirement of 15% for the PAH compounds. If 15% RSD is not achieved, additional calibration options specified in Method 8000B may be employed.

Should questions arise concerning TPH analysis by Method 8270C, feel free to contact either of the following people:

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