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Questions regarding PAM-16130-SSI Determination of 2,3,7,8-Substituted Tetra- through Octa-Chlorinated Dibenzo-p-Dioxins and Dibenzofurans (CDDs/CDFs) Using Shimadzu Gas Chromatography Mass Spectrometry (GC-MS/MS)

What methods are approved by EPA for measurement of the 17 2,3,7,8-substituted PCDDs/PCDFs that are regulated under the Clean Water Act?

The only method currently approved at 40 CFR Part 136 for measurement of the 17 2,3,7,8-substituted PCDDs/PCDFs is EPA Method 1613B. EPA Method 625.1 may be used for screening purposes when a specific NPDES permit requires monitoring of only 2,3,7,8-Tetrachlorodibenzo-p-dioxin. EPA Method 613 may be used for analyte-specific determination of 2,3,7,8-Tetrachlorodibenzo-p-dioxin and for its quantitation provided that the laboratory can demonstrate that the method is sufficiently sensitive to allow for quantitation at the limit specified in a given permit in a given matrix.

Has EPA approved PAM-16130-SSI as an alternate test procedure (ATP) for determination of 2,3,7,8-substituted tetra- through octa-chlorinated PCDDs/PCDFs in wastewater?

Not at the present time. The method was submitted to EPA for review as an ATP for nationwide use under the Clean Water Act ATP program. The method and supporting information, including the validation study report and method performance data, were reviewed and EPA determined that the method met all requirements for measurement of the regulated PCDDs/PCDFs in wastewater. As a result, this method is a candidate for inclusion in a future regulatory action in which EPA periodically updates the methods approved at 40 CFR Part 136 for use in CWA compliance monitoring. An ATP review does not replace the normal notice-and-comment rulemaking process required for promulgation (approval) of the method at Part 136.

Can PAM-16130-SSI be used to support NPDES permits right now? How does one obtain limited use approval?

PAM-16130-SSI can be approved for NPDES permits on a case-by-case basis as a limited-use ATP, as is outlined in the regulatory text at 40 CFR 136.5, "Approval of alternate test procedures for limited-use." Limited use approval is granted by the Regional ATP Coordinator, typically for a specific discharge or facility (and its laboratory). For more information, see the full text at §136.5.

What prompted EPA to consider the ATP?

EPA routinely considers requests for ATPs to promote innovation in the CWA analytical methods program. Any person or organization may apply for an ATP, and EPA considers every ATP application that is received. In the case of PAM-16130-SSI, EPA heard from instrument vendors and laboratories analyzing PCDDs/PCDFs for some time that there was likely to be little further development or support of the high-resolution mass spectrometers (GC/HRMS) required to run Method 1613B. Several vendors have ceased development of high-resolution mass spectrometers, so it will become increasingly challenging for laboratories to obtain parts and supplies for specific instrument models from these vendors. EPA recognized the need to consider other detector systems, such as GC/MS/MS. Therefore, when EPA was approached by the ATP applicant several years ago, we were open to discussions about an ATP to Method 1613B

What is different about PAM-16130-SSI, compared to EPA Method 1613B?

The major difference between the two methods is the use of a GC/MS/MS instrument that uses Multiple Reaction Monitoring (MRM) in place of the GC/HRMS instrument. This change of detector system required the ATP applicant to come up with an alternative metric for mass resolution and ionization stability, in lieu of that in Method 1613B, as well as specifying the mass fragments and mass transitions and transition response ratios that must be monitored by the MS/MS system to accurately identify the target analytes.

All the sample preparation, extraction, and cleanup steps in Method 1613B are included in the ATP. That is, the matrix-specific extraction procedures in both methods are the same. In both methods, each sample is spiked with the same suite of carbon-labeled standards prior to extraction and those standards are used for isotope dilution quantitation, in the same way. All the relevant QC acceptance criteria are the same in both methods as well.

Will PAM-16130-SSI become an EPA-approved method?

PAM-16130-SSI needs to go through the same approval process as any other ATP that has been positively reviewed for nationwide use. EPA will have to propose the ATP in a future regulatory action that adds it to the list of approved methods at 40 CFR Part 136. These regulatory actions are called "Method Update Rules" (MURs). After a MUR is proposed, there is a public comment period, and an ATP will not be approved if there are any adverse public comments that cannot be addressed in EPA's formal Response to Comments document. If EPA has not received any unresolvable public comments regarding the ATP, then it will be included in the final MUR, and is approved and added to the relevant table at §136.3.

How should we refer to the method?

The name of the method is "PAM-16130-SSI" and it should be identified that way in any applications for limited-use approval and for any subsequent reports and correspondence.

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