

EPA Method ATP 16130 and GC-MS/MS Approaches for Chlorinated POP Analysis

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Background/Objectives. Standard methods for the analysis of chlorinated dioxins and furans, PCBs and other halogenated compounds for site characterization, remediation and risk assessment currently use high resolution magnetic sector mass spectrometry (GC-HRMS) to achieve high sensitivity, specificity, and reliable quantification. However, magnetic sector technology is being replaced by newer options such as tandem mass spectrometry (MS/MS) which offers the specificity and sensitivity advantages of HRMS with lower costs, more versatility in choice and operation and recent advancements in instrument technology. Compatibility with existing EPA regulatory methods, quality criteria, and the equivalence of this data with decades of existing GC-HRMS data is critical in the adoption of these new approaches as alternatives/replacements. The EPA recognized this need and instituted a process for development and validation of a GC-MS/MS alternative to EPA 1613B for dioxins and furans.

Our objectives with this study were to develop and validate a method equivalent to EPA Method 1613B to PCDD/PCDF analysis by MS/MS, produce an Alternate Test Method for review the EPA, and extend this approach to PCB congener analysis by EPA method 1668 and chlorinated pesticides analysis.

Approach/Activities. Using a Waters APGC-TQ-XS MS/MS system and an Agilent 7010 system (dioxins/furans only), we developed MS/MS acquisition protocols for the 17, 2,3,7,8-substituted dioxins and furans and total dioxin/furans by level of chlorination, the 209 PCB congeners, and 43 chlorinated pesticides. Sample workup and GC separation protocols were unchanged from the reference methods (for dioxins/PCBs) and all comparative tests were run using the same extracts. All method-specific interference and resolution checks were implemented. The method was validated using EPA standard protocols, and comparative data generated across multiple matrices and projects spanning multiple use cases. The methods were accredited across multiple quality protocols including ISO-17025 and NELAP.

Results/Lessons Learned. The methods passed all validation requirements. PCB and pesticide methods showed better sensitivity than equivalent methods. The dioxin and furan method has been reviewed by the EPA and has been notified as an alternate test procedure titled SGS AXYS 16130 pending posting in 40 CFR part 136. The methods have been implemented fully in the laboratory and comparative data on select projects will be presented, along with technical and regulatory lessons learned through the method transitions.