FluoroCouncil
Global Industry Council
for FluoroTechnology

May 2, 2011

Water Docket
Attn: Docket ID No. EPA-OW-2009-0090
U. S. Environmental Protection Agency
Mail Code 282211T
1200 Pennsylvania Ave., NW
Washington DC 20460
Submitted via regulations.gov

RE: FluoroCouncil comments on "Revisions to the Unregulated Contaminant Monitoring Regulation (UCMR 3) for Public Water Systems," Docket EPA-OW-2009-0090

To Whom It May Concern:

The Global Industry Council for FluoroTechnology (FluoroCouncil) respectfully submits to the U.S. Environmental Protection Agency (EPA) these comments concerning the Agency's proposed "Revisions to the Unregulated Contaminant Monitoring Regulation (UCMR 3) for Public Water Systems" (76 Fed. Reg. 11713, Mar. 3, 2011).

The FluoroCouncil is a global membership organization representing the world's leading manufacturers of fluoropolymers, fluorotelomers, and other fluorinated surfactants and surface property modification agents.[1] The FluoroCouncil has a fundamental commitment to product stewardship and, as part of its mission, addresses science and public policy issues related to fluoro-technology including perfluorinated chemicals.

The FluoroCouncil supports drinking water standards that protect public health and reflect the best available scientific evidence and the EPA's commitment to a sound science approach to the development of National Primary Drinking Water Standards (NPDWS). To that end, we support the collection of reliable and accurate occurrence data for unregulated drinking water contaminants. Because perfluorinated chemical occurrence data collected through the UCMR 3 program will be used to inform the Agency as it evaluates and prioritizes unregulated contaminants for potential future regulation, such data must be gathered using sound analytical methods and laboratory techniques. Only through the use of sound, validated methodologies can EPA's regulatory decision-making process be informed by reliable data that accurately reflect perfluorinated chemical occurrence in drinking water.

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[1] The FluoroCouncil's members are Asahi Glass Co., Ltd., Clariant Corporation, Daikin Industries, Ltd., E. I. du Pont de Nemours and Company, and Solvay S.A.

In response to EPA's request for comment on changes between UCMR 2 and UCMR 3 (76 FR 11716), the FluoroCouncil offers the following comments on the proposed UCMR 3 to help ensure the collection of accurate monitoring data for the six perfluorinated chemicals.

1. **To ensure UCMR 3 data are representative of actual sample concentrations, EPA should minimize perfluorinated chemical background contamination in UCMR 3 laboratories and adequately account for laboratory background contamination when establishing reporting levels for perfluorinated chemicals.**

In Table 1 – UCMR Contaminant List (76 FR 11735), EPA proposes a minimum reporting level (MRL) of 20 ng/L for perfluorooctanoic acid (PFOA) and MRLs of 10 to 90 ng/L for the remaining UCMR 3 perfluorinated chemicals. Section 6.12.1 of EPA's new Method 537 (Version 1.1) notes, "It is not possible to remove all PFAA [perfluorinated alkyl acid] background contamination" from the liquid chromatography with tandem mass spectrometry (LC/MS/MS) instrument.

The inclusion of such low MRLs in the UCMR 3 proposal, when combined with the reality of background levels of perfluorinated compounds in the laboratory, creates a major challenge for water systems trying to perform UCMR 3 monitoring for perfluorinated chemicals.

a. *UCMR 3 procedures for monitoring perfluorinated chemicals must adequately address the prevalence of background contamination of perfluorinated chemicals in analytical laboratories and minimize such background in UCMR 3 laboratories performing perfluorinated chemical analysis.*

FluoroCouncil member company experience indicates that background contamination of perfluorinated chemicals in standards and samples at contract analytical laboratories is common. Members report that one laboratory took three months to reduce their background levels and had to use two additional laboratories (one for cleaning and a "new" lab to receive the cleaned items) before they were ready to resume work. Another laboratory had to do a thorough lab cleanup, including a solvent scrubbing of their hood to reduce background. This laboratory then instituted a policy to keep samples and standards in a different part of the building on a separate ventilation system. A third laboratory inadvertently contaminated all surfaces by drying contaminated soil samples on the bench top. Another laboratory usually reports 5 to 10 ppt background in most sample sets.

A number of authors in the scientific literature have also noted the importance of background contamination during the analysis of perfluorinated chemicals. Yamashita *et al.* (2005) state "One of the major problems associated with trace-level analysis of perfluorinated acids, particularity PFOS [perfluorooctane sulfonate] and PFOA, is background contamination in analytical blanks." Martin *et al.* (2004) observe "Contamination sources of perfluoroalkyl substances in the laboratory are not well characterized but presumably are numerous given their current use in many retail products and common laboratory consumables." De Voogt and Saez (2006) point out the importance of proper sample handling to reduce contamination and analyte loss in their review of analytical techniques for perfluoroalkylated substances. Martin *et al.* (2004) mention ways to minimize contamination including the use of solvent-only injections "after high-concentration samples or standards to examine for carryover." Larsen and Kaiser (2007) suggest other techniques such as flushing and rinsing the chromatography system in case of analyte buildup ("i.e., if the minimum background rises with time").

In order to minimize potential background levels at UCMR 3 laboratories, the EPA should

- provide clear warning to potential laboratories for UCMR 3 perfluorinated chemical analysis about background contamination due to the unique nature of these compounds (e.g., sublimation, see comment 2.a),
- advise such laboratories on special measures to reduce the possibility of cross-contamination,
- address the need for sample storage and distribution (including the balance) to be distinct from the area where standards are made and stored in either Method 537 or the final UCMR 3 regulation,
- eliminate Method 537's requirement to evaporate extracts to dryness to help prevent cross-contamination (see comment 2.a),
- carefully evaluate the potential for and presence of background contamination at prospective laboratories during the laboratory approval process, and
- monitor contamination prevention performance as part of routine laboratory auditing during the conduct of UCMR 3 monitoring.

b. *EPA should rely on actual laboratory blank analytical results to factor laboratory background contamination into the determination of final UCMR MRLs for perfluorinated chemicals.*

EPA proposes to use a statistical methodology as the basis for Lowest Concentration Minimum Reporting Levels (LCMRLs) and MRLs in UCMR 3 (76 FR 11720).

Trace analysis for chemicals that are common laboratory contaminants warrants a different approach than the proposed LCMRL/MRL methodology to minimize false positives. The Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs (FACDQ) expressed concern about false positives, recommending "a ≤1% false positive rate be used for detection" (FACDQ, 2007).

Method 537 recognizes the potential for background contamination and invalidates results where the laboratory blank exceeds 1/3 of the MRL, effectively indicating that results within a factor of 3 of the MRL should be considered non-detect. However, longstanding EPA data validation guidelines provide for qualifying sample results within 5 times the value of the highest laboratory blank associated with an analytical batch as non-detect (EPA, 1999; EPA Region IV, 1999). Consistent with this approach, the 1/3 MRL threshold would be replaced by a 1/5 MRL threshold.

Risha *et al.* (2005) used 20% of the lower limit of quantitation (LLOQ) as the criterion for the absence/presence of significant interferences in the analysis of compounds including PFOA and perfluorononanoic acid (PFNA). These authors "recommended that a standard solution be run on the LC/MS/MS corresponding to the estimated LLOQ (25 ng/L) in matrix and a signal-to-noise ratio be obtained for the analyte transition of at least 5:1 compared to a reagent blank." In this way, Risha *et al.* (2005) assured a consistent way for assuring that results reported at or above the LLOQ (equivalent to MRL) were clearly distinguishable from background contamination present in the laboratory.

The FluoroCouncil agrees with the following statement from Section 9.3.1. of Method 537 (Version 1.1), "Because background contamination is a significant problem for several method analytes, it is highly recommended that the analyst maintain a historical record of LRB data." These Laboratory Reagent Blank data would provide the information needed to have a sufficient population of actual laboratory blank results (e.g., ≥10) to serve as the basis for calculating MRLs as 5 times the average blank data. A laboratory could begin assembling its historical record of LRB data during Proficiency Testing (as part of the Laboratory Approval Process) prior to the start of UCMR 3 monitoring. EPA could assess Proficiency Testing blank results against a set of target MRLs as part of its evaluation and approval of laboratories for UCMR 3 perfluorinated chemical monitoring.

Setting MRLs for perfluorinated chemicals in the final UCMR 3 regulation to 5 times the average blank level (rather than through the approach described in the LCMRL document accompanying the proposed UCMR 3 regulation) would make Method 537 reporting for UCMR 3 consistent with the ultimate reporting approach supported by EPA data validation guidelines (EPA, 1999; EPA Region IV, 1999; EPA, 2008).

In addition, the FluoroCouncil recommends that EPA set MRLs for UCMR 3 laboratories performing perfluorinated chemical analysis at 5 times the laboratory's average blank level to assure that UCMR 3 concentrations are representative of actual concentrations rather than background contamination. Alternatively, EPA could prescribe national MRL values in Table 1 of the final UCMR 3 regulation that take available data (including those generated during demonstration testing proposed in comment 2) on laboratory background contamination (e.g., 25 ng/L MRL for PFOA based on 5 ng/L background) into account and then require UCMR 3 laboratories performing perfluorinated chemical analysis to demonstrate background levels less than or equal to 1/5 of the MRL on an ongoing basis during UCMR 3 monitoring.

c. *UCMR 3 procedures should provide for routine analysis of laboratory and field blanks for adequate assessment and qualification of pefluorinated chemical blank contamination.*

Section 9.3.1 of Method 537 (Version 1.1) requires analysis of a Laboratory Reagent Blank with each analytical batch and states "Background from method analytes or other contaminants that interfere with the measurement of method analytes must be below 1/3 of the MRL". Section 9.3.8 of Method 537 (Version 1.1) notes "Analysis of the FRB [Field Reagent Blank] is required only if a Field Sample contains a method analyte or analytes at or above the MRL".

Longstanding EPA data validation guidelines provide for qualifying sample results within 5 times the value of the highest laboratory blank associated with an analytical batch as non-detect (EPA, 1999; EPA Region IV, 1999). More recent EPA guidelines (EPA, 2008) effectively continue this practice, especially for laboratory blanks or field blanks containing common laboratory contaminants relevant to the target analytes.

Method 537 or the final UCMR 3 regulation should allow for qualifying low level apparent detections within a factor of 5 of the highest laboratory blank in the corresponding analytical batch as non-detects, in addition to those situations with greater than or equal to 1/3 the MRL where Method 537 specifies re-analysis or re-sampling. Additionally, in order to maximize the utility of the laboratory blanks for assessing background contamination, it is important for EPA to assure that each UCMR 3 perfluorinated chemical analytical laboratory consistently concentrates the extract to the same fixed final volume (see comment 2) and to always use the same volumes for lab blanks as are used for study samples through periodic EPA audits of the laboratories performing UCMR 3 analysis for perfluorinated chemicals.

In order to ensure that background contamination in the laboratory is not included in the UCMR 3 data set as actual results, analytical laboratories must be required to take special measures to reduce the possibility of cross-contamination, MRLs must adequately factor in laboratory blank contamination levels, a sufficient number of blanks must be collected, analyzed, and assessed, and water systems must be allowed to qualify apparent detections within 5 times the highest blank in corresponding analytical batch as non-detects.

2. **EPA's new Method 537 should be revised and demonstrated as fit for purpose across the expected range of drinking water samples before widespread use in UCMR 3 monitoring.**

a. *EPA should modify Method 537 to exclude reduction of solid-phase extraction (SPE) cartridge extracts to dryness in order to prevent analyte loss and subsequent cross-contamination from that loss to other samples in the laboratory.*

The UCMR 3 proposed rule designates EPA's new Method 537 for analysis of perfluorinated chemicals in drinking water (Table 1, 76 FR 11735). In Section 11.5 of Method 537 (Version 1.1), the extract from the solid-phase extraction (SPE) cartridge is concentrated to dryness under a gentle stream of nitrogen in a heated water bath (60-65°C) to remove all the water/methanol mix.

Data from the peer-reviewed literature show that PFOA sublimes (i.e., goes from the solid to the gas phase) (Barton *et al.* 2008 and Barton *et al.* 2009). Another paper provides vapor pressure data for PFOA and PFNA (Kaiser *et al.*, 2005) which indicate PFNA's vapor pressure is slightly lower than vapor pressure of PFOA. Experimental results indicate an average rate of PFOA sublimation loss of 0.36 mg/h at a temperature of 45°C (Kaiser *et al.*, 2010). The vapor pressure of perfluoroheptanoic acid is expected to be slightly greater than that of PFOA. Because these perfluorocarboxylic acids are known to sublime, heating them to dryness at 60-65 °C would provide a significant opportunity for analyte loss. Such loss would lead to low bias in the determination and contribute to background contamination in the analytical laboratory, in turn potentially affecting other samples.

The FluoroCouncil recommends that EPA revise Section 11.5 of Method 537 to specify a final extract volume of 0.5 mL consistent with Section 8.1.4 of ISO 25101 (ISO, 2009) in accord with the Agency's use of consensus organization-developed methods elsewhere in UCMR 3. (The remaining solvent can be mixed with a small quantity of the initial chromatographic mixture to reduce overall solvent strength of the injected volume.)

b. *EPA should conduct additional near-term studies to ensure acceptable performance of Method 537 across the range of drinking water samples subject to UCMR 3 monitoring.*

Section 13.5 of Method 537 (Version 1.1) indicates that EPA and two external laboratories have demonstrated Method 537 performance. Method 537 is a new method with novel approaches to certain aspects of the analysis. For example, Section 6.9.1 of Method 537 (Version 1.1) specifies use of SPE cartridges containing styrenedivinylbenzene (SDVB) adsorbent.

Of over 500 peer-reviewed literature papers concerning analysis of perfluoroalkyl compounds, only two were found (Shoemaker *et al.*, 2009 and Hori *et al.*, 2004) that used SDVB cartridges for extraction. The Shoemaker paper was a contribution from the EPA describing the development of Method 537. The Hori paper used modified SDVBs. All other relevant papers made use of C_{18}, weak anion exchange (WAX), or hydrophilic lipophilic balanced (HLB) cartridges for SPE. For example, a method widely used in the U.S. for analysis of PFOA and PFNA in water relies on C_{18} for extraction (Risha, *et al.*, 2005). Analysis of split samples has shown that analysis based on WAX for SPE was able to detect perfluorohexanoic acid (PFHxA) not detected when HLB was the SPE cartridge (Taniyasu, *et al.*, 2005), thereby highlighting the importance of SPE cartridge selection during analysis of perfluorinated chemicals. While Section 13.5 of Method 537 (Version 1.1) indicates that two external laboratories have demonstrated Method 537 performance, details on experimental design and data resulting from those demonstrations have not been provided.

The paucity of SDVB experience in perfluorinated chemical analysis in water raises a number of questions about the adsorption-desorption characteristics of the cartridge in this service and the ability of the method to accommodate the potential effects of hardness, pH, and other differences among the variety of drinking water sources in the U.S. The use of a different SPE cartridge than the types generally used in the literature, and the relatively small sample set for results reported in Section 17 of the Method, indicate the possibility of untested problem areas in Method 537. As proposed, the first comprehensive evaluation of this method over a wide range of drinking water samples would come during UCMR 3 monitoring. Requiring thousands of water systems to use a new analytical method with a somewhat untried SPE cartridge at its core is not a wise use of resources.

Prior to widespread use in UCMR 3 monitoring at thousands of water utilities, Method 537 (revised to exclude reduction of SPE extract to dryness) should undergo further testing at two or three external laboratories to demonstrate performance of the SPE cartridge and the entire method with about 20 samples representative of the expected range of drinking water samples.

3. The final UCMR 3 requirements should maintain the existing 60-day time period for water systems to review, reanalyze (if needed), approve, and submit UCMR data.

In draft 40 CFR § 141.35(c)(6)(ii) (76 FR 11733) and in Exhibit 2 (76 FR 11717), EPA has proposed to limit water systems to 30 days to "review, approve, and submit" UCMR 3 analytical data for 30 constituents across a total of 7 (6 chemical and 1 biological) test methods. EPA requested comment on this shortened reporting timeframe (76 FR 11723).

In UCMR 1 and UCMR 2, EPA allowed water systems 60 days for review, approval, and submission of data resulting from traditional water analytical methods such as GC/MS. Although EPA included LC/MS/MS monitoring (Method 535) for Screening Survey List 2 contaminants in UCMR 2, the UCMR 3 proposed rule includes List 1 Assessment Monitoring for analytes using LC/MS/MS for the first time in the Unregulated Contaminant Monitoring Program. In fact, the EPA proposal includes LC/MS/MS Assessment Monitoring for two groups of UCMR 3 analytes. Without adequate time to review the results from these new LC/MS/MS analytical methods, water systems are less likely to be able to assess the impact of contamination on reported values.

In light of general constraints on laboratory turnaround time and the potential for LC/MS/MS instrument outages, a 30-day review period would effectively preclude water systems from being able to have samples reanalyzed where such reanalysis is triggered by data validation review. Cutting the available review time in half, while requiring water systems to review data from a new and untested analytical technology, is not consistent with providing data upon which EPA can reliably predict national occurrence of chemical contaminants in drinking water.

EPA should maintain the existing 60-day period for water systems to review, reanalyze (if needed), approve, and submit UCMR 3 data.

4. EPA's proposed new LCMRL approach is flawed and, to ensure it results in the development of accurate and reliable information, needs independent technical review before re-proposal for potential use in a regulatory setting.

EPA has proposed "revisions to the definition of the minimum reporting level (MRL)" (76 FR 11719) that reflect "improvements in the statistical procedures for determining the LCMRL and MRL" (76 FR 11719). The Agency requested comment "regarding the proposed definition of MRL" (76 FR 11720) after briefly describing the statistical procedure for estimating the LCMRL and, in turn, the computation of MRL. The introduction to the LCMRL Calculator tool document announced in the proposal ("Technical Basis for the Lowest Concentration Minimum Reporting Level (LCMRL) Calculator," EPA, 2010) notes, "The MRL may be useful as an alternative to the PQL [practical quantitation limit] for setting future regulatory limits" (p. 2 of 24). The closing section of this LCMRL Calculator tool document states, "Although the procedure is complex and computationally intensive, implementing it in user-friendly software in the public domain and freely available over the internet makes it easy for laboratories to use" (p. 22 of 24).

In fact, the LCMRL Calculator Tool document begins to describe an extremely complex procedure for establishing LCMRLs and MRLs that is not warranted in UCMR 3 and that is not an appropriate alternative to the PQL for other regulatory purposes. Furthermore, the document is incomplete in several ways:

1) The document lacks a practical start to finish demonstration of how these calculations should be done.
2) Contrary to basic science and engineering principles, the document does not present comparative examples or otherwise demonstrate a reasonable return for investing the extra effort required for its computational approach relative to the LCMRL methodology used for UCMR 2.
3) EPA's claim in the document that Bayesian bootstrapping is applicable to very small datasets, while "ordinary" bootstrapping is not, is not supported by, and seems contrary to, the Bayesian literature (e.g., Parzen *et al.*, 2005, and Gu, *et al.*, 2008). If Bayesian bootstrapping is in fact applicable to very small datasets, that should be shown by appropriate citations and subjected to further review.
4) The document's general claims of "improvements" via adoption of the highly complex approach therein for computing the prediction variance in comparison to the

methods of Rocke and Lorenzato (1995), EPA (2004), or Helsel (2005) have not been shown to be grounded in real data.

5) No specific reference is included in the document for the Matlab and R code used in the LCMRL calculations, thereby making it difficult to comment on the accuracy of the code or any limitations the code may have.

The document requires extensive revision to fill in the above enumerated gaps to provide adequate documentation and explanation of the separate "black box" LCMRL calculator tool proposed for use by UCMR 3 laboratories. Furthermore, the statistical approaches used in the proposed LCMRL Calculator tool do not appear to address the potential for false positives due to background contamination of chemicals typically present in analytical laboratories, as is the case for perfluorinated chemicals in UCMR 3.

Not only is the approach in the LCMRL Calculator tool document technically flawed, but it has not been subjected to the necessary peer review and public comment pursuant to information quality, transparency, and administrative procedural requirements. EPA should refrain from further use of the proposed revised LCMRL approach in UCMR 3 until it has undergone proper review and comment. To do otherwise would disregard EPA's own information quality and peer review guidance, along with the Administration's emphasis on transparency and openness in the regulatory process.[2]

Following incorporation of the suggested revisions described above, EPA should seek technical, independent peer review of the LCMRL document. With similar documents and methodologies, EPA has convened Science Advisory Board (SAB) panels to provide the Agency with its independent assessment and recommendations. We urge EPA to consider convening a SAB panel of laboratory data validation experts, analytical chemists, and statisticians as a possible approach to obtaining the needed peer review of the revised LCMRL document. As FACDQ stated in its 2007 final report, "Looking ahead to further work by EPA on procedure/s for detection and quantitation, the Committee recommended that a formal peer review of the procedure proposed for promulgation be undertaken and that a follow up pilot study be completed to confirm the performance of whatever procedure(s) EPA proposes to promulgate."

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[2] *See* Pub. L. No. 106-554; U.S. EPA, *Guidelines for Ensuring and Maximizing the Quality, Objectivity, Utility, and Integrity of Information Disseminated by the Environmental Protection Agency* (Oct. 2002) (establishing the appropriate administrative mechanisms and review processes for ensuring the quality of influential information disseminated by EPA. As the underlying foundation for the MRLs proposed in the UCMR 3, the FluoroCouncil considers the LCMRL approach to be unarguably influential.); Peer Review Handbook, 3rd Edition, U.S. EPA, Science Policy Council, January 2006, EPA 100-B-06-002, at http://www.epa.gov/peerreview/pdfs/peer_review_handbook_2006.pdf (requiring peer review for scientific and technical work products that support a regulatory program and establish "a significant precedent, model, or methodology," which, the FluoroCouncil contends, includes the LCMRL approach used in the UCMR 3). *See also* Exec. Order No. 13,563, 76 Fed. Reg. 3821 (Jan. 21, 2011) (emphasizing the need for public participation and transparency in the rulemaking process and the importance of ensuring "objectivity of any scientific and technological information and process used to support the agency's regulatory actions").

Following independent peer review, if EPA still wishes to utilize the revised LCMRL approach in a regulatory context, the Agency should first issue the revised document (and accompanying computer code) for public notice and comment. In the interim, an alternate approach to establishing MRLs for perfluorinated chemicals is proposed earlier in these comments (see comment 1). For the other UCMR 3 constituents, use of the LCMRL approach currently used under UCMR 2 may be appropriate.

5. Conclusion

Overall, the FluoroCouncil is concerned that, as proposed, Method 537 and other requirements for UCMR 3 monitoring of perfluorinated chemical occurrence in drinking water will lead to the generation of inaccurate data, inappropriate as the basis for future regulation. Additionally, the proposed new LCMRL approach should not be implemented in support of UCMR 3 or any other rulemaking without appropriate peer review and additional public comment.

We appreciate the opportunity to provide information and comment on the proposed UCMR 3. In addition, we would welcome the opportunity for our technical experts to meet with EPA to discuss our suggestions to minimize laboratory background contamination and to improve Method 537 prior to its use in UCMR 3. If you have any questions or would like to arrange a meeting, please contact Jessica Steinhilber at 202-249-6737 or jessica_steinhilber@americanchemistry.com.

Sincerely,

Jessica S.

Jessica S. Steinhilber
FluoroCouncil

Appendix: References

APPENDIX

References

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FluoroCouncil Perspective on EPA's Proposed UCMR 3 for Public Water Systems (March 5, 2012)

- **Context**
    - The point of UCMR 3 is to collect scientifically valid data on contaminant occurrence as a key step in the process of evaluating the need for new drinking water standards.
    - UCMR 3 results could affect capital decisions at >50,000 community water systems.
    - Perfluoroalkyl substances are present in many lab instruments and are ubiquitous in lab consumables, resulting in significant lab background levels impacting results without proper care.
        - Elevated backgrounds contributed to poor reproducibility in water in 1st interlaboratory study (ILS).
        - Improvements seen in 2nd ILS after ≥12 months effort show benefit of reducing background levels.
    - EPA developed an LC/MS/MS method (Method 537) for analysis of the 6 designated perfluorinated chemicals in drinking water.
    - A consensus ISO method, developed through a collaboration of many ILS participants, exists for 2 of these 6 perfluorinated chemicals (PFOA and PFOS) in water.
    - UCMR 3 represents the <u>first time</u> that LC/MS/MS monitoring is be used for List 1 Assessment Monitoring in the Unregulated Contaminant Monitoring Program.
    - UCMR 3 is the <u>first time</u> that Method 537 would be used on a variety of drinking water samples.
    - Method 537 is **not** an approved method and will not be an approved method until the UCMR 3 final rule is issued. Now is the time to address deficiencies in Method 537.

- **Goal 1: Ensure results will be meaningful and representative before doing the analysis.**
    - EPA should rely on actual laboratory blank results (reflective of laboratory background contaminant levels) to calculate Minimum Reporting Limits (MRLs) rather than using statistical calculator tools (which are not designed to address laboratory background contamination).
    - Consistent with long-standing EPA data validation guidelines, Method 537 MRLs should be set at 5 times the average blank level for the analysis batch.
    - EPA should upgrade lab review/approval/audit regulatory procedures and give guidance to UCMR 3 labs to minimize background levels at UCMR 3 laboratories.

- **Goal 2: Assure the analysis will be done correctly.**
    - Method 537's requirement to reduce solid-phase extraction (SPE) cartridge extracts to dryness needs to be replaced with drying to a fixed final extract volume as in ISO 25101: 2009 for PFOA and PFOS <u>in order to preserve the relationship between a sample and its reported concentration</u>.
    - Internal standards (^{13}C-labeled compounds) are available for 2 more Method 537 analytes than specified; the method for UCRM 3 should use all available ^{13}C-labeled internal standards.
    - Without these corrections and additional guidance in the method or in the final rule to minimize likely background levels at UCMR 3 laboratories, the ensuing sample cross-contamination would result in over-reporting results for many waters and possibly under-reporting for others.

- **Goal 3: Demonstrate that the method for UCMR 3 will provide quality results before wide use.**
    - Method 537 has not been demonstrated on a representative range of drinking water samples.
    - The small number of drinking water samples (apparently 2) that EPA and its two contract labs performed during development of Method 537 are insufficient to assure that the novel SPE cartridge and the rest of Method 537 will work across the range of drinking water samples (>10,000) in UCMR 3 monitoring.
    - The ISO consensus method provides a tested alternative for PFOA and PFOS, and laboratories have extended this method to add the other perfluorinated chemicals in UCMR 3.
    - Regardless of which method is selected, a pilot demonstration of the method is needed to assure high quality data for each designated perfluorinated chemical prior to widespread use across 5,000 public water systems. (AWWA 5/2/2011 comments endorse a "multi-laboratory method trial" prior to UCMR 3 sampling.)