Message

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Sent: 4/26/2018 2:13:01 PM
To: Nadine Kotlarz [nkotlar@ncsu.edu]; Jane Hoppin [/o=ExchangeLabs/ou=Exchange Administrative Group (FYDIBOHF23SPDLT)/cn=Recipients/cn=userebcfc262]; Detlef R. U. Knappe [/o=ExchangeLabs/ou=Exchange Administrative Group (FYDIBOHF23SPDLT)/cn=Recipients/cn=user17c3f77b]; Lindstrom, Andrew [/o=ExchangeLabs/ou=Exchange Administrative Group (FYDIBOHF23SPDLT)/cn=Recipients/cn=04bf7cf26aa44ce29763fbc1c1b2338e-Lindstrom, Andrew]
CC: McCord, James [/o=ExchangeLabs/ou=Exchange Administrative Group (FYDIBOHF23SPDLT)/cn=Recipients/cn=McCord, James]
Subject: RE: Update on serum method

Good job Nadine. I hope we can figure out the PFHxS and PFOS issue. The values still appear to agree with the SRM 1957 pretty well. Something with the Nafion BP1 calibration curve at the bottom seems weird.

However good to see we can measure the Nafion BP2 in the serum at appropriate levels in our curve.

Mark

From: Nadine Kotlarz [mailto:nkotlar@ncsu.edu]
Sent: Wednesday, April 25, 2018 9:31 PM
To: Jane Hoppin <jahoppin@ncsu.edu>; Detlef R. U. Knappe <knappe@ncsu.edu>; Lindstrom, Andrew <Lindstrom.Andrew@epa.gov>
Cc: McCord, James <mccord.james@epa.gov>; Strynar, Mark <Strynar.Mark@epa.gov>
Subject: Update on serum method

Hi everyone,

James, Mark and I have been working on the serum method using EPA's Orbitrap.

Our method is 50 uL serum with formic acid denaturation and acetonitrile protein crash. After centrifugation to separate proteins, we load 100 uL of the acetonitrile fraction into an LC vial for analysis. Injection volume is 25 uL.

We prepared standards in calf serum for PFMOAA, GenX, Nafion byproduct 1, Nafion byproduct 2, and the legacy PFAS at concentrations of 0.1, 0.25, 0.5, 1, 2.5, 5, 10, 15, 20, 25 ng/mL for each compound. We ran 10 randomly selected serum samples from the GenX Exposure Study and we ran SRM1957 to see if our measurement of PFAS in this material is close to what others have reported. We did not do internal standard correction for this run but would include internal standards in the future.

Results are summarized in the summary tab of the excel spreadsheet. Calibration curves (with some points omitted for the different compounds) are in the ppt.

Initial observations:
1. GenX standard curve looks okay down to 0.5 ng/mL.
2. Poor response for Nafion byproduct 1 at the lower end of the cal curve (< 10 ng/mL)
3. For GenX and Nafion byproduct 1, the serum sample response is not significantly different from the blanks (calf serum).

4. We do not see PFMOAA in the samples or the standards (James, can you please confirm that we do not see it even in the 25 ng/mL?)
5. For Nafion byproduct 2, the mean concentration across the 10 serum samples is 3.4 ng/mL, and sample response is significantly higher than the blanks. The concentrations in the serum samples ranged from approx. 1-7 ng/mL.
6. Based on area counts, mean response for PFO4DA across the samples is 2 orders of magnitude above the blanks. We did not calibrate for this compound but Mark did get the standard from Chemours so we could reprepare standards with it.
7. Based on area counts, mean response for PFO5DoDA across the 10 samples is one order of magnitude above the blanks. We do not have a standard for this compound.
8. We see significant background for legacy PFAS in the blanks. For now, we do not know how to get rid of it. This background flattens the standard curves for PFHxS and PFOS and makes it difficult to quantify the legacy compounds in the serum samples.
9. In an interlab study of SRM1957, the reference values were 5 +/0 0.40 ng/mL PFOA and 21.1 +/- 1.2 ng/mL PFOS. We measured 4.2 ng/mL and 23.7 ng/mL PFOA and PFOS, respectively.

Please let us know your thoughts. Thanks,
Nadine