



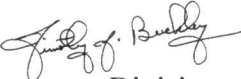
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
NATIONAL EXPOSURE RESEARCH LABORATORY
RESEARCH TRIANGLE PARK, NC 27711


OFFICE OF
RESEARCH AND DEVELOPMENT

October 25, 2017

MEMORANDUM

SUBJECT: Laboratory PFAS Report No. 6 for NC DEQ: Chemours Process Samples

FROM: Timothy J. Buckley, Director 
Exposure Methods and Measurements Division

THRU: Tim Watkins, Acting Director 
National Exposure Research Laboratory

TO: Linda Culpepper, Deputy Director
Division of Water Resources
North Carolina Department of Environmental Quality

Attached please find our laboratory report for samples collected September 18, 2017 from the process lines of the Chemours-Fayetteville, NC facility. This is our 6th in a series of reports intended to support NC DEQ decisions related to industrial discharge of PFAS to the Cape Fear River and surrounding community.

Thank you for providing us with the opportunity to contribute to this effort for the protection of North Carolina public health and the environment. It is worthy of note that our work together is well aligned with EPA's new Strategic Plan Goal 2 – Cooperative Federalism: supporting the primary implementers of environmental programs—states and tribes “to create tangible environmental results for the American people.”

These results represent the effort and expertise of Drs. Mark Strynar, Andy Lindstrom, James McCord, and Seth Newton in conducting the laboratory analyses. Dr. Myriam Medina-Vera provided invaluable management support and coordination, and Ms. Sania Tong Argao is acknowledged for her support in quality assurance review.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2454 or email buckley.timothy@epa.gov. I look forward to our continued work together.

Enclosure

CC: Becky B. Allenbach, USEPA Region 4
Jeff Morris, USEPA OPPT
Betsy Behl, USEPA, OW
Peter Grevatt, USEPA, OW

Summary of Methods and Results

The results reported here are from sampling that was conducted by Chemours' staff on September 18, 2017 and witnessed by NC DEQ and Dr. Andy Lindstrom from our lab. For this report, we limited our analysis to GenX and the five non-targeted analytes (Table 1) that previous reports had shown to be of concern. As we have indicated in prior reports, an important limitation to our non-targeted analysis is that these results are considered semi-quantitative. We cannot know the exact concentration because no authentic standards are available for these chemicals. However, we are very confident of the chemical identity based on the high resolution mass spectrometry and knowledge of Chemours' chemical products.

Table 1. PFAS Analytes Measured by Non-Targeted LC/TOFMS Analysis

Short Name	Chemical Name	Formula	CAS no.	Monoisotopic Mass (Da)
GenX	Perfluoro(2-methyl-3-oxahexanoic) acid	C ₆ HF ₁₁ O ₃	13252-13-6	329.9750
PFESA (Nafion) Byproduct 1	Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid	C ₇ HF ₁₃ SO ₅	29311-67-9	443.9337
PFESA (Nafion) Byproduct 2	Ethanesulfonic acid, 2-[1-[difluoro(1,2,2,2-tetrafluoroethoxy)methyl]-1,2,2,2-tetrafluoroethoxy]-1,1,2,2-tetrafluoro-	C ₇ H ₂ F ₁₄ O ₅ S	749836-20-2	463.9399
PFMOAA	(2,2-difluoro-2-(trifluoromethoxy)acetic acid)	C ₃ HF ₅ O ₃	674-13-5	179.9846
PFO2HxA	perfluoro-3,5-dioxaheptanoic acid	C ₄ HF ₇ O ₄	39492-88-1	245.9763
PFO3OA	perfluoro-3,5,7-trioxaoctanoic acid	C ₅ HF ₉ O ₅	39492-89-2	311.9680

We determined the concentration of GenX against a standard calibration curve derived from an authentic standard using a traditional targeted analysis approach. In contrast, our quantification of the non-targeted analytes is semi-quantitative. Since standards are not available for these analytes, we estimate their concentrations based on the GenX instrument response as shown in the equation below.

$$[NTA] = [GenX] * \frac{NTA_{PA}}{GenX_{PA}}$$

Where: [NTA] is the concentration of the non-targeted analyte (ng/L)
 [GenX] is the concentration of GenX (ng/L)
 NTA_{PA} is the integrated peak area for the non-targeted analyte
 GenX_{PA} is the integrated peak area for GenX

This method of estimation assumes that the mass spectrometer responds to the non-targeted analyte as if it were GenX. The actual instrument response may be weaker or stronger resulting in an under- or over-estimation of the non-targeted concentration. Our experience with this class of analytes suggests that estimates of this fashion are accurate to within ~10-fold of the estimated value. Additional uncertainty in estimated concentration of non-targeted analytes is introduced for samples where GenX exceeded the calibration curve (because the determination of GenX concentration is more uncertain) and for samples where there is a large discrepancy between the GenX concentration and the non-target analyte (due to scaling). For both GenX and the non-targeted analytes, laboratory analysis was performed on a quadrupole time of flight mass spectrometer. Additional details on the method of analysis can be found in Sun *et al.*, 2016¹ and Strynar *et al.*, 2015².

Results are presented in Table 2. Concentrations varied by sampling location and analyte and ranged by 5 orders of magnitude from non-detect to 141,000 nanograms per liter (ng/L). Concentrations of GenX exceeded the highest value on our calibration curve (i.e., 1000 ng/L) for five samples. These values have been flagged accordingly.

Quality control samples included a trip blank, a lab blank, and solvent blanks. In all cases, none of the analytes were detected. Two trip spikes at a low and high concentration (250 ng/L or 1000 ng/L) were within 17% of the target value. Replicate samples (Huske L&D - %RSD 26.1) (Chemours old waste tributary - %RSD 2.7) and (Chemours outfall 001 - %RSD 1.6) all showed good precision.

¹ Sun M; Arevalo E; Strynar M; Lindstrom A; Richardson M; Kearns B; Pickett A; Smith C; Knappe DRU: Legacy and Emerging Perfluoroalkyl Substances Are Important Drinking Water Contaminants in the Cape Fear River Watershed of North Carolina. Environmental Science & Technology Letters. 2016

²Strynar M, Dagnino S, McMahan R, Liang S, Lindstrom A, Andersen E, McMillan L, Thurman M, Ferrer I, Ball C. Identification of Novel Perfluoroalkyl Ether Carboxylic Acids (PFECAs) and Sulfonic Acids (PFESAs) in Natural Waters Using Accurate Mass Time-of-Flight Mass Spectrometry (TOFMS). Environ Sci Technol. 2015

Table 2. PFAS Concentrations (ng/L)

Location	Quantified Using Targeted Analysis	Quantified Using Non-Targeted Analysis				
	GenX	PFMOAA	PFO2HxA	PFO3OA	PFESA BP1	PFESA BP2
Huske L&D rep 1	53.1	185	117	23.2	641	1,910
Huske L&D rep 2	77.1	183	120	15.3	712	2,240
Chemours old waste tributary rep1	9,600*	24,000	10,500	4,030	2,540	4,010
Chemours old waste tributary rep2	9,980*	24,600	11,500	4,240	2,240	3,690
Chemours 001 rep1	306	13,700	23,200	2,910	13,900	128,000
Chemours 001 rep2	299	14,400	23,500	2,900	15,500	141,000
Chemours 002	49	1,300	2,670	221	6,560	45,200
Waste Acid Tank	336	268	637	536	4,500	735
Deg. Tank	8,860*	-	30.4	-	-	34,800
Common Waste Tank	1,270*	-	479	64.0	3,700	26,500
Product Sump	759	50.0	244	5.55	30,300	7,400
Tank Farm Sump	265	851	363	517	14,200	20,500
Hydrolysis	1,290*	-	17.6	-	-	5,010

Flag Codes:

* = analyte above upper range of calibration curve (1000 ng/L); consider as estimate.
dash "-" = analyte not detected