

**RESPONSES TO COMMENTS  
TABLE 1 - PARAGRAPH 11(b) CHARACTERIZATION PLAN**

Document	Comments From	Comment ID	Comment	Proposed Responses
PFAS Characterization Sampling Plan	EPA Region 4	T1-1	Page 4, 3.3.2 Sample Collection Procedures Common to All Locations: There are no "procedures" listed in this section nor in Table 4, Sampling Containers, Preservation, and Holding Times.	Samples will be collected according to guidelines set out in the PFAS Quality Assurance Project Plan (QAPP) (AECOM, 2018). This document is referenced in Section 3.3.2. The title of Section 3.3.2 is updated to "Sample Collection Considerations".
PFAS Characterization Sampling Plan	EPA Region 4	T1-2	Page 5, first paragraph, first sentence: If the sample containers used for EPA Method 537 Mod contain Trizma® as a buffer, they should not be used to fill the flow-through cell.	Noted. Trizma® is recommended for drinking water samples. This buffer will not be used for these non-drinking water samples.
PFAS Characterization Sampling Plan	EPA Region 4	T1-3	Page 5, first paragraph, third sentence: "For each sample type" should be "For each sample" or "For each sample station".	The text is updated to "for each sample".
PFAS Characterization Sampling Plan	EPA Region 4	T1-4	Page 5, second paragraph: For consistency between sampling events, more detail of how the sampling will be conducted for each of the various media (i.e. stormwater, process water, NCCW, etc.). When temporal composite samples are being collected, the duration and interval of the aliquot collection should be specified. During storm events, the "composite samplers" may need to be reprogrammed for each storm, but it is necessary to detail the criteria for the field personnel and the regulators what information is desired for each sampling event. Additionally, the protocol used to split the composite sample and fill the nine sample containers needs to be stated.	Temporal composite samples will be collected once every two months when there is a 70% or greater chance of a storm event forecasted to last at least 4 hours and have at least 0.5" of rainfall, following an antecedent dry period lasting at least 72 hours. National Oceanic and Atmospheric Administration (NOAA) weather data will be used to track regional weather and identify storm events that meet these criteria. Local and site meteorological data suggest that there will be multiple storm events of this nature in each two month period, however if there is a two month period with no storm events meeting these criteria, dry stormwater locations will not be sampled. Autosamplers will be switched on 1-2 hours ahead of the predicted storm event, and they will start collecting aliquots once their dedicated rain gages or similar device collect 0.1" of water. Water will be composited over either 4 or 8 hours, depending on the predicted length of the storm event, and 10-12 aliquots will be composited in the autosampler over the course of the storm event. These criteria may be updated at a later date based on experiences and data from implementing these characterization events. Following sample collection, the composited samples will be poured into 250 mL HDPE bottles. All grab samples except for 21A and 21B will be collected by directly filling the HDPE bottles with sample. 21A and 21B (sediment basin effluent samples) will be collected from shore using a pole to reach to within 8 feet of the riser pipe, from the depth of the sediment basin equivalent to the height of the riser pipe. Samples will be collected into HDPE bottles that can be secured to the pole, then samples will be transferred into 250 mL HDPE bottles.
PFAS Characterization Sampling Plan	EPA Region 4	T1-5	Page 5, second paragraph: While it may be advantageous to collect composite samples for variable solids loading in stormwater samples, it should be acknowledged that there may be PFAS losses in the composite sampling process. From Section 3.1 Equipment and Supplies of the Interstate Technology Regulatory Council's Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances [PFAS]: "Not all PFAS are hydrophilic, and some are volatile. As a result, these chemicals may sorb to sampling equipment and supplies or be lost from samples during sample collection. Preliminary data suggest that sorption may occur quickly. Additionally, volatile losses have not yet been characterized. Until they are better quantified, sampling efforts should consider whether these losses would affect project objectives and adjust accordingly." To evaluate the PFAS losses, it is recommended that a grab sample (or two) be collected in addition to one of the proposed composite sample locations for each sampling event.	To address this concern, a grab sample will be collected in addition to the temporal composite sample at one stormwater location during each sampling event. Detection and abundance of PFAS will be compared in the grab sample and the temporal composite sample to assess whether there were losses in the temporal composite sample. Location 15 will be used for this evaluation for the first sampling event. Teledyne 6712C autosamplers are being used for temporal composite sample collection, which have HDPE sample reservoirs and use HDPE tubing and silicon tubing. Use of HDPE and silicon is consistent with ITRC guidance on material selection when sampling PFAS.
PFAS Characterization Sampling Plan	EPA Region 4	T1-6	Pages 5 & 6, Stormwater, Intake and Outfall, NCCW, Process Wastewater, and WWTP Discharge Sampling: Due to the potential stratification of PFAS in solution, the sampling location/depth in the water column needs to be specified for each sampling medium. (See Section 3.6.2 Surface Water of the ITRC's Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances [PFAS]).	Most sample locations with water streams that reach the Cape Fear River are flowing, well mixed, and often shallow, so stratification is unlikely to occur. The exceptions may be at Locations 21A and 21B, the sediment basins, which can contain deeper standing water. At these locations, water will be sampled from the surface of the sediment basins within 8 feet of the riser pipe to capture water near the point where overflow into the riser pipe occurs. The other locations with a column of standing water are process wastewaters from Chemours (Locations 16, 17A, and 17B) which are stored in containers on site prior to being sent for off-site disposal by tanker truck. These locations have an accessible sampling port that will be used during this program.
PFAS Characterization Sampling Plan	EPA Region 4	T1-7	Page 6, Non-Contact Cooling Water: For the spatial composite samples, the length of each ditch and the number aliquots collected needs to be stated so that data from subsequent sampling events can be appropriately assessed.	Spatial composite samples are no longer included in the workplan. Direct NCCW samples will be collected as individual grab samples from infrastructure (sumps and above ground pipes) during the first sampling event. The grab samples may be combined during subsequent events if the individual samples are similar to each other in compounds detected and concentrations of compounds detected during the first sampling event.
PFAS Characterization Sampling Plan	EPA Region 4	T1-8	Page 6, Process Wastewater: Process wastewaters have the potential for great variability due to batch processing, process upsets, wash-downs, start-ups, etc., but grab samples are being proposed for each production area. Grab samples are appropriate where temporal variability over the course of one day is not expected at the sampling location	Grab samples are appropriate for the Chemours process wastewater samples (Locations 16, 17A, and 17B) since these samples are collected from tanks that composite wastewater over several days before being taken off site by tanker truck. Grab samples are also appropriate for the DuPont process wastewater samples (Locations 19A and 19B) since these samples accumulate in a tank at the DuPont facility over several days before being released to the sump at the wastewater treatment plant. For the first sampling event, temporal variability of the Kuraray process wastewater locations (Locations 18 and 23) will be assessed by collecting and analyzing 4 separate grab samples at 4 hour intervals over the course of 12 hours. If there is temporal variability in the samples, autosamplers will be acquired to collect temporal composite samples during subsequent sampling events. If there is no temporal variability, single grab samples will be collected at these locations during subsequent sampling events.

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PFAS Characterization Sampling Plan	EPA Region 4	T1-9	Page 7, 3.5 Quality Assurance/Quality Control, second sentence: "Table 4" should be "Table 5".	This table reference has been corrected.
PFAS Characterization Sampling Plan	EPA Region 4	T1-10	Page 8, 3.5.1 Field QA/QC, second sentence: Insert at the beginning of the section "Criteria for achieving...". Also, "Table 3" should be "Table 2".	This table reference has been corrected and the title for Table 2 has been updated.
PFAS Characterization Sampling Plan	EPA Region 4	T1-11	Page 8, Equipment Blanks, first sentence: Equipment blanks should also be used to demonstrate that sampling equipment is not contaminating the sample with analytes of interests. Modify the first sentence to "Equipment blanks are used to evaluate equipment and cleaning or decontamination procedures."  The complete sampling system (containers, gloves, tubing, composite samplers, labels, bags, etc.) should be evaluated to ensure all the equipment is PFAS-free and not contributing to any PFAS detected in the samples. Initially this can be done by using the composite sampler to collect a sample from PFAS-free water and labeling, bagging, ice, and shipping to the laboratory for analyses. If PFAS are detected above the PQLs, then further investigation will be required to determine the cause.	The text describing equipment blanks has been updated. Equipment blanks collected using existing equipment have been routinely non-detect for PFAS. Contamination due to any new equipment will also be evaluated through routine equipment blank collection.
PFAS Characterization Sampling Plan	EPA Region 4	T1-12	Table 2: Title should be "CRITERIA FOR ACHIEVING DATA QUALITY OBJECTIVES".	The title for Table 2 has been updated.
PFAS Characterization Sampling Plan	EPA ORD	T1-13	In section 2 of the data quality objectives PFAS are split into 2 groups: 1) Emanating from Chemours; and 2) Not emanating from Chemours. A third group is needed: 3) contributed to by Chemours. PFAS are in the Cape Fear River, however there are legacy PFAS present contributed to by Chemours.	The goal of this workplan is not to comment on sources of PFAS, therefore the language in this section has been adapted to remove these distinctions.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-14	The influent to the Wastewater Treatment Plant should be included as one of the sampling locations.	The wastewater treatment plant combined influent is now included as Location 22.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-15	The effluent from whichever sedimentation basin is discharging (only one basin is in use at a time) should be included as one of the sampling locations.	Effluent from the sediment basins are now included as Locations 21A and 21B.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-16	Page 6: Process Wastewater - "Process wastewater samples will be collected as grab samples from the Chemours Monomers IXM Area, Chemours PPA Area, Kuraray and DuPont (Locations 16 to 19). The project team is working with Chemours, Kuraray and DuPont facility staff to identify and access the appropriate sampling points for the combined process wastewater from each of these areas." Discrete process wastewater samples should be collected from Kuraray and DuPont.	The appropriate discrete sampling points have been identified for these locations. They are described in Table 3 and identified in Figure 2.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-17	All PFAS reported as a result of the 537.1 test method shall be reported in the results including PFOS and PFOA (i.e. Table 1 seems incomplete only one compound is listed for Method 537).	All PFAS included in Method 537 Mod are now included in the workplan.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-18	Page 8: Equipment, Trip and Field Blanks - "Equipment blanks will be collected at a frequency of one per day during the Initial Characterization, then their frequency and the need for other blanks will be re-evaluated for Ongoing Sampling." Because contamination can be random as well as systemic, analysis of all three types of blanks, not just if there is a value above the PQL for Equipment Blank should be required.	The workplan is revised to note that equipment blanks, trip blanks, and field blanks will be analyzed during each sampling event.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-19	On the first page, a) it should say "requirements specified in Paragraph 11(b)" (rather than "Paragraph 11" since there are additional requirements and plans due under Paragraph 11); b) off-site disposal is for all Chemours process wastewater (not just process wastewater containing GenX); c) Chemours uses "emissions" to refer to both air and water; should it be "discharges" for water?	The text has been revised as recommended.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-20	Section 3.3.1 – Are Clean Water Act approved methods [40 CFR Part 136] being used for the Field Parameter measurements taken during sampling. If so, those SOPs should be included with the document.	Field parameters will be measured according to protocols described in the USGS National Field Manual for the Collection of Water Quality Data (USGS, 2008).
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-21	Section 3.3.2 – where are the boundaries for spatial and temporal composite sampling defined? Please reference or provide detail for consistency in sampling events and documentation requirements.	The boundaries for temporal composite samples are described above following a similar comment from EPA Region 4. Spatial composite samples are no longer included in the workplan, and NCCW samples will be collected as individual grab samples during the first sampling event instead.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-22	Section 3.5.1 – Field duplicates are used to assess the precision associated with sample collection, handling and storage procedures as well as laboratory analytical processes. This statement should be added to the first sentence under Field Duplicates.	The text has been revised as recommended.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-23	The map should clearly and accurately depict all sampling locations. Additionally, clearer descriptions of the sampling points should be provided (specifically, sampling points #14, #9, and #4.)	The updated version of Figure 2 identifies the specific location for each sample. The sampling points for locations 4, 9, and 14 have all been specified following a site walk.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-24	Sampling point #9 was unclear; however, we request that there be a sampling point prior to the Chemours discharge located at pipe A26 (as marked on Eddie's map), one of the discharge from A26, and one after the discharge from A26 but prior to the open channel going underground.	The updated version of the plan includes the addition of three NCCW samples at Monomers IXM Locations 24A, 24B, and 24C. Location A26 is the NCCW from Vinyl Ethers South, which is captured at Location 24A. In addition, the updated workplan specifies two samples to capture the effluent from Monomers IXM, at Location 9 and 10. These new sampling locations should satisfy the intent of this request.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-25	In addition and if possible, we would like a separate sample from the Kuraray Sentry Glass process wastewater and lab waste areas (separate from the stormwater). This additional sampling location should be marked on the map.	The process wastewater from Kuraray Sentry Glass is included as Location 23. The laboratory wastewater will be collected as part of the stream of water exiting the Wastewater Treatment Plant captured at Location 8 using an autosampler. This collection point represents the contributions of the Wastewater Treatment Plant to the drainage network and Outfall 002.

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PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-26	Please add a sampling point halfway between Outfall 001 prior to comingling with discharge at points #13 and 15.	A sampling point (Location 7B) has been added between Outfall 001 (the Wastewater Treatment Plant) and the point of comingling with Locations 13 and 15 as requested. For the first sampling event, this location will be collected as a grab sample due to the delay associated with acquiring additional autosamplers. For subsequent sampling events, this location will be collected as a temporal composite sample.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-27	Please show on the map where the DuPont process wastewater samples will be taken from both DuPont facilities (i.e. at the collection sump for the WWTP). Samples should be collected from both DuPont facilities and marked as two separate samples in the Plan.	DuPont process wastewater samples will be collected at locations 19A and 19B, located in the WWTP collection sump on the updated version of Figure 2. Sample 19A comes from DuPont Plant 1 and sample 19B comes from DuPont Plant 2.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-28	Does Chemours have a device to measure the volume of flow that is discharged from the sedimentation basins be measured?	Chemours does not have a device to measure flow from the sedimentation basins.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-29	Does the stormwater from sampling point #3 cross under the road to the wood-lined ditch in a pipe? We were unable to locate this pipe during our visit.	Yes, Location 3 crosses from the open ditch under the road to the wood lined ditch in a pipe.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-30	Please clarify what is being sampled at point #4. Is this stormwater from Kuraray's Sentry Glass manufacturing area? (We may have clarified this in our discussion, but just in case we still want to ask Chemours to clarify this in the Plan because the naming of the Kuraray manufacturing areas is slightly confusing.)	Location 4 includes stormwater from Kuraray Sentry Glass and from Chemours PPA.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-31	Clearly define sampling point #14. Will two separate NCCW samples be taken from each of the DuPont facilities or will this be one sample? Where exactly is this sample being taken?	In the updated version of Figure 2, location 14 is one sampling point, collected from the ditch at the southeast corner of the DuPont plant which includes NCCW and stormwater.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-32	1. For clarification, does sampling point #15 include stormwater and non-contact cooling water from Chemours' Vinyl ethers north, Vinyl ethers south, and the IXM manufacturing areas? Will this sample contain any water from DuPont?	Location 15 includes stormwater and NCCW from the Chemours Monomers IXM area, which includes Vinyl Ethers North and South. No water from DuPont is captured in this sample.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-33	1. Also, please specify what type of DI water treatment system is used by DuPont (i.e. reverse osmosis, ion exchange).	DuPont receives filtered water from Chemours and treats it with ion exchange and reverse osmosis.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-34	As requested, the revised sampling plan included a separate sample of the wastewaters from Kuraray Sentry Glass (point 23). Please state in the plan, that if the single sample taken from the Kuraray lab shows PFAS levels above water intake concentrations, additional lab samples will be collected in the future.	A sample of the Kuraray Laboratory Wastewater will be collected during the first sampling event as Location 23B (original Location 23 becomes 23A). If analytical results indicate PFAS concentrations greater than historical ranges for intake water then Location 23B will also be sampled in subsequent sampling events.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-35	What sampling point includes stormwater from Chemours' Vinyl ethers north and Vinyl ethers south manufacturing areas? Please identify that sampling point in Table 3's Descriptions.	Stormwater falling in the concrete containment areas of Vinyl Ethers North (VEN) and Vinyl Ethers South (VES) is currently directed to a sump and is subsequently sent offsite for disposal. Rainwater falling adjacent to these containment areas in VEN and VES will be captured in samples collected at Location 9. The description in Table 3 has been modified to note that this location includes stormwater falling in the Monomers IXM Area including VEN and VES.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T1-36	Section 5 – It is unclear what data deliverables will be provided. We request that the data table submitted with the quarterly reports includes the sampling data for each point, as well as any pertinent blank sample data. We request this table be set up in sequential order following the direction of flow to Outfall 002.	The quarterly reports will provide at minimum the following data deliverables: 1. A data table of PFAS sample results that have become available since the prior quarterly report including blank sample results; 2. A map of sample locations with detected PFAS results plotted (HFPO-DA; Total Table 3+; Total EPA 537 Mod); 3. An appendix with measured field parameter results; and 4. An appendix of laboratory reports.
PFAS Characterization Sampling Plan	CFRW	T1-37	Figures 1 and 2 only show the "known drains and ditches." Chemours should analyze the site for other areas that receive process and non-process wastewater flow and stormwater flow during rain events. Only then can Chemours adequately characterize all process and non-process wastewater and stormwater streams from each of the facility's manufacturing areas.	Chemours has additional site maps that identify all drains and ditches at the facility. These are available upon request. Under Paragraph 12 Outfall 002 Reductions, Chemours is conducting additional non-routine sampling to characterize other drainage ditches on site, however these locations are not included in this workplan. This plan includes process wastewater, non-process wastewater, and stormwater in the main drainage ditches.
PFAS Characterization Sampling Plan	CFRW	T1-38	On page 5 of the plan, it states that "Locations 4, 7, 10, 12, 13, and 15 are a combination of stormwater and treated process and non-process water." However, we were informed during the site visit that not all of the locations contain all three types of water (combination, process, and non-process water). Looking at the sampling locations, it appears that locations 4, 7, 10, and possibly 13 and 15, should not contain process wastewater. Please clarify exactly what types of water are at each location.	The updated version of Figure 2 identifies the types of water found at each location.
PFAS Characterization Sampling Plan	CFRW	T1-39	At the very least, Chemours should sample at the intake and discharge for each of the manufacturing areas at the facility. It is not entirely clear that is being done within this sampling plan.	Chemours is sampling the Intake from the Cape Fear River (Location 1) that goes to each manufacturing area at the facility. Process wastewater from each manufacturing location is already included in the workplan.
PFAS Characterization Sampling Plan	CFRW	T1-40	The sampling plan should also include a sampling location at Chemours' intake location for the Cape Fear River. Sample location 1 is not representative of actual background concentrations in the Cape Fear River.	The direct intake pipe in the Cape Fear River is not included in the workplan due to health and safety risks in accessing this location. Location 1, water directly from the intake system, is representative water drawn from the Cape Fear River and background PFAS levels in water used at Site.
PFAS Characterization Sampling Plan	CFRW	T1-41	The sampling plan should include more sampling locations along the cooling water channel, particularly the areas that historically carried process wastewater in order for the sampling to be representative of potential contamination throughout that channel.	The cooling water channel was not a conduit for process wastewater, the Terra Cotta pipe was used to convey process wastewater to the wastewater treatment plant. The Terra Cotta pipe section which was used solely by Chemours has now been fully grouted. The cooling water channel was lined as of November 2018. It does not receive groundwater. We do not believe that additional sampling locations along this channel are necessary for this routine characterization program.

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<b>Document</b>	<b>Comments From</b>	<b>Comment ID</b>	<b>Comment</b>	<b>Proposed Responses</b>
PFAS Characterization Sampling Plan	CFRW	T1-42	<i>The sampling plan does not include a sampling point in the open channel to Outfall 002 anywhere from location 7 to location 20. There should be more sampling points along this channel (i.e., after it receives discharges from the WWTP, from the cooling water channel, and from DuPont's ditches).</i>	This workplan is meant to conduct routine evaluation of streams entering Outfall 002. All inputs to the open channel are currently included in the workplan. Additional sampling of other locations in the Site Drainage Network will be conducted under Paragraph 12 Outfall 002 Reductions, however these locations are not part of this workplan.
PFAS Characterization Sampling Plan	CFRW	T1-43	<i>Flow should be analyzed as well to determine mass loading of PFAS from each stream.</i>	Flow readings will be collected where possible and results will be reported in the quarterly reports to the NCDEQ.
PFAS Characterization Sampling Plan	CFRW	T1-44	<i>How does DuPont's process wastewater reach the wastewater treatment plant?</i>	DuPont's process wastewater is collected in a tank at the DuPont facility then released as a batch via underground piping to a sump where it combines with other sources to the WWTP.
PFAS Characterization Sampling Plan	CFRW	T1-45	<i>How does Kuraray's process wastewater reach the wastewater treatment plant? Through the wood-lined channel?</i>	Kuraray's process wastewater continually flows via the Terra Cotta pipe to a sump where it combines with other sources to the WWTP. Note, the section of Terra Cotta pipe which was used solely by Chemours has been fully grouted.
PFAS Characterization Sampling Plan	CFRW	T1-46	<i>Why is there non-process wastewater at location 4? Is there non-process wastewater in areas of the site outside of the cooling water channel as depicted in Figure 2?</i>	There is no non-process wastewater at Location 4, only stormwater.

**References**

AECOM (2018). Poly and Perfluoroalkyl Substance Quality Assurance Project Plan. August, 2018  
 USGS (2008). Field Measurements: U.S. Geological Survey Techniques of Water-Resources Investigation Book 9, Chapter A6.

**Notes**

CFRW - Cape Fear River Watch  
 DWR - Division of Water Resources  
 EPA - Environmental Protection Agency  
 NCDEQ - North Carolina Department of Environmental Quality  
 ORD - Office of Research and Development

RESPONSES TO COMMENTS  
TABLE 2 - PARAGRAPH 11(a) NON-TARGETED PLAN

Document	Comments From	Comment ID	Comment	Proposed Responses
PFAS Non- Targeted Analysis and Methods Development Plan	EPA Region 4	T2-1	<i>The use of high-field NMR would be of potential benefit in structural identification of unknown compounds.</i>	While this is true for unknown PFAS at high concentrations, the unknown PFAS in these samples are expected to be present at sub-ppb levels. NMR methods are not sensitive enough to support compound identification at these low levels.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T2-2	<i>Non-Targeted Analytical Background – what will be done in the absence of an authentic standard?</i>	As discussed in section 3.3.2, if authentic standards are not commercially available, standards will be synthesized for this purpose.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T2-3	<i>Scope and Rationale – how is the “...prioritized set of the highest abundance additional PFAS,” set? What is the cutoff for the highest?</i>	Chemours is planning to select the five largest unknown peak areas in each sample chromatogram to proceed with enhanced assessment to the extent possible under the schedule requirements. To date prior assessment of unknown Q-TOF-MS peaks have resulted in the 23 compounds being identified and the development of the Table 3+ analytical method. Therefore, any potentially remaining unknown peaks are likely to be much lower in intensity and as a consequence likely comprise a minimal fraction of sample PFAS mass.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T2-4	<i>Enhanced Assessment - Again, how is the prioritization conducted?</i>	As noted above, Chemours is planning to select the five largest unknown peak areas in each sample chromatogram to conduct enhanced assessment.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T2-5	<i>Reporting - Include that the QTOFMS datafiles will be provided to DEQ.</i>	Chemours can share Q-TOF-MS data files and chromatograms with NCDEQ upon request.

**Notes**

EPA - Environmental Protection Agency  
ORD - Office of Research and Development

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**TABLE 3 - ANALYTICAL METHODS**

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PFAS Characterization Sampling Plan	EPA ORD	T3-1	<i>The PQLs listed in Table 2 for most of the analytes is too high at 50-120 ng/L . PQLs from laboratories have been 5-10 ng/L.</i>	The PQLs (now listed in Table 1) were improved by the laboratories after initial submission of this workplan. The PQLs reflect the current capabilities of the analytical laboratories.
PFAS Characterization Sampling Plan	EPA ORD	T3-2	<i>TestAmerica and Eurofins Lancaster Current PQL's are higher than expected.</i>	As noted above, the PQLs (now listed in Table 1) are revised.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-3	<i>It appears that samples may either be sent to Eurofins or TestAmerica [both are referenced in Tables 2 and 4]. Please submit the TestAmerica's SOP(s) so we can understand how any of the standard operating procedures may differ between labs.</i>	Revised SOPs from both laboratories will be provided in the near future.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-4	<i>Section 4.2 – If SPE is used, Method Blanks should be used at a frequency of one per extraction batch rather than one per 20 samples when an extraction batch is less than 20 samples. This will test the day-to-day variability between extraction batches.</i>	The laboratory will perform one method blank per extraction batch, where an extraction batch is not to exceed 20 field samples.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-5	<i>Section 4.2 – Since this is a location- and time-limited project and since the bias these matrices may attribute to these methods of analyses for these compounds is unknown, each sample location should be characterized with Matrix Spikes rather than relying on random selection during through ongoing QC requirements.</i>	Matrix spikes are analyzed for every sample under the Table 3+ SOP. For Method 537 Mod, internal spikes are analyzed for every sample.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-6	<i>Section 4.2 – Indicate that CCVs are analyzed initially, after so many samples and at the end of each analysis batch to bracket samples analyzed.</i>	CCVs are analyzed before sample analysis, after every 10 samples until the analytical sequence ends, and at the end of the batch.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-7	<i>Section 5 – Provide all associated QC results and not just case narratives.</i>	All QC results will be provided, as is the case for all Level 4 Reports.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-8	<i>Table 2 – There are fairly significant differences in PQLs between the two contract laboratories, TestAmerica and Eurofin Lancaster. Please describe how it is determined which lab is used.</i>	The PQLs (now listed in Table 1) were improved by the laboratories after initial submission of this workplan, and are now identical for Table 3+ SOP between the two laboratories. The PQLs reflect the current capabilities of the analytical laboratories.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-9	<i>It appears that Eurofins is not using the branched isomers of PFOS, PFHxS, NetFOSAA and MeFOSAA in their calibration curves. What the rationale for that decision? Eurofins does analyze a solution at the mid-point of the curve that contains the previous analytes and their branched isomers. This is simply done for the analyst so they know how to fully integrate the analytes in real samples. As such it seems that Eurofins is capturing the linear/branches isomers when they analyze real samples, but they are not including them in the calibration. The extraction/cleanup was left out as “proprietary”.</i>	In the updated SOP, branched isomers for these compounds are included in the calibration curve.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T3-10	<i>Table 2 – There are fairly significant differences in PQLs between the two contract laboratories, TestAmerica and Eurofins Lancaster, and there is no description of how it is determined which lab is used. Please describe the process for determining which lab will be analyzing samples. The greatest discrepancies between the PQLs is seen with 4:2, 6:2, and 8:2 fluorotelomersulfonic acids, and NEtFOSAA. If TestAmerica analyzes a sample and the concentration is non-detect at their PQL of 20 ng/L, please send a sample to Eurofins Lancaster to verify non-detect at their lower PQLs for these parameters in particular. (The other differences in PQLs are not as concerning.)</i>	Once Eurofins Lancaster is approved by NCDEQ, both laboratories may be used depending on their availability and capacity to run samples. The fluorotelomersulfonic acids and NEtFOSAA are not produced or used at the Chemours facility. Chemours will report the data provided by the laboratory that analyzed the data.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T3-11	<i>Sample Preparation - How is it determined what gets SPE? If after a direct injection is done and no peaks are seen or many large peaks are seen, how does the process proceed? Results can indicate many PFAS of widely varying concentrations.</i>	If direct injection results in a chromatogram with many large peaks, Chemours will add an internal standard to the sample to provide a reference concentration for the unknown peaks. If there are no peaks, an internal standard will be added and Chemours will conduct solid phase extraction to also enhance the sensitivity of the analysis.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T3-12	<i>Compound Identification - Direct Injection is the least sensitive approach. If no peaks are detected via direct injection how do you proceed?</i>	As noted above, if no peaks are detected, solid phase extraction will be used to enhance the sensitivity. Chemours may also use large volume injection as an alternative method to improve sensitivity.
Table 3 SOP	EPA ORD	T3-13	<i>Sample Preparation – dilutions listed for calibration curve do not include values low enough to support 5-10 ppt PQL.</i>	The laboratory SOPs have been revised to address this comment.
Table 3 SOP	EPA ORD	T3-14	<i>Note on mixed stock standard - has the stock stability on water been assessed, and then assessed as stored in a refrigerator?</i>	Experiments are being planned to answer this question.
Table 3 SOP	EPA ORD	T3-15	<i>“A 2x dilution will be appropriate for most groundwater analyses (trace levels expected).” This is not the case for Chemours groundwater nor many of the surface water and effluent samples.</i>	A 2x dilution will be used initially, then further dilution will be carried out as needed for higher concentration samples.
Table 3 SOP	EPA ORD	T3-16	<i>QC sample: “... stock standard solution separate from the one used for calibration...” Is this a secondary standard prepared from the same chemical or from a secondary source?</i>	The laboratory SOPs have been revised to address this comment.

**RESPONSES TO COMMENTS**  
**TABLE 3 - ANALYTICAL METHODS**

Document	Comments From	Comment ID	Comment	Proposed Responses
Table 3 SOP	EPA ORD	T3-17	<i>Analytical Method: LC Operating Conditions: Column A – given a 50:50 methanol:water is injected into a 85:15 water:acetonitrile, you would expect early eluting peaks to split and not chromatograph well.</i>	The laboratory SOPs have been revised to address this comment.
Table 3 SOP	EPA ORD	T3-18	<i>QQQ Acquisition Parameter – major concern in monitoring only one MRM for all of the compounds. Modern QQQs such as your Agilent 6470 has no problems with two MRMs for all if available, which makes ion ratios possible to evaluate. If not monitored, this is impossible. One is listed for HFPO-DA but for no others. No stable isotope labeled standards are listed. Most may not be currently available but Wellington Labs does have 13C3 HFPO-DA.</i>	Chemours is exploring adding additional MRMs to the method in the future. There must be a balance between sensitivity and selectivity, and sensitivity has been the primary focus to date.
Table 3 SOP	EPA ORD	T3-19	<i>Data Processing – reference indicates “The curve should be linear,...” Dr. Strynar indicates quadratic generally works better even if the data is linear. If the data is non-linear, a linear fit is not good.</i>	The laboratory SOPs have been revised to address this comment. Both linear and quadratic curves can be used.
Table 3 SOP	EPA ORD	T3-20	<i>Blanks – is the target analyte concentrations in all blanks defined as 0.01 ppb?</i>	The laboratory SOPs have been revised to address this comment.
Table 3 SOP	EPA ORD	T3-21	<i>Duplicates – how will you work with data when one sample is above the LOQ and one is below the LOQ?</i>	If one result is less than the LOQ and the other is not, and if the two results differed by a value less than the LOQ, the replicate is said to have met the acceptance criteria. In this case, the result that is above the LOQ will be reported.
Table 3 SOP	EPA ORD	T3-22	<i>Appendix A - Please add molecular weight as another column of information in the table. DFSA, Byproduct 4 and Byproduct 5 – are diprotic, what about M-2H? MMF – this compound is diprotic and low molecular weight. The MRM is for the M-H. It will be missed if it is M-2H as that MRM is not monitored for. The structure given is difluoromalonate. What about monofluoromalonate as a possible contaminant?</i>	Molecular weight will be added to the table as requested. To date, we have seen no evidence of M-2H for the diprotic compounds on the analyte list.
Table 3 SOP	EPA ORD	T3-23	<i>Cover letter dated January 30, 2019 from Brian Long “The lab standards are in the form of a 0.1% (by weight) solution in water for each compound, and Chemours can ship these standards solutions to DEQ promptly upon request.” - have stability tests for standards in this solution been performed?</i>	Experiments are being planned to answer this question.
Table 3+ SOP	EPA Region 4	T3-24	<i>Details in this SOP are minimal, with limited procedural information available. A more detailed version of the methodology is necessary for evaluation of the procedures.</i>	The laboratory SOPs have been revised to address this comment.
Table 3+ SOP	EPA Region 4	T3-25	<i>Most of the target analytes are not available commercially. Suggest that funding be directed toward synthesis of these compounds with non-related companies which specialize in production of analytical laboratory standards so that primary and secondary source standards are available. Doing so will give greater validity to the data.</i>	Upon NCDEQ request Chemours will provide samples synthesized compounds.
Table 3+ SOP	EPA Region 4	T3-26	<i>Page 1, Sample Preparation. The note states that the 100 ppb mixed stock standard is prepared in ultrapure water. Studies have shown that PFAS will bind to the sample container in the absence of an organic solvent. Preparing stock or mixed standards in water may cause low bias in quantitative results because of PFAS binding to the sample container.</i>	Experiments are being planned to answer this question.
Table 3+ SOP	EPA Region 4	T3-27	<i>The 10-point calibration curve spans a concentration range from 10 – 50,000 ng/L. This calibration range seems to span a range greater than possible with MS/MS detection. Clarify how this is performed.</i>	The laboratory SOPs have been revised to address this comment.
Table 3+ SOP	EPA Region 4	T3-28	<i>Page 2, Sample Preparation. The QC samples are prepared at a concentration of 5 ppb (5,000 ng/L). Relative to the NC DHHS provisional health goal of 140 ng/L for HFPO-DA and the US EPA health advisory for PFOA and PFOS of 70 ng/L, the QC spike concentrations are quite high and not representative of protective health goals.</i>	The laboratory SOPs have been revised to address this comment. QC samples will be prepared at lower concentrations more representative of protective health goals.
Table 3+ SOP	EPA Region 4	T3-29	<i>Page 4, QQQ Acquisition Parameters. If possible, add confirmatory ions where possible. The qualifier ion ratio and retention time criteria for selection or exclusion of a compound should be stated. This may require multiple MRM functions to obtain an adequate number of scans across peaks.</i>	The laboratory SOPs have been revised to address this comment.
Table 3+ SOP	EPA Region 4	T3-30	<i>Page 5, Data Processing, Calibration. No mention is made of the minimum number of scans required across a chromatographic peak. A minimum of eight and preferably ten scans are required to produce quantitative data.</i>	The laboratory SOPs have been revised to address this comment.
Table 3+ SOP	EPA Region 4	T3-31	<i>Performing two calibration curves; one prior to sample analysis and one following the sample sequence is not a technique typically seen. This may have the potential to introduce bias to the determinations, especially if absolute instrument sensitivity has been lost during analysis. If the practice is retained, precision and accuracy criteria should be developed to judge acceptability of the closing calibration curves with respect to the opening calibration.</i>	The laboratory SOPs have been revised to address this comment.
Table 3+ SOP	EPA Region 4	T3-32	<i>Page 5, Data Processing, Blanks. As currently written, method blanks are optional (e.g., and/or). The requirement for a method blank is mandatory.</i>	The laboratory SOPs have been revised to address this comment.
Table 3+ SOP	EPA Region 4	T3-33	<i>Page 5, Data Processing. Suggest adding a positive control spiked at a concentration equal to the minimum reporting limit to verify method performance at the limit of quantitation. A 5% frequency is reasonable (One per batch of 20 samples).</i>	The laboratory SOPs have been revised to address this comment.
Table 3+ SOP	EPA Region 4	T3-34	<i>There is no mention of a method detection limit study. Suggest implementation of an MDL study.</i>	The laboratory SOPs have been revised to address this comment.

**RESPONSES TO COMMENTS**  
**TABLE 3 - ANALYTICAL METHODS**

Document	Comments From	Comment ID	Comment	Proposed Responses
Table 3+ SOP	EPA Region 4	T3-35	<i>Page 5, Data Processing, Duplicates. Suggest eliminating the RPD 50% criterion and using a single 25% criterion for all duplicates.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-36	<i>The use of isotope dilution, where allowed, is to be commended. Isotope dilution and extracted internal standards allow recoveries of target analytes to be corrected for losses during sample processing.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-37	<i>The dependencies between internal standards (ISTDs) and the compounds they are used to quantitate seems reasonable. Chemistry and chain length of ISTDs generally corresponds to target compounds.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-38	<i>Branched isomers not in ICAL – are they included elsewhere? Integration of samples should include both linear and branched chain isomers, where present. This is implied in section D.12 (page 16) but needs clarification. If branched chain isomers are not included during integration, the potential for underestimation of results exists.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-39	<i>No MRL confirmation performed. Recommend that a positive control sample spiked at the MRL concentration be carried through the process to verify method performance.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-40	<i>With respect to the SOP title, and the use of “Modified” 537 1.1: The allowed changes to Method 537 are limited and clearly stated. Once those allowed changes are exceeded, the method is no longer considered Method 537. There have been major chemistry changes made to this SOP when compared to Method 537. The title connotes something that this method is not; this method can no longer be considered Method 537.</i>	Method 537 was used as a baseline and modified according to analytical requirements. It is referred to as "EPA 537 Modified" to reflect this.
Method 537 Mod SOP	EPA Region 4	T3-41	<i>Redaction of certain SOP elements as proprietary make evaluation of the method in its entirety impossible. The SPE and cleanup sections are critical to full evaluation of the method.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-42	<i>There is no mention of HFPO-DA (aka, GenX) within the document. Presumed to not be a target compound.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-43	<i>Page 5, Precaution to minimize method interference. This section is redacted as proprietary content. No evaluation can be made.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-44	<i>Pages 6 – 7, Sample collection/preservation. Trizma is not added to samples unless they originate from a chlorinated source. Method 537 uses Trizma as both a buffer and free-chlorine scavenger. Its use in 537 increased recoveries for several compounds using SDVB SPE (unpublished data). It is impossible to know what effect Trizma will have on the samples analyzed by this method because of the (1) major differences in chemistry and (2) the addition of compounds relative to Method 537.</i>	Chlorinated drinking water supplies are not being sampled for this project.
Method 537 Mod SOP	EPA Region 4	T3-45	<i>Page 10, Initial Calibration (A.2). States there must be a detection of all analytes in the MDL standard. If there is a signal-to-noise requirement for this criterion, state it.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-46	<i>Page 10, Initial Calibration (A.1/A.4). Section A.1 requires a minimum of five calibration points. Section A.4 provides for the allowance of a second-order (quadratic) calibration model. While not stated in Method 537, quadratic calibration requires a minimum of six calibration points. Likewise, a first-order model requires a minimum of five calibration points. These requirements should be stated in the SOP.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-47	<i>Page 11, Continuing Calibration (7.B.1.a) The requirement for use of the CS3 level standard is stated. Method 537 §10.3 states “The beginning CCC of each analysis batch must be at or below the MRL in order to verify instrument sensitivity prior to any analyses.”</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-48	<i>Page 11, Procedure, Sample Preparation (A.1) State how many significant figures are necessary during weighing. Also applies to page 12, section A.3.j, and elsewhere.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-49	<i>Page 11, Procedure, Sample Preparation (A.3) The option for processing of an aliquot is described. Numerous workers have shown that PFAS sorption to sample containers is a source of sample loss and non-quantitative transfer. If aliquots are taken from the original sample container, the sample must be qualified explaining the possibility for low bias in reported values.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-50	<i>Pages 12 - 13, Procedure, Solid Phase Extraction (B.1 - 16) Most (16 of 21) procedural steps of this section, which are critical to successful sample analysis have been redacted as proprietary. As such, no judgement of the extraction can be made. NOTE: EXTRACTION IS A CRITICAL STEP IN THE ANALYSIS.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-51	<i>Pages 13 - 14, Procedure, Solid Phase Extraction (B.18 -19) Steps 18 (tare bottle) and 19 (weigh bottle) appear to conflict with each other. Clarify.</i>	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-52	<i>Page 14, Procedure, Solid Phase Extraction (B.20) The SOP states that following addition of labeled internal standards, the sample is ready for instrumental analysis. However, section C (Extract Cleanup) follows this stated sample processing endpoint. Clarify the flow of the sample extract and state if Section C Extract Cleanup is optional. In section B.20, state what equipment is allowed or disallowed (pipette, syringe, etc.) for sample transfer.</i>	The laboratory SOPs have been revised to address this comment.

**RESPONSES TO COMMENTS**  
**TABLE 3 - ANALYTICAL METHODS**

Document	Comments From	Comment ID	Comment	Proposed Responses
Method 537 Mod SOP	EPA Region 4	T3-53	Page 14, Procedure, Extract Cleanup (C) All procedural steps of this section, which are critical to successful sample analysis have been redacted as proprietary. As such, no judgement of the cleanup procedure can be made. NOTE: CLEANUP STEPS ARE CRITICAL TO THE ANALYSIS.	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-54	Page 14, LC/MS/MS Analysis, Mass Calibration and Tuning (D.1.b) The SOP provides a criterion for mass axis calibration to be within $\pm 0.5$ Daltons of the true value. There is no statement indicating how this is performed. Note: Failure to maintain mass calibration will impact instrument sensitivity.	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-55	Page 15, LC/MS/MS Analysis, Mass Calibration and Tuning (D.3) Acquisition Method, Attachment 3 is redacted indicating it to be proprietary. Therefore, no judgement of the LC/MS/MS acquisition procedure can be made. NOTE: ACQUISITION PARAMETERS ARE CRITICAL TO THE ANALYSIS.	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-56	Page 15, LC/MS/MS Analysis, (D.5) The acronym L/B standard is used but undefined; assume this to be Liner/Branched-chain. Clarify.	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-57	Page 16, LC/MS/MS Analysis, (D.11) The use of surrogates is mentioned; however, their identity is not given. Clarify.	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-58	Page 16, LC/MS/MS Analysis, (D.12) The use of an MDL standard by which to judge data quality is referenced. Its use is also mentioned in section A.2 (page 10). Clarify the origin of the MDL standard.	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-59	Page 18, Mass Transitions AB Sciex 4500, Attachment 1. The stated transitions appear to be nominal values. Mass transitions should be optimized on an instrument-by-instrument basis. During optimization, the analyst should determine the precursor and product ion masses to one decimal place (0.1 Daltons).	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	EPA Region 4	T3-60	The precursor and product transitions for PFOS (2) appears to be incorrect. The table entry $m/z$ 413 > 169 should probably be 499 > 99.	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-61	Information about the LC/MS/MS triple quadrupole instrumentation to be used for the Chemours PFAS sample analyses	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-62	Information about analyst technical requirements and demonstration of analyst capability	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-63	List of standards used and source of each (please use free acid CAS #s)	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-64	Method detections of each PFAS (see attached list – please confirm if this is correct)	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-65	Table of method modifications to define how sample prep and analysis deviates from EPA Method 537.1 (See attached table)	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-66	Matrices analyzed for PFAS – phone call indicated that matrices include drinking water, surface water, product water, extracts from soils, and extracts from other sources such as vegetables and resins	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-67	DOD certified for Method 537, or other certifications	The laboratory SOPs have been revised to address this comment.
Method 537 Mod SOP	NCDEQ, DWR	T3-68	When use Method 537 or 537.1, what type of SPE column is used	The laboratory SOPs have been revised to address this comment.

**Notes**

DWR - Division of Water Resources  
EPA - Environmental Protection Agency  
NCDEQ - North Carolina Department of Environmental Quality  
ORD - Office of Research and Development

**RESPONSES TO COMMENTS**  
**TABLE 4 - GENERAL METHODS**

Document	Comments From	Comment ID	Comment	Proposed Responses
PFAS Characterization Sampling Plan	EPA Region 4	T4-1	Page 5, second paragraph, seventh sentence: Provide details for the Hach SD900 and the other composite samplers. Are they refrigerated or capable of storing samples on ice? Is there one container for the aliquots that will comprise the of temporal composite sample or are there several containers? What is the composition of the sampler's tubing and components?	We are not aware of any published studies that document degradation of these compounds due to ambient temperatures. Ice is often used to cool waters to mitigate biodegradation processes. At this time ice is not planned for use in the autosamplers. As noted in an earlier response to a comment, Teledyne 6712C autosamplers are being used for temporal composite sample collection, which have HDPE sample reservoirs and use HDPE tubing and silicon tubing. Samples will be composited into one 2.5 gallon HDPE vessel and subsequently poured into 250 mL HDPE bottles after sample collection.
PFAS Characterization Sampling Plan	EPA Region 4	T4-2	Page 7, 3.3.3 Decontamination Procedures: Tap water and de-ionized water should be analyzed for PFAS and the one with the lower PFAS constituents should be used as the final rinse in the decontamination procedure. If they both exceed the criteria to achieve the DQOs for the project, PFAS-free water should be used as the final rinse. Decontaminated equipment should be	Deionized water is used as the final rinse in the decontamination procedure, and rinsate blanks have been consistently non-detect for PFAS. Equipment is typically decontaminated immediately before use. If sample collection is delayed, sampling equipment is covered with PFAS-free plastic until it is ready for use.
PFAS Characterization Sampling Plan	EPA Region 4	T4-3	Page 7, 3.4 Sample Shipping, Chain of Custody, and Holding Times, first sentence: To prevent ice melt water from potentially contaminating the samples, it is recommended that the samples be bagged in Ziplocs® or Whirl-Paks®.	During shipment, samples will be stored in a large heavy plastic bag to keep melt water from potentially contaminating the samples as outlined in the QAPP (AECOM, 2018).
PFAS Characterization Sampling Plan	EPA Region 4	T4-4	Page 7, 3.4 Sample Shipping, Chain of Custody, and Holding Times, first sentence: Composite sampling requiring ice preservation, should have composite aliquots stored on ice or refrigerated during the sampling process.	As noted in a response to a comment earlier, ice is not planned for use in the autosamplers.
PFAS Characterization Sampling Plan	EPA Region 4	T4-5	Table 4: In the preservation column, "Ice" should be listed for each of the analytical methods.	While ice is being used in the coolers to limit overheating during transport, the preservation column is intended to refer to chemical preservatives. No chemical preservatives are being used.
PFAS Characterization Sampling Plan	EPA ORD	T4-6	The plan does not incorporate any spiked field sample or spiked trip samples to assess recovery and accuracy of the intended analytes. Table 5 does indicate the plan for matrix spikes which seem to be prepared on the day of analysis. EPA and DEQ have implemented trip spikes in our project. Spiked field sample or spiked trip samples are essential. If not done it is hard to assess what happens to samples during holding times, while shipped on ice, frozen and archived and then extracted.	Due to the difficulty of spiking in the field and risk of contamination, spiked field samples are not being included. As far as Chemours and its subcontractors are aware, spiked trip samples are not standard practice for PFAS characterization. There are no established protocols for spiked trip samples including the compounds to be spiked and the concentrations to target. We do not believe that spiked trip samples are necessary for the objectives of this program.
PFAS Characterization Sampling Plan	EPA ORD	T4-7	Sample Collection Procedures Common to All Locations. Page 5, 5th line: "...archive for potential future analyses." How are these samples going to be managed? Frozen? Extracted and archived?	Archived samples will be refrigerated at <6°C, as noted in the Standard Operating Procedures for Method 537 (Modified) (TestAmerica, 2018).
PFAS Characterization Sampling Plan	EPA ORD	T4-8	Field QA/QC. The plan does not include spiked field samples. Chemours has all of these standards for assessment. Add: 1) spiked blank water samples and/or 2) spikes of field collected sample. These samples should go out into the field (or are spiked in the field) and are cooled, transported and extracted as unknown samples are managed. This will determine if there is good recovery for the chemicals analyzed, which determines accuracy. The assessment of blank contamination and duplicate precision is in the proposal but NOT accuracy. Also, this is NOT the same as the Matrix Spikes in section 4.2 below as the samples here are in the lab on the day of analysis.	As noted above, spiked trip samples and spiked field samples are not planned for this program.
PFAS Characterization Sampling Plan	EPA ORD	T4-9	Spike sample must be included for Field samples.	As noted above, spiked trip samples and spiked field samples are not planned for this program.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-10	Section 3.3.2 – NOTE: Thick size markers (e.g., Sharpie and others) are to be avoided for sample labelling as they may contain PFAS.	Markers to be used for sample labelling do not contain PFAS.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-11	Section 3.3.2 – Powderless nitrile gloves should be used and changed between samples. Gore-Tex or other water-resistant, insect-resistant, or UV-protected synthetics or products should not be worn during sampling.	Powderless nitrile gloves will be used during sampling and will be changed between sample locations. PFAS-containing products will not be worn or used during sampling.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-12	Section 3.4 – Define storage temperature for archived samples since we do not know how long archived samples may be stored. What type of QA/QC samples will be maintained (e.g., Field Blank) should those archived samples be analyzed at a much later date?	Archived samples will be refrigerated at <6°C. All QA/QC samples will be analyzed in the same batch as field samples, but will also have bottles archived for potential future analysis.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-13	Section 3.4 – The section states that sample coolers will be "taped shut" and signed across the lid of the cooler. It is unclear what kind of tape is to be used. The use of custody seals is recommended, and the bill of lading is to be retained with the sample records.	Coolers are sealed by taping both ends with clear packing tape, with the custody seal under the tape on one side.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-14	Section 3.5.1 – Recommend the use of Temperature Blanks to verify proper thermal preservation during shipment.	As a general standard practice temperature blanks are already routinely included in coolers sent by the laboratories.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-15	Section 3.5.1 – Because Field Blanks are required by the reference method, please analyze them regardless of detections in the Equipment blanks.	In the updated version of the workplan, field blanks will be analyzed at a frequency of one per sampling day regardless of the equipment blank results.

**RESPONSES TO COMMENTS**  
**TABLE 4 - GENERAL METHODS**

Document	Comments From	Comment ID	Comment	Proposed Responses
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-16	<i>Table 4 – There is no mention of thermal preservation requirements [e.g., &lt;10 degrees C without evidence of freezing] for shipment and storage. Please clarify the requirements.</i>	Samples will be kept at <6°C during shipment and storage.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-17	<i>Table 4 – The significant difference in hold time [i.e., 40 vs 28 days unpreserved] used by the two contract laboratories [TestAmerica and Eurofins] is concerning. Without stability validation data in the matrices of interest, it is not known whether data may be impacted by the significantly longer hold time especially if samples are not to be thermally preserved as indicated in the Sampling Plan. The conditions for storage of extracts must also be addressed. Table 5 – Footnote 2 may need adjustment based on previous comment regarding analysis of Field Blanks and frequency of CCV based on previous comments.</i>	The holding times for each laboratory are provided by the individual laboratory. All samples will be kept at <6°C. Footnote 2 has been removed from Table 5.
PFAS Characterization Sampling Plan	NCDEQ, DWR	T4-18	<i>There isn't any mention on how the proposed methods have been validated for analysis of PFAS in the matrices involved in this project. Were any spike studies, etc. done? That would be good background information to have, if so. This type of method validation is indirectly referenced for the non-targeted analyses by reference to following EPA's Protocol for Review and Validation of New Methods for Regulated Organic and Inorganic Analytes in Wastewater under EPA's Alternate Test Procedure Program (EPA, 2016), but is not mentioned for the targeted analyses. This approach would also be appropriate for each of the proposed methods in this plan. Keep in mind that MDL and IDOC studies are generally performed in laboratory water matrix.</i>	MDL and IDOC studies are included in the validation process for PFAS in aqueous matrices. Method validation is also done by spiking stable isotope analogs and calculating the recovery for Method 537 Mod compounds and HFPO-DA.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA Region 4	T4-19	<i>The planning document overall, seems sound. There should be greater emphasis given to synthesis of authentic standards by companies which maintain an arms-length relationship with Chemours. Companies which specialize in the production of high-purity analytical standards should be focused on for their expertise. This is especially important, as new compounds are discovered and methods developed for their quantification in real-world samples.</i>	Chemours is planning to synthesize the authentic standards in-house. The types of compounds that may require synthesis are likely to have chemical properties unique to Chemours, so it will be most practical for the Chemours laboratory to synthesize them to meet the timelines required by the executed consent order. It would also be challenging to organize shipping to and from an external laboratory for newly synthesized compounds and feedstocks required for synthesis. Nevertheless, analysis by external laboratories will be important to verify the chemical structure of any newly synthesized standards, and Chemours will provide the newly synthesized standards to external laboratories upon request.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T4-20	<i>Quality Assurance/Quality Control – how are the archived duplicates going to be managed? Frozen? Extracted and archived?</i>	Archived duplicates will be refrigerated at <6°C.

**References**

AECOM (2018). Poly and Perfluoroalkyl Substance Quality Assurance Project Plan. August, 2018  
 TestAmerica (2018). SOP No. WS-LC.0025, Rev. 3.2. Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue; Method 537 (Modified), Method PFAS by LCMSMS Compliant with QSM 5.1 Table B-15. August, 2018.

**Notes**

DWR - Division of Water Resources  
 EPA - Environmental Protection Agency  
 NCDEQ - North Carolina Department of Environmental Quality  
 ORD - Office of Research and Development

**RESPONSES TO COMMENTS  
TABLE 5 - COMMENTS UNRELATED TO PARAGRAPH 11 PLANS**

<b>Document</b>	<b>Comments From</b>	<b>Comment ID</b>	<b>Comment</b>	<b>Proposed Responses</b>
PFAS Characterization Sampling Plan	EPA ORD	T5-1	<i>Groundwater analysis is not included. Comments on groundwater analysis may be provided separately.</i>	This plan is for process water, non-process water, and stormwater. Groundwater sampling and analysis is part of the ongoing groundwater monitoring programs being conducted at the Site.
PFAS Characterization Sampling Plan	CFRW	T5-2	<i>Slide 31 of Chemours' January 31, 2019 Conceptual Site Model Updates and Old Outfall 002 Presentation demonstrated that there are extremely high levels of contamination in the southeast corner of the DuPont manufacturing area. The sampling plan should include more sampling points in that area of the facility to determine the source of that contamination.</i>	This characterization plan is intended to characterize waters above ground in the drainage network at Site, not groundwater. The Southeast Perched Zone investigation installed multiple wells in this area. Paragraph 18 of the consent order will discuss the sources of PFAS contamination onsite.
PFAS Non- Targeted Analysis and Methods Development Plan	EPA ORD	T5-3	<i>Introduction - What if any are the differences in how groundwater analysis is going to be conducted?</i>	Groundwater analysis is not included in the non-targeted plan.

**Notes**

CFRW - Cape Fear River Watch  
 EPA - Environmental Protection Agency  
 ORD - Office of Research and Development