Saint-Gobain Performance Plastics Air Sample Collection Plan for Non-Targeted Per- and Polyfluoroalkyl Substances Analyses

April 18, 2018

1.0 Objective

The objectives of this sampling program are to identify the full spectrum of per- and polyfluoroalkyl substances (PFAS) in air emissions, process feedstocks, and process residuals from Saint-Gobain Performance Plastics (SGPPL) in Merrimack, New Hampshire and to assess the performance of a candidate pilot-scale air pollution control system. The identification and possible quantification of PFAS compounds will be conducted using multiple GC/LC/mass spectrometric techniques (e.g. low and high resolution time of flight preceded by gas chromatography separation and tandem mass spec preceded by liquid chromatography separation). These analyses will be conducted at the United States Environmental Protection Agency's Office of Research and Development (ORD) laboratories located at Research Triangle Park, North Carolina and Athens, Georgia.

The results of the program will be used to identify the specific PFAS compounds and related byproducts associated with air emissions and compounds that are being detected in the environment. NHDES will utilize this information to:

(1) Document emissions of fluorinated compounds utilizing current chemical formulations;
(2) Determine whether conditions warrant the installation of air pollution controls to prevent the environmental impact with the next generation of raw materials;
(3) Ensure that a pilot scale air pollution control system is designed to effectively control PFAS and associated analogues, some of which are known precursors to the formation of PFOA and PFOS.
(4) Develop "source type signatures" to differentiate multiple sources of contamination; and
(5) Develop risk based prioritization procedures for evaluating air emissions (both deposition and inhalation pathways) for contaminant(s) measured in the environment.

2.0 Approach to Work

The approach to completing the work is described in Tasks 1-4, below. The sampling locations associated with each task are shown in the Figures 3 – 5 of the attached SGPPL Stack Test Plan dated April 11, 2018.

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Task 1 – Air Emission Samples

SGPPL has contracted with Barr Engineering to conduct the stack testing that will collect the air emission samples. Barr Engineering will be using SGS Laboratories to prepare field reagents and perform analytical work for SGPPL. NHDES staff will be on site during the stack testing to observe the entire test program and SGPPL has agreed to collect additional samples[1] for submittal to ORD for non-routine analyses.

The combined program will involve the testing of one cast film and two fabric coating towers as listed below:

- MA Tower – Emission samples from this fabric coating tower will be collected at the uncontrolled exhaust prior to the dilution fan/exhaust stack.
- MS Tower – Emission samples from this fabric coating tower will be collected at the uncontrolled exhaust prior to the dilution fan/exhaust stack.
- QX Tower – Emission samples from this cast film tower will be equipped with a pilot-scale wet cyclone/fiberbed mist collection system (APCE). Emission samples will be collected simultaneously at the inlet and outlet locations of the APCE.

A modified method 5 (MM5) train will be used to collect PFAS compounds with nominal boiling points greater than 100°C. For the purpose of collecting samples for ORD, three 2-hour test runs[2] will be conducted for each tower location for a total of 12 sample sets. In addition, reagent blanks will be collected for each MM5 fraction and a field biased blank sampling train will be set up and recovered to assess any field contamination issues.

Each MM5 sample train consists of a nozzle, heated probe, heated glass fiber filter, XAD-2/Condenser Module, three Greenberg-Smith impingers [one containing 100 mL DI water, one containing 100 mL 0.1N sodium hydroxide (NaOH), and one containing 0.01N sodium borate], a second unheated filter followed by an indicating silica gel impinger for water vapor removal. Since each train has 6 fractions plus a methanol (MeOH) rinse fraction, there will be a total of 7 fractions per sampling train for a total of 98 samples to be analyzed separately.

Summa Canisters will be used to collect volatile compounds with boiling points less than 100°C. Canister samples will be collected during each of the 2-hour test runs summarized above. Sampling will be conducted in accordance with the methodology specified in EPA's *Compendium of Methods for the Determination of Toxic Organic Compound – TO14A and TO15*. Canister orifices will be adjusted to

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[1] Since the tower ductwork is anticipated to be less than 24 inches in diameter, concurrent sampling following standard EPA methodology is prohibitive. Therefore, a total of 6 test runs will be conducted for each tower sample location with every other test run sent to ORD and the remaining sent to the commercial lab (SGS Laboratory).

[2] Test runs will be two hours in duration unless labs state that longer timeframes are required for lower detection limits.

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collect integrated samples during the specified 2-hour test period. NHDES will conduct the sampling according to EPA's Miscellaneous Operating Procedure (MOP) that will be provided as part of the Summa Canister shipment. A total of 12 samples, 1 field blank and 3 ambient locations (TBD) will be collected.

The industry standard when conducting stack testing using sampling trains involving resins is for the test company to send the glass resin traps and glass fiber filters to the lab conducting the sample analyses. The lab cleans the glass traps according to their QC protocol, prepares and QCs the resin batch (in this case XAD-2), packs the resin traps, spikes the traps with surrogates to assess recovery and ships the sampling media to the field for the sampling team to use. Similarly, the laboratory may QC the filter media/reagents and/or pre-clean the filters and provide these reagents to the sampling team. This is important because the sampling company must choose glass traps that are compatible with their sampling equipment. Also, on past tests for PFAS, the lab provided the other reagents that were QC'd as is the case with the XAD. This included the DI water, 0.1N NaOH, MeOH, 0.01N sodium borate, pre-cleaned glass fiber filters and sample recovery bottles.

In this case, there are two labs conducting the post-testing analyses: ORD and SGS Laboratories. Therefore, it is imperative that coordination of the pre-test preparation work be facilitated in a way that all parties are aware of the details and schedule. Barr Engineering will provide XAD glassware to SGS Laboratories for cleaning, packing resin, and spiking and SGS will provide the reagents for samples that are QC'd. The field team will collect reagent blanks in the field and assemble and recover a field-biased blank train to account for any handling bias during one of the test runs.

ORD will provide a surrogate mixture to SGS and SGS will add this surrogate mixture as a field spike to the resin traps designated for the ORD sample sets. These traps will be spiked with the labeled compounds listed below:

Compound	**Abbreviation**
Perfluoro-n-[3,4,5-13C3]pentanoic acid	M3PFPeA
Perfluoro-n-[1,2-13C2]decanoic acid	MPFDA
Perfluoro-n-[1,2,3,4-13C4]octanoic acid	MPFOA
Sodium perfluoro-1-[1,2,3,4-13C4]octanesulfonate	MPFOS
2-Perfluorohexyl-[1,2-13C2]-ethanol (6:2)	M2FHET
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-13C3-propanoic acid	M3HFPO-DA

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Task 2 – Sample PFAS-Based Raw Materials

Samples of raw materials thought to contain PFAS compounds were collected at this facility as part of the initial ORD sampling project in the fall of 2017. Based on process information submitted by SGPPL to NHDES, the raw materials that are planned to be used at the time of the stack testing are the same as those already sent to ORD. The actual coating formulations used at the time of the stack test will be collected from each dip pan(s) on each tower during each test run and sent to ORD for analysis. At a minimum, 1 sample from the dip pan from the MA and MS Towers for each of the stack test runs will be collected, for a total of 6 samples from these two towers. At a minimum, 1 sample from each dip pan from the QX Tower for each of the stack test runs will be collected. Since the QX Tower will be operated using four passes, there will be 4 samples for each stack test of the QX Tower for a total of 12 samples. In total, ORD will receive 18 dip pan samples.

Task 3 – Sample Char/Carbon Material Taken from Air Emission Towers

Three samples of solid materials that accumulate on the interior of the towers were collected as part of the initial ORD sampling project in the fall of 2017. Three additional samples of this material will be collected during the stack testing program and are summarized as follows:

- One sample from the MA Tower which has been in operation at SGPPL since 1994. The ductwork was replaced and the oven and ancillary process components were cleaned in 2016. Therefore, the solid material that will be collected from this stack will most likely represent new dispersions used since 2016.

- One sample from the MS Tower which has been in operation at SGPPL since 2002. The solid material that will be collected from this stack may potentially contain residue components from pre-2006 PFOA based dispersions AND new dispersions that have been used since 2006.

- One sample from the QX Tower which has been in operation at SGPPL since 1989. This tower has the highest PFOA partition factor based on previous stack test results and therefore receives the highest load of emissions. The solid material coating the interior of the tower would likely be associated with emissions that occurred while PFOA-based dispersions were in use and emissions that occurred after the use of PFOA had been phased out.

Task 4 – Sampling of the Pilot-Scale Air Pollution Control Equipment (APCE)

SGPPL is planning on conducting a pilot-scale evaluation of a candidate air pollution control technology on the QX Tower in addition to collecting samples for ORD. Inlet and outlet samples will be collected as described under Task 1 above.

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The candidate APCE is a wet cyclone/fiberbed mist collection system. Gas enters the unit and passes through a wet cyclone where larger particles are removed. The material that is removed falls into the cyclone sump and the water in the sump is recirculated through the cyclone. In a full scale system, the sump has a makeup water feed and a slow solids removal cycle for higher solids applications. In the case of water soluble PFAS, the situation exists where the PFAS concentrations in the water could exceed the solubility of the sump liquid if the makeup water input is not sufficient.

The filter system is a spun fiber type (depth filter type) and would not normally have a pulse system to clean the surface of collected particulate. Any particulate that is not embedded in the filter structure drops to the hopper and the fine particles enter the depths of the filter. At some point, the filter plugs and must be changed.

In conjunction with the APCE evaluation and in addition to the air samples, NHDES will collect samples of the make-up water (from plant water source – 1 sample), sump water (for each of the three APCE test runs on QX Tower – 3 samples) and any solid matter that can be removed from the internals of the unit after the test is done and the unit is taken offline (e.g. sump solids, particulate filter material – 2 samples).

The following types and quantities of samples will be collected for each project task:

	Air Emission Samples	Summa Canister Samples	Dip Tank Coatings	Char Material from Tower Stacks	Process Water and Solids from APCE
Task 1	**98**	**16**			
Task 2			**18**		
Task 3				**3**	
Task 4					**6**
Total	**98**	**16**	**18**	**3**	**6**
	Total Number of Analytical Fractions				**141**

3.0 Sampling Procedures and Quality Assurance Project Plan

NHDES' Quality Assurance Project Plan (QAPP) was included in the initial ORD request package. The procedures for sampling for PFAS in the QAPP begin on document page 246.

4.0 Schedule

The sample collection described in this work plan shall be conducted late April and early May, 2018. Sampling for Tasks 1-4 will occur concurrently.

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