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1. INTRODUCTION

1.1 FACILITY AND BACKGROUND INFORMATION

The Chemours Fayetteville Works (Chemours) is located in Bladen County, North Carolina, approximately 10 miles south of the city of Fayetteville. The Chemours operating areas on the site include the Fluoromonomers, IXM and Polymer Processing Aid (PPA) manufacturing areas, Wastewater Treatment, and Powerhouse.

Chemours contracted Weston Solutions, Inc. (Weston) to perform HFPO Dimer Acid emission testing on the PPA Stack. Testing was performed on 7-8 January 2019 and generally followed the "Emissions Test Protocol" reviewed and approved by the North Carolina Department of Environmental Quality (NCDEQ). This report provides the results from the emission test program.

1.2 TEST OBJECTIVES

The specific objectives for this test program were as follows:

- Measure the emissions concentrations and mass emissions rates of HFPO Dimer Acid from the PPA process stack which is located in the PPA process area.
- Monitor and record process data in conjunction with the test program.
- Provide representative emissions data.

1.3 TEST PROGRAM OVERVIEW

During the emissions test program, the concentrations and mass emissions rates of HFPO Dimer Acid were measured on the PPA process stack source.

Table 1-1 provides a summary of the test locations and the parameters that were measured along with the sampling/analytical procedures that were followed.

Section 2 provides a summary of test results. A description of the processes is provided in Section 3. Section 4 provides a description of the test locations. The sampling and analytical procedures are provided in Section 5. Detailed test results and discussion are provided in Section 6.

Appendix C includes the summary reports for the laboratory analytical results. The full laboratory data packages are provided in electronic format and on CD with each hard copy.

Table 1-1
Sampling Plan for PPA Process Stack

Sampling Point & Location	**PPA Process Stack**				
Number of Tests:	3				
Parameters To Be Tested:	HFPO Dimer Acid (HFPO-DA)	Volumetric Flow Rate and Gas Velocity	Carbon Dioxide	Oxygen	Water Content
Sampling or Monitoring Method	EPA M-0010	EPA M1, M2, M3A, and M4 in conjunction with M-0010 tests	EPA M3A		EPA M4 in conjunction with M-0010 tests
Sample Extraction/ Analysis Method(s):	LC/MS/MS	NA[6]	NA		NA
Sample Size	> 1m^3	NA	NA	NA	NA
Total Number of Samples Collected[1]	3	3	3	3	3
Reagent Blanks (Solvents, Resins)[1]	1 set	0	0	0	0
Field Blank Trains[1]	1 per source	0	0	0	0
Proof Blanks[1]	1 per train	0	0	0	0
Trip Blanks[1,2]	1 set	0	0	0	
Lab Blanks	1 per fraction[3]	0	0	0	0
Laboratory or Batch Control Spike Samples (LCS)	1 per fraction[3]	0	0	0	0
Laboratory or Batch Control Spike Sample Duplicate (LCSD)	1 per fraction[3]	0	0	0	0
Media Blanks	1 set[4]	0	0	0	0
Isotope Dilution Internal Standard Spikes	Each sample	0	0	0	0
Total No. of Samples	7[5]	3	3	3	3

Key:

[1] Sample collected in field.

[2] Trip blanks include one XAD-2 resin module and one methanol sample per sample shipment.

[3] Lab blank and LCS/LCSD includes one set per analytical fraction (front half, back half and condensate).

[4] One set of media blank archived at laboratory at media preparation.

[5] Actual number of samples collected in field.

[6] Not applicable.

2. SUMMARY OF TEST RESULTS

A total of three test runs were performed on the PPA process stack. Table 2-1 provides a summary of the HFPO Dimer Acid emission test results. Detailed test results summaries are provided in Section 6.

It is important to note that emphasis is being placed on the characterization of the emissions based on the stack test results. Research conducted in developing the protocol for stack testing HFPO Dimer Acid Fluoride, HFPO Dimer Acid Ammonium Salt and HFPO Dimer Acid realized that the resulting testing, including collection of the air samples and extraction of the various fraction of the sampling train, would result in all three compounds being expressed as simply the HFPO Dimer Acid. However, it should be understood that the total HFPO Dimer Acid results provided on Table 2-1 and in this report include a percentage of each of the three compounds.

Table 2-1

Summary of HFPO Dimer Acid Test Results

Source	Run No.	Emission Rates	
		lb/hr	g/sec
PPA Process Stack	1	1.32E-04	1.66E-05
	2	1.40E-04	1.76E-05
	3	1.14E-04	1.44E-05
	Average	1.29E-04	1.62E-05

3. PROCESS DESCRIPTIONS

The PPA area is included in the scope of this test program.

3.1 POLYMER PROCESSING AID (PPA) AREA

The PPA facility produces surfactants used to produce fluoropolymer products at other Chemours facilities, such as Teflon®, as well as sales to outside producers of fluoropolymers.

Process streams are vented to a caustic wet scrubber (ACD-A1), carbon bed and vented to a process stack (AEP-A1). The process inside the building is under negative pressure and the building air is vented to the process stack (AEP-A1) and the carbon bed.

3.2 PROCESS OPERATIONS AND PARAMETERS

Source	Operation/Product	Batch or Continuous
PPA	AF Column Reboiler/Virgin	Continuous once it starts taking off to feed tank
	Pressure Transfers/Virgin or Purified	Batch (pressure transfers from one vessel to another – every 2 hours)

During the test program, the following parameters were monitored by Chemours and are included in Appendix A.

- PPA Process
  - Caustic Wet Scrubber (ACD-A1)
    - Caustic recirculation flow rate
    - Differential pressure across the packing

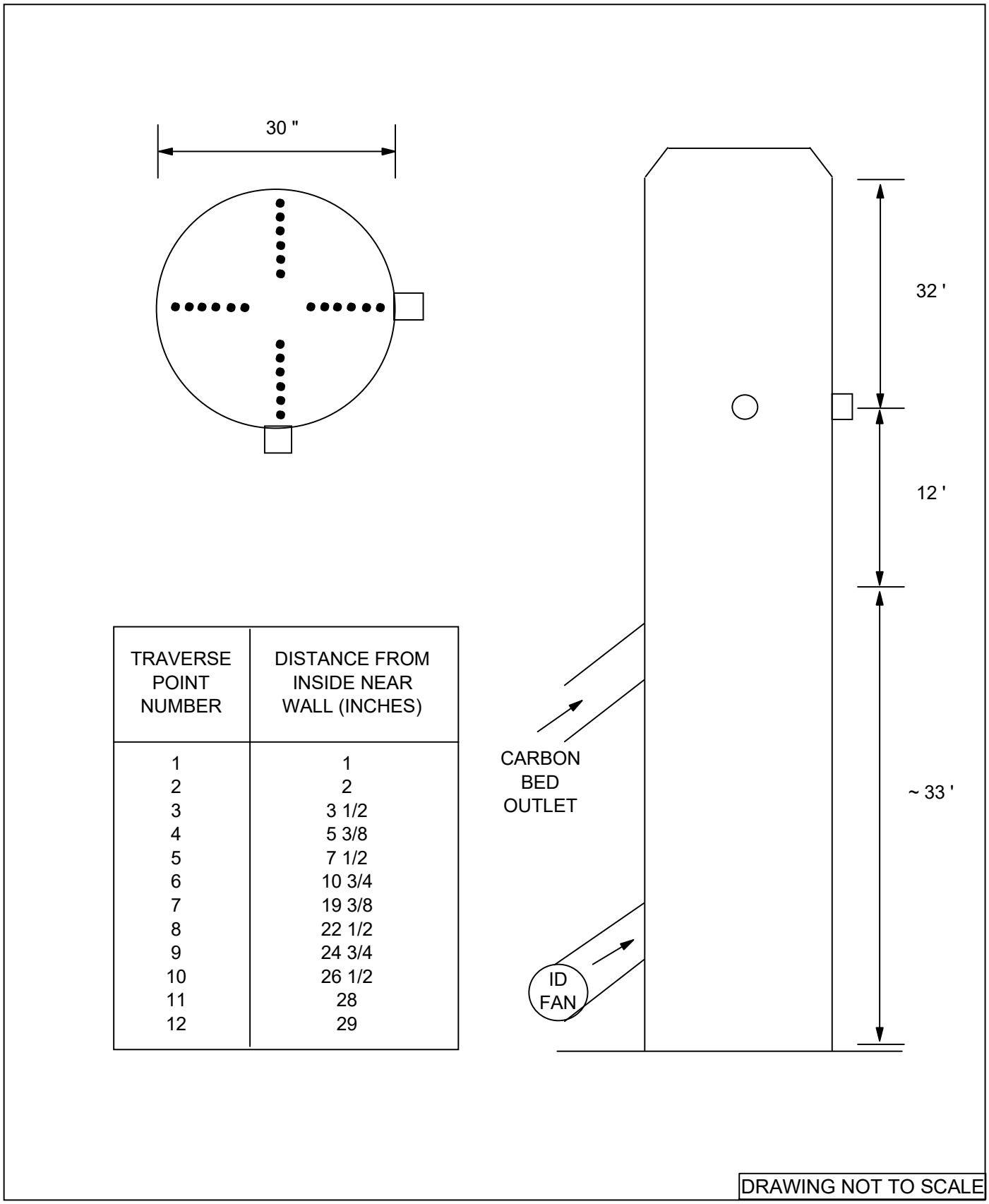
4. DESCRIPTION OF TEST LOCATIONS

4.1 PPA PROCESS STACK

Two 4-inch ID test ports are in place on the 30-inch ID fiberglass stack. The ports are 12 feet (4.8 diameters) from the nearest downstream disturbance (carbon bed outlet) and 32 feet (12.8 diameters) from the nearest upstream disturbance (stack exit).

Per EPA Method 1, a total of 24 traverse points (12 per axis) were used for M-0010 isokinetic sampling. See Figure 4-1 for a schematic of the test port and traverse point locations.

Note: All measurements at the test location were confirmed prior to sampling.



TRAVERSE POINT NUMBER	DISTANCE FROM INSIDE NEAR WALL (INCHES)
1	1
2	2
3	3 1/2
4	5 3/8
5	7 1/2
6	10 3/4
7	19 3/8
8	22 1/2
9	24 3/4
10	26 1/2
11	28
12	29

FIGURE 4-1
PPA PROCESS STACK TEST PORT
AND TRAVERSE POINT LOCATION

IASDATA\CHEMOURS\15418.002.009\FIGURE 4-1 PPA EXHAUST STACK

5. SAMPLING AND ANALYTICAL METHODS

5.1 STACK GAS SAMPLING PROCEDURES

The purpose of this section is to describe the stack gas emissions sampling train and to provide details of the stack sampling and analytical procedures utilized during the emissions test program.

5.1.1 Pre-Test Determinations

Preliminary test data was obtained at the test location. Stack geometry measurements were measured and recorded, and traverse point distances verified. A preliminary velocity traverse was performed utilizing a calibrated S-type pitot tube and an inclined manometer to determine velocity profiles. Flue gas temperatures were observed with a calibrated direct readout panel meter equipped with a chromel-alumel thermocouple. Preliminary water vapor content was estimated by wet bulb/dry bulb temperature measurements.

A check for the presence or absence of cyclonic flow was previously conducted at the test location. The cyclonic flow check was negative (< 20°) verifying that the source were acceptable for testing.

Preliminary test data was used for nozzle sizing and sampling rate determinations for isokinetic sampling procedures.

Calibration of probe nozzles, pitot tubes, metering systems, and temperature measurement devices was performed as specified in Section 5 of EPA Method 5 test procedures.

5.2 STACK PARAMETERS

5.2.1 EPA Method 0010

The sampling train utilized to perform the HFPO Dimer Acid sampling was an EPA Method 0010 train (see Figure 5-1). The Method 0010 consisted of a borosilicate nozzle that attached directly to a heated borosilicate probe. In order to minimize possible thermal degradation of the HFPO Dimer Acid, the probe and particulate filter were heated above stack temperature to

minimize water vapor condensation before the filter. The probe was connected directly to a heated borosilicate filter holder containing a solvent extracted glass fiber filter.

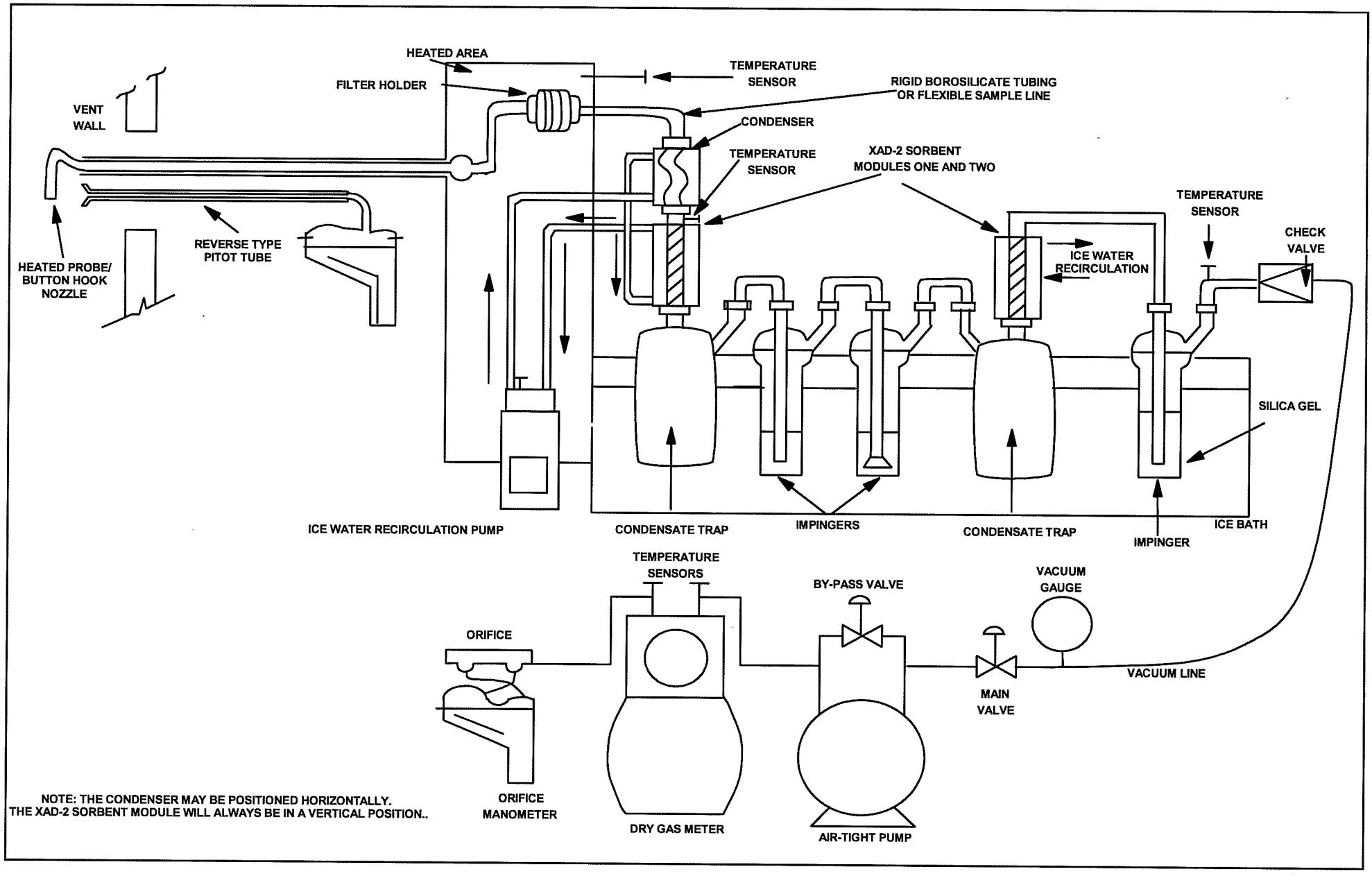


FIGURE 5-1
EPA METHOD 0010 SAMPLING TRAIN

IASDATA\CHEMOURS\15418.002.009\FIGURE 5-1 METHOD 0010

A section of borosilicate glass (or flexible polyethylene) tubing connected the filter holder exit to a Grahm (spiral) type ice water-cooled condenser, an ice water-jacketed sorbent module containing approximately 40 grams of XAD-2 resin. The XAD-2 resin tube was equipped with an inlet temperature sensor. The XAD-2 resin trap was followed by a condensate knockout impinger and a series of two impingers that contained 100 milliliters of high purity distilled water. The train also included a second XAD-2 resin trap behind the impinger section to evaluate possible sampling train breakthrough. Each XAD-2 resin trap was connected to a 1-liter condensate knockout trap. The final impinger contained 300 grams of dry pre-weighed silica gel. All impingers and the condensate traps were maintained in an ice bath. Ice water was continuously circulated in the condenser and both XAD-2 modules to maintain method required temperature. A control console with a leakless vacuum pump, a calibrated orifice, and dual inclined manometers was connected to the final impinger via an umbilical cord to complete the sample train.

HFPO Dimer Acid Fluoride (CAS No. 2062-98-8) that is present in the stack gas is expected to be captured in the sampling train along with HFPO Dimer Acid (CAS No. 13252-13-6). HFPO Dimer Acid Fluoride undergoes hydrolysis instantaneously in water in the sampling train and during the sample recovery step and will be converted to HFPO Dimer Acid such that the amount of HFPO Dimer Acid emissions represents a combination of both HFPO Dimer Acid Fluoride and HFPO Dimer Acid.

During sampling, gas stream velocities were measured by attaching a calibrated S-type pitot tube into the gas stream adjacent to the sampling nozzle. The velocity pressure differential was observed immediately after positioning the nozzle at each traverse point, and the sampling rate adjusted to maintain isokineticity at 100% ± 10. Flue gas temperature was monitored at each point with a calibrated panel meter and thermocouple. Isokinetic test data was recorded at each traverse point during all test periods, as appropriate. Leak checks were performed on the sampling apparatus according to reference method instructions, prior to and following each run, component change (if required), or during midpoint port changes.

5.2.2 EPA Method 0010 Sample Recovery

At the conclusion of each test, the sampling train was dismantled, the openings sealed, and the components transported to the field laboratory trailer for recovery.

A consistent procedure was employed for sample recovery:

1. The two XAD-2 covered (to minimize light degradation) sorbent modules (1 and 2) were sealed and labeled.

2. The glass fiber filter(s) were removed from the holder with tweezers and placed in a polyethylene container along with any loose particulate and filter fragments.

3. The particulate adhering to the internal surfaces of the nozzle, probe and front half of the filter holder were rinsed with a solution of methanol and ammonium hydroxide into a polyethylene container while brushing a minimum of three times until no visible particulate remained. Particulate adhering to the brush was rinsed with methanol/ ammonium hydroxide into the same container. The container was sealed.

4. The volume of liquid collected in the first condensate trap was measured, the value recorded, and the contents poured into a polyethylene container.

5. All train components between the filter exit and the first condensate trap were rinsed with methanol/ammonium hydroxide. The solvent rinse was placed in a separate polyethylene container and sealed.

6. The volume of liquid in impingers one and two, and the second condensate trap, were measured, the values recorded, and the sample was placed in the same container as Step 4 above, then sealed.

7. The two impingers, condensate trap, and connectors were rinsed with methanol/ ammonium hydroxide. The solvent sample was placed in a separate polyethylene container and sealed.

8. The silica gel in the final impinger was weighed and the weight gain value recorded.

9. Site (reagent) blank samples of the methanol/ammonium hydroxide, XAD resin, filter and distilled water were retained for analysis.

Each container was labeled to clearly identify its contents. The height of the fluid level was marked on the container of each liquid sample to provide a reference point for a leakage check during transport. All samples were maintained cool.

During each test campaign, an M-0010 blank train was set up near the test location, leak checked and recovered along with the respective sample train. Following sample recovery, all samples were transported to TestAmerica Laboratories, Inc. (TestAmerica) for sample extraction and analysis.

See Figure 5-2 for a schematic of the M-0010 sample recovery process.

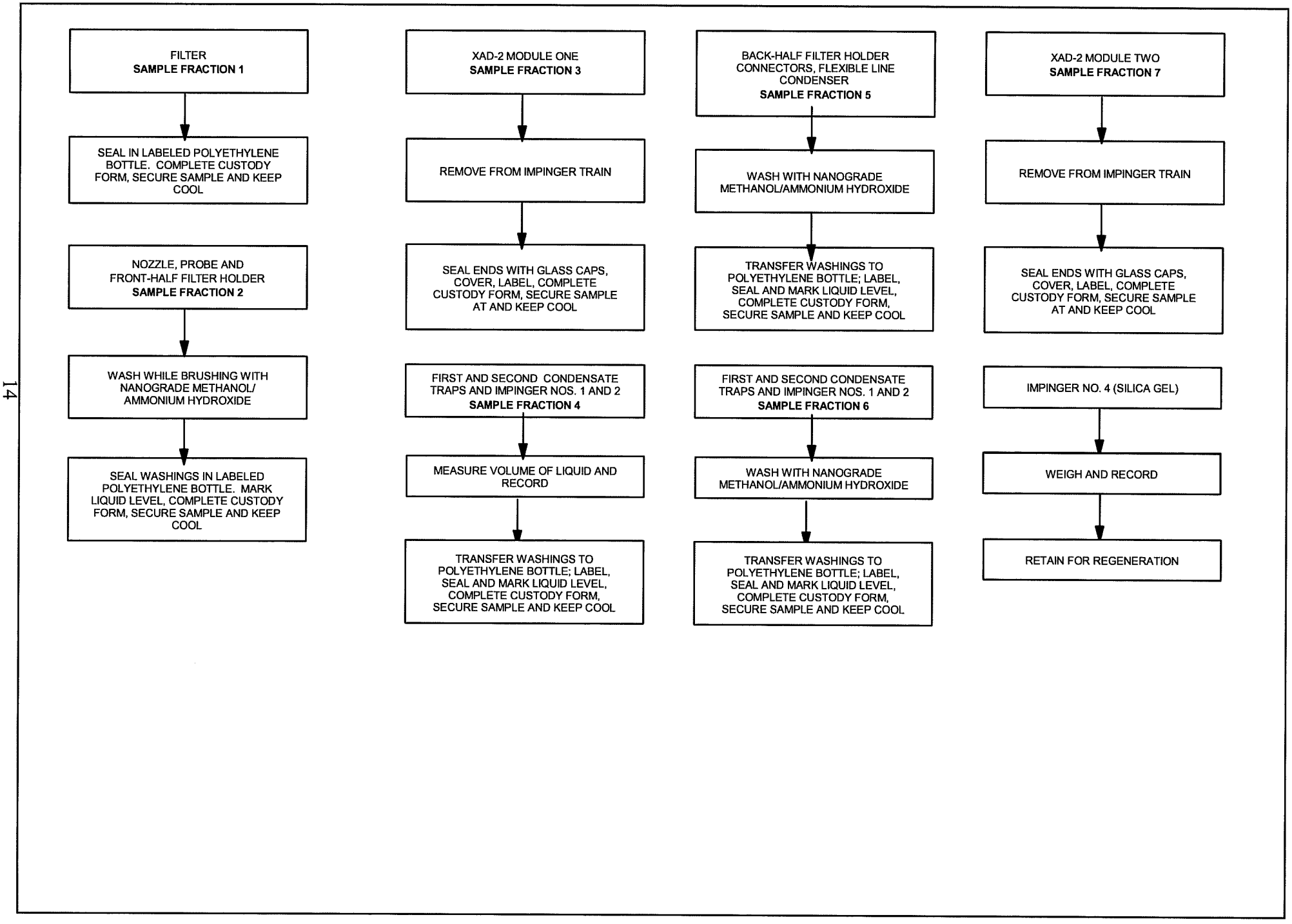


FIGURE 5-2
HFPO DIMER ACID SAMPLE RECOVERY PROCEDURES FOR METHOD 0010

IASDATA\CHEMOURS\15418.002.009\FIGURE 5-2 EPA 0010 PG 14

5.2.3 EPA Method 0010 – Sample Analysis

The Method 0010 sampling trains resulted in four separate analytical fractions for HFPO Dimer Acid analysis according to SW-846 Method 3542:

- Front-Half Composite—comprised of the particulate filter, and the probe, nozzle, and front-half of the filter holder solvent rinses;
- Back-Half Composite—comprised of the first XAD-2 resin material and the back-half of the filter holder with connecting glassware solvent rinses;
- Condensate Composite—comprised of the aqueous condensates and the contents of impingers one and two with solvent rinses;
- Breakthrough XAD-2 Resin Tube—comprised of the resin tube behind the series of impingers.

The second XAD-2 resin material was analyzed separately to evaluate any possible sampling train HFPO-DA breakthrough.

The front-half and back-half composites and the second XAD-2 resin material were placed in polypropylene wide-mouth bottles and tumbled with methanol containing 5% NH_4OH for 18 hours. Portions of the extracts were processed analytically for the HFPO dimer acid by liquid chromatography and duel mass spectroscopy (HPLC/MS/MS). The condensate composite was concentrated onto a solid phase extraction (SPE) cartridge followed by desorption from the cartridge using methanol. Portions of those extracts were also processed analytically by HPLC/MS/MS.

Samples were spiked with isotope dilution internal standard (IDA) at the commencement of their preparation to provide accurate assessments of the analytical recoveries. Final data was corrected for IDA standard recoveries.

TestAmerica developed detailed procedures for the sample extraction and analysis for HFPO Dimer Acid. These procedures were incorporated into the test protocol.

The following Weston employees participated in this project.

Paul Meeter	Senior Project Manager
Jacob Little	Team Member
Kyle Schweitzer	Team Member
Austin Squires	Team Member
Chris Hartsky	Team Member