

**SEMI-WORKS MANUFACTURING PROCESS
EMISSIONS TEST REPORT
TEST DATES: 10-11 JANUARY 2019**

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FAYETTEVILLE, NORTH CAROLINA**

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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 Polymers Processing Aid (PPA) manufacturing areas, Wastewater Treatment, and Powerhouse.

Chemours contracted Weston Solutions, Inc. (Weston) to perform HFPO Dimer Acid Fluoride, captured as HFPO Dimer Acid, emission testing on the Semi-Works stack. Testing was performed on 10-11 January 2019 and generally followed the “Emission 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 Fluoride from the Semi-Works stack.
- 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 Fluoride was measured on the Semi-Works stack.

Table 1-1 provides a summary of the test location 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 location. The sampling and analytical procedures are provided in Section 5. Detailed test results and discussion are provided in Section 6.

Appendix B 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 Semi-Works Stack**

Sampling Point & Location	Semi-Works Stack				
Number of Tests:	2				
Parameters To Be Tested:	HFPO Dimer Acid Fluoride (HFPO-DAF)	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	2	2	2	2	2
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	6 ⁵	2	2	2	2

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 two tests were performed on the Semi-Works 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
Semi-Works	1	1.00E-03	1.26E-04
	2	6.19E-04	7.79E-05
	Average	8.10E-04	1.02E-04

3. PROCESS DESCRIPTIONS

The Semi-Works area is included in the scope of this test program.

3.1 SEMI-WORKS AREA

Semi-Works is generally a Research and Development facility. However, there are two products made in this unit on a periodic basis: Dimer Peroxide and a high Equivalent Weight (EW) polymer. The Dimer Peroxide is then used in the IXM Polymers manufacturing area and the high EW polymer is used in the IXM Products area to make a specific membrane product.

The following process streams vent to the Semi-Works building stack:

- Continuous Polymerization Process – when making high EW polymer
- Batch Polymerization – when making the Dimer Peroxide

3.2 PROCESS OPERATIONS AND PARAMETERS

Source	Operation/Product	Batch or Continuous
Semi-Works	Dimer Peroxide	Batch

There are no parameters to monitor from Semi-Works, as there is no control device associated with this stack.

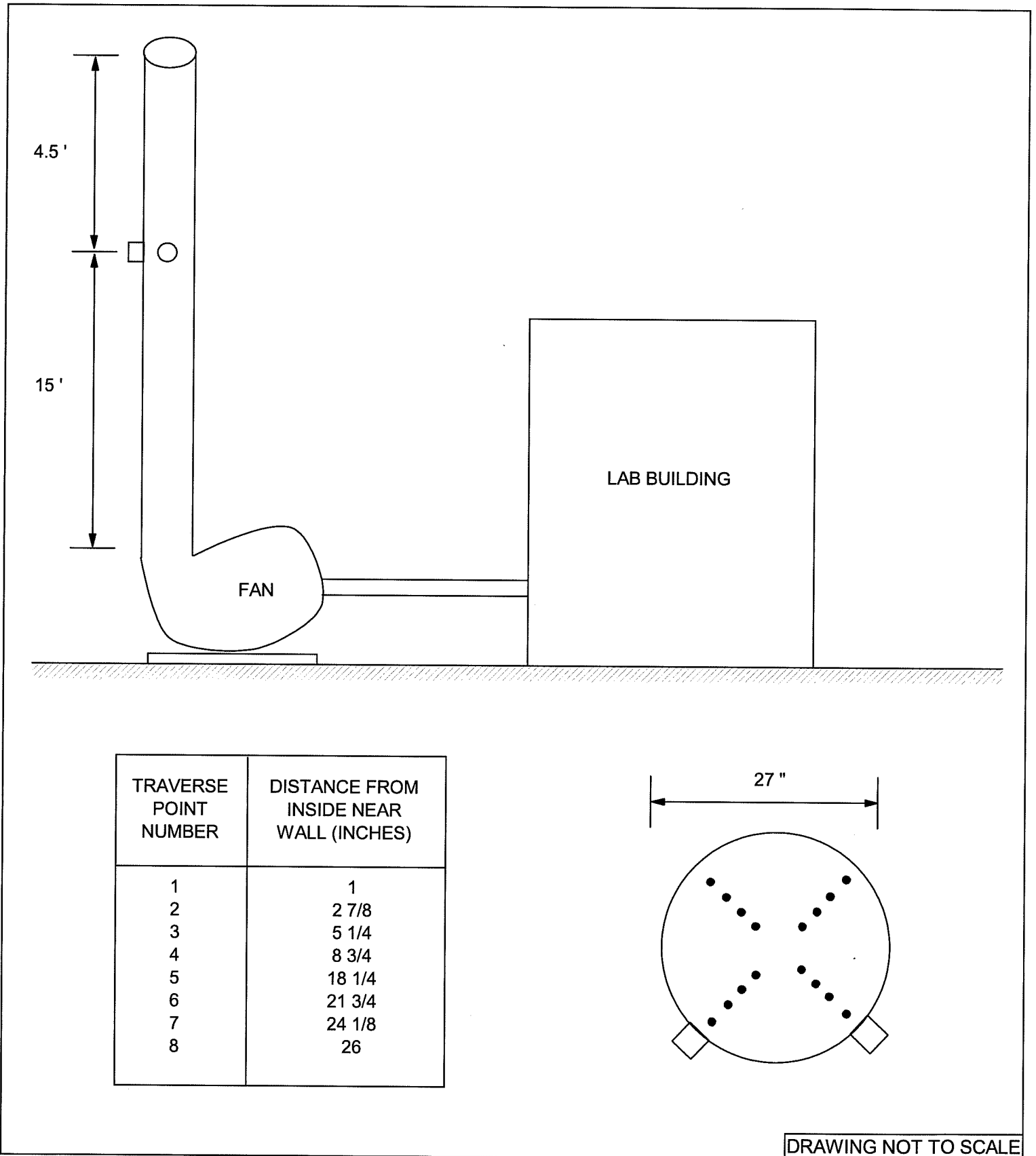
4. DESCRIPTION OF TEST LOCATIONS

4.1 SEMI-WORKS STACK

The Semi-Works stack is a circular steel stack outside the laboratory building. The ID fan is located at ground level. The stack ID is 27 inches. Two sample ports, 90° apart are installed 4.5 feet down from the top of the stack and 15 feet up from the ID fan discharge. Per EPA Method 1, sixteen traverse points, eight per port, were used for sampling.

Figure 4-1 provides a schematic of the test port and traverse point locations.

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



**FIGURE 4-1
SEMI-WORKS STACK
TEST PORT AND TRAVERSE POINT LOCATION**

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 ($< 20^\circ$) verifying that the source was 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.

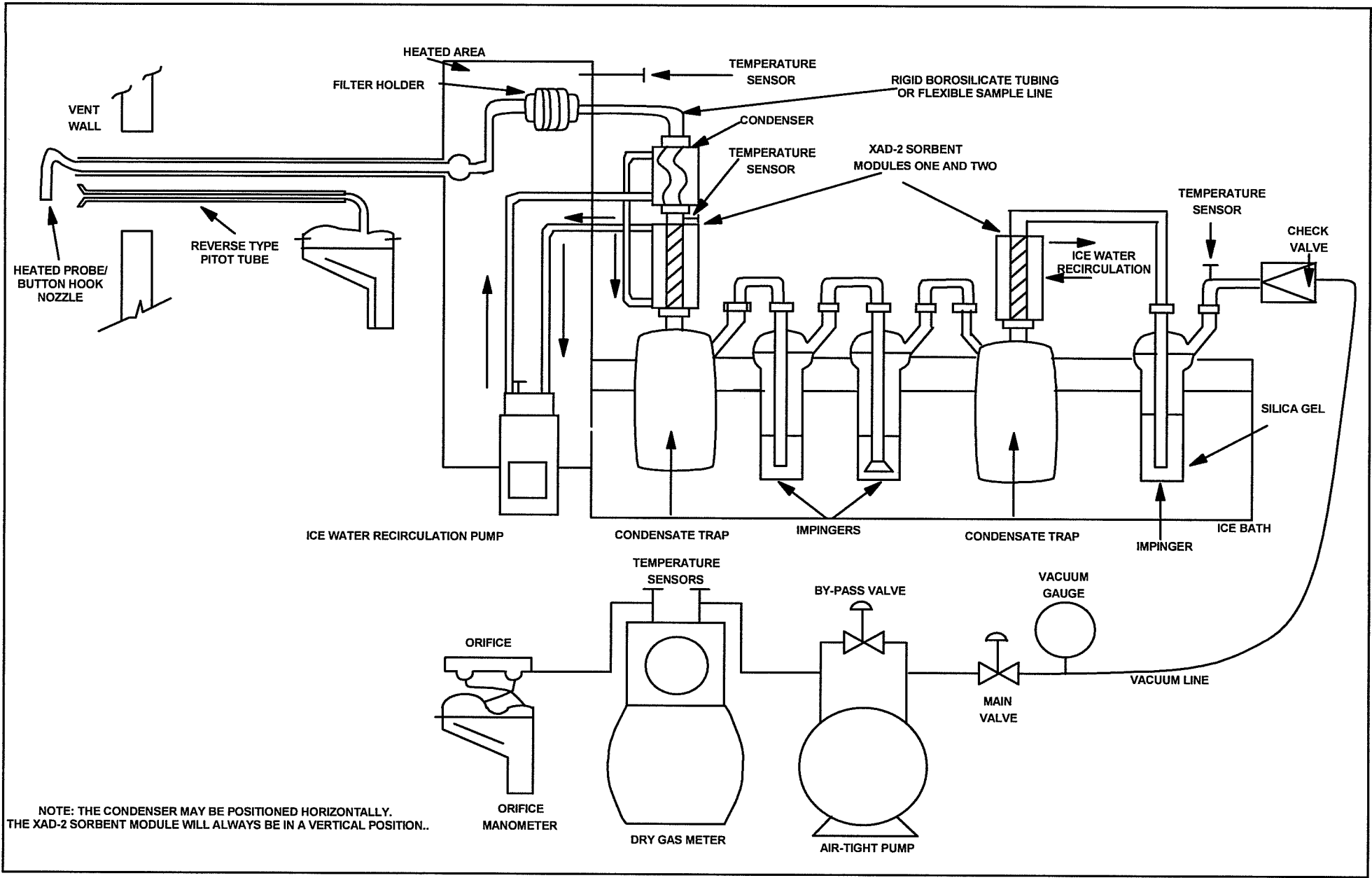


FIGURE 5-1
EPA METHOD 0010 SAMPLING TRAIN

A section of borosilicate glass or flexible polyethylene tubing connected the filter holder exit to a Graham (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 each 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 $100\% \pm 10$. 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 the test campaign, an M-0010 blank train was set up near the test location, leak checked and recovered along with the 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.

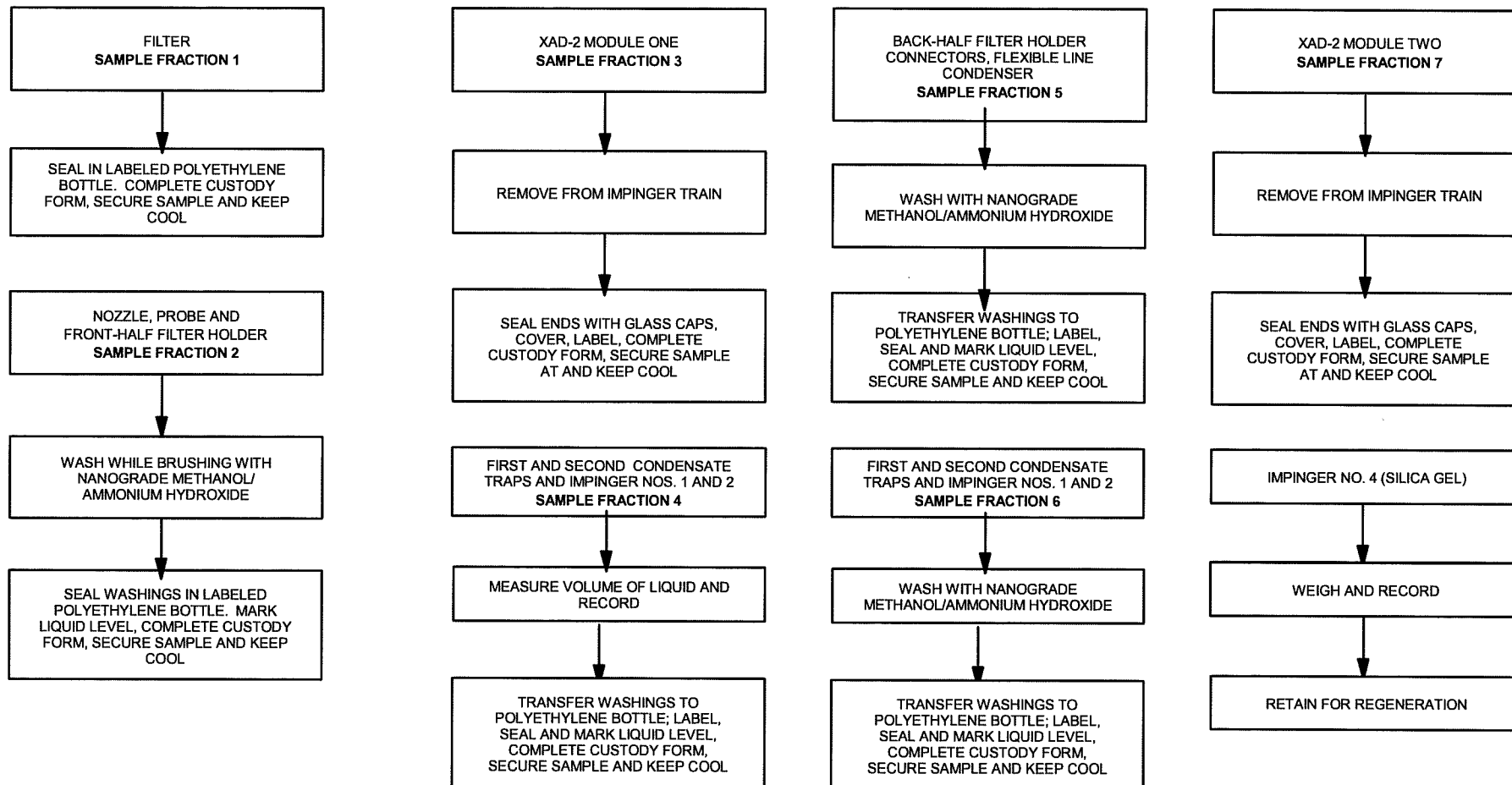


FIGURE 5-2
HFPO DIMER ACID SAMPLE RECOVERY PROCEDURES FOR METHOD 0010

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% NH₄OH for 18 hours. Portions of the extracts were processed analytically for the HFPO dimer acid by liquid chromatography and dual 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.

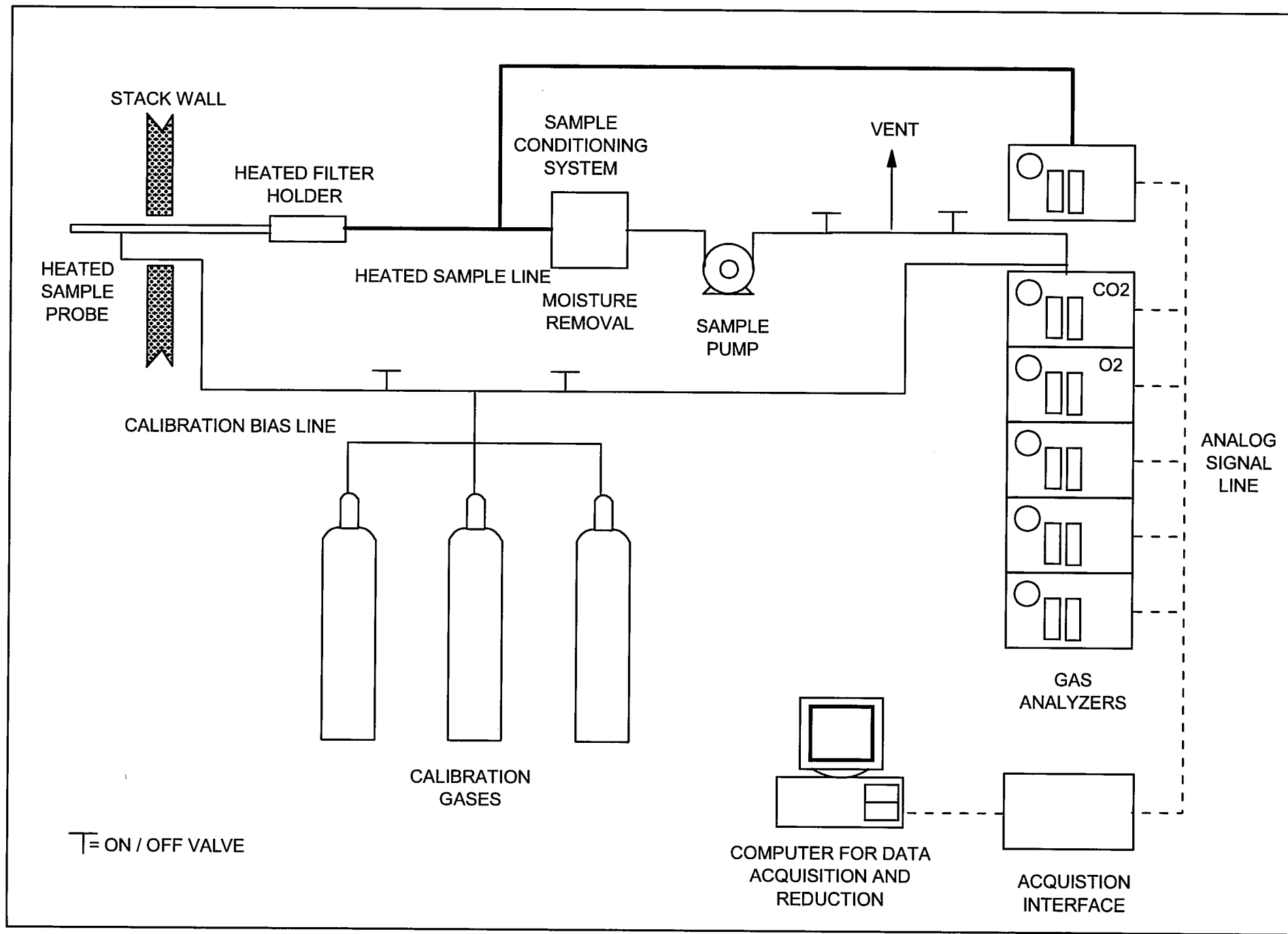
5.3 GAS COMPOSITION

The Weston mobile laboratory equipped with instrumental analyzers was used to measure carbon dioxide (CO₂) and oxygen (O₂) concentrations. A diagram of the Weston sampling system is presented in Figure 5-3.

Each analyzer was set up and calibrated internally by introduction of calibration gas standards directly to the analyzer from a calibration manifold. The calibration manifold is designed with an atmospheric vent to release excess calibration gas and maintains the calibration at ambient pressure. The direct calibration sequence consisted of alternate injections of zero and mid-range gases with appropriate adjustments until the desired responses were obtained. The high-range standards were then introduced in sequence without further adjustment.

The sample line integrity was verified by performing a bias test before and after each test period. The sampling system bias test consisted of introducing the zero gas and one up-range calibration standard in excess to the valve at the probe end when the system was sampling normally. The excess calibration gas flowed out through the probe to maintain ambient sampling system pressure. Calibration gas supply was regulated to maintain constant sampling rate and pressure. Instrument bias check response was compared to internal calibration responses to ensure sample line integrity and to calculate a bias correction factor after each run using the ratio of the measured concentration of the bias gas certified by the calibration gas supplier.

The oxygen and carbon dioxide content of each stack gas was measured according to EPA Method 3A procedures which incorporate the latest updates of EPA Method 7E. A Servomex Model 4900 analyzer (or equivalent) was used to measure oxygen content. A Servomex Model 4900 analyzer (or equivalent) was used to measure carbon dioxide content of the stack gas. Both analyzers were calibrated with EPA Protocol gases prior to the start of the test program and performance was verified by sample bias checks before and after each test run.



**FIGURE 5-3
WESTON SAMPLING SYSTEM**

6. DETAILED TEST RESULTS AND DISCUSSION

Preliminary testing and the associated analytical results required significant sample dilution to bring the HFPO Dimer Acid concentration within instrument calibration, therefore, sample times and sample volumes were reduced for the formal test program. This was approved by the North Carolina Department of Environmental Quality (NCDEQ).

Each test was 96 minutes in duration. A total of two tests were performed on the Semi-Works Stack.

Table 6-1 provides detailed test data and test results for the Semi-Works stack.

The Method 3A sampling on all sources indicated that the O₂ and CO₂ concentrations were at ambient air levels (20.9% O₂, 0% CO₂), therefore, 20.9% O₂ and 0% CO₂ values were used in all calculations.

TABLE 6-1
CHEMOURS - FAYETTEVILLE, NC
SUMMARY OF HFPO DIMER ACID TEST DATA AND TEST RESULTS

Test Data

	1	2
Run number		
Location	Semi-Works	Semi-Works
Date	1/10/2019	1/11/2019
Time period	1310-1458	0851-1049

SAMPLING DATA:

Sampling duration, min.	96.0	96.0
Nozzle diameter, in.	0.235	0.235
Cross sectional nozzle area, sq.ft.	0.000301	0.000301
Barometric pressure, in. Hg	29.93	30.24
Avg. orifice press. diff., in H ₂ O	1.37	1.34
Avg. dry gas meter temp., deg F	60.5	52.9
Avg. abs. dry gas meter temp., deg. R	521	513
Total liquid collected by train, ml	36.8	25.8
Std. vol. of H ₂ O vapor coll., cu.ft.	1.7	1.2
Dry gas meter calibration factor	0.9915	0.9915
Sample vol. at meter cond., dcf	61.011	58.493
Sample vol. at std. cond., dscf ⁽¹⁾	61.563	60.510
Percent of isokinetic sampling	104.4	100.9

GAS STREAM COMPOSITION DATA:

CO ₂ , % by volume, dry basis	0.0	0.0
O ₂ , % by volume, dry basis	20.9	20.9
N ₂ , % by volume, dry basis	79.1	79.1
Molecular wt. of dry gas, lb/lb mole	28.84	28.84
H ₂ O vapor in gas stream, prop. by vol.	0.027	0.020
Mole fraction of dry gas	0.973	0.980
Molecular wt. of wet gas, lb/lb mole	28.54	28.62

GAS STREAM VELOCITY AND VOLUMETRIC FLOW DATA:

Static pressure, in. H ₂ O	-0.21	-0.22
Absolute pressure, in. Hg	29.91	30.22
Avg. temperature, deg. F	61	54
Avg. absolute temperature, deg.R	521	514
Pitot tube coefficient	0.84	0.84
Total number of traverse points	16	16
Avg. gas stream velocity, ft./sec.	34.5	34.0
Stack/duct cross sectional area, sq.ft.	3.98	3.98
Avg. gas stream volumetric flow, wacf/min.	8229	8117
Avg. gas stream volumetric flow, dscf/min.	8108	8252

⁽¹⁾ Standard conditions = 68 deg. F. (20 deg. C.) and 29.92 in Hg (760 mm Hg)

**TABLE 6-1(cont.)
CHEMOURS - FAYETTEVILLE, NC
SUMMARY OF HFPO DIMER ACID TEST DATA AND TEST RESULTS**

TEST DATA

	1	2
Run number	Semi-Works	Semi-Works
Location	Semi-Works	Semi-Works
Date	1/10/2019	1/11/2019
Time period	1310-1458	0851-1049

LABORATORY REPORT DATA, ug.

HFPO Dimer Acid	57.6084	34.2887
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EMISSION RESULTS, ug/dscm.

HFPO Dimer Acid	33.0	20.0
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EMISSION RESULTS, lb/dscf.

HFPO Dimer Acid	2.06E-09	1.25E-09
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EMISSION RESULTS, lb/hr.

HFPO Dimer Acid	1.00E-03	6.19E-04
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EMISSION RESULTS, g/sec.

HFPO Dimer Acid	1.26E-04	7.79E-05
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The following Weston employees participated in this project.

Paul Meeter	Senior Project Manager
Jeff O'Neill	Technical Manager
Steve Rathfon	Team Leader
Kyle Schweitzer	Team Member
Jack Mills	Team Member
Chad Walker	Team Member