# 1.0 EXECUTIVE SUMMARY

Mostardi Platt conducted a Title V compliance test program for Billerud Quinnesec LLC at the Quinnesec Mill on the Smelt Dissolving Tank Outlet on November 8 and 9 and December 14, 2022. This report summarizes the results of the test program and test methods used.

Test location, test dates, and test parameters are summarized below.

TEST INFORMATION					
Test Location Test Dates Test Parameters					
Smelt Dissolving Tank Stack	November 8 and 9, 2022	Total Particulate Matter (TPM), Volatile Organic Compounds (VOC) and Total Reduced Sulfur (TRS)			
	December 14, 2022	Sulfur Dioxide (SO <sub>2</sub> )			

The purpose of the test program was to demonstrate compliance of the above emissions with the permitted limits. Selected results of the test program are summarized below. A complete summary of emission test results follows the narrative portion of this report.

		TEST RESULTS		
Test Location	Test Date	Test Parameter	Emission Limit	Emission Rate
	11/8/2022	TPM (PM <sub>10</sub> )	0.107 lb/TBLS 8.5 lb/hr	0.057 TBLS and 5.197 lb/hr
	11/8/2022	11/8/2022 Filterable Particulate Matter		0.050 TBLS and 4.556 lb/hr
Smelt Dissolving Tank	11/9/2022	VOC	As CH₄ 200 ppmvd 6.5 lb/hr	18.1 ppmvd CH₄ and 1.07 lb/hr
	11/8/2022 & 11/9/2022	TRS	As H <sub>2</sub> S 0.033 lb/TBLS 0.0168 lb/TBLS	0.0050 lb/TBLS
	12/14/2022	SO <sub>2</sub>	0.016 lb/TBLS 1.27 lb/hr	0.0019 lb/TBLS and 0.17 lb/hr

Emissions on Ib/TBLS basis were calculated with Black Liquor Solids (BLS) supplied by Billerud Quinnesec LLC. Plant operating data as provided by Billerud Quinnesec LLC is included in Appendix A.

The identifications of individuals associated with the test program are summarized below.

TEST PERSONNEL INFORMATION				
Location	Address	Contact		
Test Facility	Billerud Quinnesec LLC U.S. Highway 2 Quinnesec, Michigan 49876	Ms. Paula Lafleur Environmental Engineer (906) 779-3494 (phone)		
Testing Company Representative	Mostardi Platt 888 Industrial Drive Elmhurst, Illinois 60126	Paula.lafleur@billerud.com Mr. Daniel Kossack Project Manager (630) 993-2100 (phone) dkossack@mp-mail.com		

The test crew consisted of Messrs. E. Thomas, J. Kukla, P. Coleman, and D. Kossack of Mostardi Platt.

## 2.0 TEST METHODOLOGY

Emissions testing were conducted following the methods specified in 40 CFR, Part 60, Appendix A. Schematics of the test section diagrams and sampling trains used are found in Appendix B and C, respectively. Calculation nomenclature and sample calculations are found in Appendix D. Sample analysis data are found in Appendix E. Copies of reference method data and field data sheets for each test run are included in Appendix F and G, respectively.

The following methodologies were used during the test program:

### Method 1 Traverse Point Determination

Test measurement points were selected in accordance with Method 1. The characteristics of the measurement location are summarized below.

	TEST POINT INFORMATION							
Location	Stack Diameter (Feet)	No. of Ports	Port Length (Inches)	Upstream Diameters	Downstream Diameters	Test Parameter	Number of Sampling Points	
			VOC	1				
			10		21.6	TRS	3	
Smelt Dissolving Tank	4	2		3.4		SO <sub>2</sub> (Stratification Test prior to or during Run 1)	12	
						SO <sub>2</sub> (Runs 2 and 3)	12	
						ТРМ	24	

### Gaseous Sampling Plan

Twelve test points were used to sample the  $SO_2$ . A single test point was used to sample  $SO_3$  as  $H_2SO_4$  and VOC and TRS at the Smelt Dissolving Tank Stack.

#### Method 2 Volumetric Flowrate Determination

Gas velocity was measured following Method 2, for purposes of calculating stack gas volumetric flow rate at all test locations. An S-type pitot tube, differential pressure gauge, thermocouple and temperature readout were used to determine gas velocity at each sample point. All of the equipment used was calibrated in accordance with the specifications of the Method. Calibration data are presented in Appendix H. All locations have passed the null point pitot check and the results are on file.

### Method 3A Oxygen (O<sub>2</sub>)/Carbon Dioxide (CO<sub>2</sub>) Determination

Stack gas molecular weight was determined in accordance with Method 3A, 40 CFR, Part 60, Appendix A at all test locations. Servomex analyzers were used to determine stack gas oxygen and carbon dioxide content and, by difference, nitrogen content. All of the equipment used was calibrated in accordance with the specifications of the Method. Calibration data are presented in Appendix H and gas cylinder certifications are presented in Appendix I.

### Method 5 Filterable Particulate Matter (FPM) Determination

Stack gas FPM concentrations and emission rates were determined in accordance with USEPA Method 5, 40CFR60, Appendix A at all test locations. An Environmental Supply Company, Inc. sampling train was used to sample stack gas at an isokinetic rate, as specified in the Method utilizing Pallflex TX40HI45 filters. Particulate matter in the sample probe was recovered using a DI water rinse. The probe wash and filter catch were analyzed by Mostardi Platt in accordance with the Method in the Elmhurst, Illinois laboratory. Sample analysis data are found in Appendix E. All of the equipment used was calibrated in accordance with the specifications of the Method. Calibration data are presented in Appendix H.

### Method 202 Condensable Particulate Determination

Stack gas condensable particulate concentrations and emission rates were determined in accordance with the Method 202, in conjunction with Method 5 filterable particulate sampling. Condensable particulate matter was collected in the impinger portion of the Method 201A sampling trains.

The condensable particulate matter (CPM) was collected in impingers, after filterable particulate material was collected, using Method 201A. The organic and aqueous fractions were then taken to dryness and weighed. The total of all fractions represents the CPM. Compared to the December 17, 1991 promulgated Method 202, this Method includes the addition of a condenser, followed by a water dropout impinger immediately after the final heated filter. One modified Greenburg Smith impinger and an ambient temperature filter follow the water dropout impinger.

CPM was collected in the water dropout, modified Greenburg Smith impinger and ambient filter portion of the sampling train as described in this Method. The impinger contents were purged with nitrogen ( $N_2$ ) immediately after sample collection to remove dissolved sulfur dioxide ( $SO_2$ ) gases from the impingers. The impinger solution was then extracted with DI water, acetone, and hexane. The organic and aqueous fractions were dried and the residues weighed. The total of the aqueous, organic, and ambient filter fractions represents the CPM. Laboratory analysis data are found in Appendix E. All of the equipment used was calibrated in accordance with the specifications of the Method. Calibration data are presented in Appendix H.

### Method 6C Sulfur Dioxide (SO<sub>2</sub>) Determination

Stack gas SO<sub>2</sub> concentrations and emission rates were determined in accordance with USEPA Method 6C, 40CFR60, Appendix A. A Thermo Scientific Model 43iQ Pulsed Fluorescence Sulfur Dioxide Analyzer was used to determine sulfur dioxide concentrations, in the manner specified in the Method. The instrument operated in the nominal range of 0 ppm to 100 ppm with the specific range determined by the high-level span calibration gas of 25.57 ppm.

The Model 43iQ operates on the principle that SO<sub>2</sub> molecules absorb ultraviolet (UV) light and become excited at one wavelength, then decay to a lower energy state emitting UV light at a different wavelength. Specifically,

 $SO_2 + hv_1 \rightarrow SO_2^* \rightarrow SO_2 + hv_2$ 

The sample is drawn into the Model 43iQ through the sample bulkhead. The sample flows through a hydrocarbon "kicker", which removes hydrocarbons from the sample by forcing the hydrocarbon molecules to permeate through the tube wall. The SO<sub>2</sub> molecules pass through the hydrocarbon "kicker" unaffected.

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The sample flows into the fluorescence chamber, where pulsating UV light excites the  $SO_2$  molecules. The condensing lens focuses the pulsating UV light into the mirror assembly. The mirror assembly contains four selective mirrors that reflect only the wavelengths which excite  $SO_2$  molecules.

As the excited  $SO_2$  molecules decay to lower energy states, they emit UV light that is proportional to the  $SO_2$  concentration. The bandpass filter allows only the wavelengths emitted by the excited  $SO_2$  molecules to reach the photomultiplier tube (PMT). The PMT detects the UV light emission from the decaying  $SO_2$  molecules. The photodetector, located at the back of the fluorescence chamber, continuously monitors the pulsating UV light source and is connected to a circuit that compensates for fluctuations in the lamp intensity.

As the sample leaves the optical chamber, it passes through a flow sensor, a capillary, and the "shell" side of the hydrocarbon kicker. The Model 43i outputs the SO<sub>2</sub> concentration to the front panel display, the analog outputs, and also makes the data available over the serial or Ethernet connection.

Stack gas was delivered to the analyzer through an EPM in-situ dilution sampling system. Stack gas concentrations were diluted at a nominal 100:1 ratio utilizing purified dilution air. The entire system was calibrated in accordance with the Method, using certified calibration gases introduced at the probe, before and after each test run.

A list of calibration gases used and the results of all calibration and other required quality assurance checks can be found in Appendix H. Copies of calibration gas certifications can be found in Appendix I.

### Method 16A Total Reduced Sulfur (TRS) Determination

Integrated gas samples were extracted in accordance with Method 16A (impinger technique), 40CFR60, Appendix A, for the determination of total reduced sulfur (TRS) emissions from stationary sources at the Smelt Dissolving Tank Stack. This method selectively removed sulfur dioxide (SO<sub>2</sub>) by bubbling the gas sample through a citrate buffer solution, then thermally oxidizing TRS compounds present to SO<sub>2</sub>. The SO<sub>2</sub> was then collected in hydrogen peroxide as sulfate and analyzed by the Method 6 barium-thorin titration procedure, 40CFR60.

Interferences caused by particulate matter entering the sampling train were eliminated by the use of a heated filter medium placed at the end of the heated Teflon®-lined (or glass-lined) probe.

System validations were performed following each set of one-hour tests in the manner described as follows.

A 30-minute system validation test was performed to validate the sampling train components and procedure. The checks involved sampling a known concentration of hydrogen sulfide ( $H_2S$ ) and comparing the analyzed concentration with the known concentration. The  $H_2S$  recovery gas was mixed with combustion gas in a dilution system. The samples were collected and analyzed in the same manner as a normal stack test. The sample was collected through the end of the probe to ensure extraction of a representative sample. Sampling included a three one-hour test followed by a 30-minute system validation to be completed as one test run. Sampling train preparation was in accordance with Method 16A, 40CFR60, and included the following:

- 1. All probes, filter holders, and sampling lines were cleaned prior to each test.
- 2. The SO<sub>2</sub> scrubber was charged with 100 mls of citrate buffer solution into each of the first two impingers prior to each test. Citrate buffer solution was prepared by dissolving 300 g of potassium citrate (or 284 g of sodium citrate) and 41 g of anhydrous citric acid in one liter of water. The pH of the solution was then adjusted to between 5.4 and 5.6 with potassium citrate or citric acid, as required.
- 3. The probe and heated filter media were maintained at approximately 250°F to prevent moisture condensation.
- 4. The thermal oxidation furnace was maintained at 800°C±100°C during the test time.
- 5. The SO<sub>2</sub> portion of the train was prepared and analyzed in accordance with the methods described for sulfur dioxide testing listed previously.

Laboratory analysis data are found in Appendix E. All of the equipment used was calibrated in accordance with the specifications of the Method. Calibration data are presented in Appendix H.

### Method 25A Volatile Organic Concentration Determination

The Method 25A sampling and measurement system meets the requirements for sampling of VOCs set forth by the United States Environmental Protection Agency (USEPA). In particular, it meets the requirements of USEPA Reference Method 25A, "Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer," 40CFR60, Appendix A. This method applies to the measurement of total gaseous organic concentration of hydrocarbons. With this method, gas samples are extracted from the sample locations through heated Teflon sample lines to the analyzers. A Thermo Scientific 51i analyzer was used to perform sampling.

The flame ionization detector (FID) used during this program was a Thermo Scientific 51i High-Temperature Total Hydrocarbon Analyzer. It is a highly sensitive FID that provides a direct reading of total organic vapor concentrations with linear ranges of 0-10, 100, 1000, and 10,000 ppm by volume. The instrument was calibrated using ultra-zero air and propane in air EPA Protocol standards. The calibrations were performed before and after sampling with calibration checks performed between each test run. Sampling was conducted continuously for three onehour periods. Sample times and locations are logged simultaneously on data loggers.

All of the equipment used was calibrated in accordance with the specifications of the Method. Calibration data are presented in Appendix H. The gas cylinder certifications are found in Appendix I.

## 3.0 TEST RESULTS SUMMARIES

Client:Billerud Quinnesec LLCFacility:Quinnesec, MichiganTest Location:Smelt Dissolving TankTest Method:5/202

Source Condition Date Start Time End Time	Normal 11/8/22 8:37 9:40 Run 1	Normal 11/8/22 10:20 11:22 Run 2	Normal 11/8/22 13:02 14:04 Run 3	Average
Stack Cond				
Average Gas Temperature, °F	172.5	170.2	167.8	170.2
Flue Gas Moisture, percent by volume	43.9%	41.7%	39.5%	41.7%
Average Flue Pressure, in. Hg	29.38	29.38	29.38	29.38
Gas Sample Volume, dscf	39.62	42.89	43.691	42.067
Average Gas Velocity, ft/sec	41.075	37.688	38.104	38.956
Gas Volumetric Flow Rate, acfm	30,970	28,416	28,730	29,372
Gas Volumetric Flow Rate, dscfm	14,240	13,627	14,352	14,073
Gas Volumetric Flow Rate, scfm	25,384	23,374	23,722	24,160
Average %CO <sub>2</sub> by volume, dry basis	0.0	0.0	0.0	0.0
Average %O <sub>2</sub> by volume, dry basis	21.2	21.0	20.9	21.0
Isokinetic Variance	100.0	96.5	93.3	96.6
Black Liquer Solids, ton/hr	91.0	91.0	91.0	91.0
Filterable Particulate	and the second se			
grams collected	0.10280	0.10160	0.10388	0.10276
grains/acf	0.0184	0.0175	0.0183	0.0181
grains/dscf	0.0400	0.0366	0.0367	0.0378
lb/hr	4.887	4.269	4.513	4.556
Ib/ton BLS	0.054	0.047	0.050	0.050
Condensable Particulate	the second se			
grams collected	0.01372	0.01540	0.01435	0.01449
grains/acf	0.0025	0.0027	0.0025	0.0026
grains/dscf	0.0053	0.0055	0.0051	0.0053
lb/hr	0.652	0.647	0.623	0.641
Ib/ton BLS	0.007	0.007	0.007	0.007
Total Particulate				
grams collected	0.11652	0.11700	0.11823	0.11725
grains/acf	0.0209	0.0202	0.0208	0.0206
grains/dscf	0.0453	0.0421	0.0418	0.0431
lb/hr	5.539	4.916	5.136	5.197
Ib/ton BLS	0.061	0.054	0.056	0.057

Billerud Quinnesec LLC Quinnesec, Michigan							
	Smelt Dissolving Tank						
		G	aseous S	Summary			
		1		THC ppm			
Test		Start	End	as CH4	Flowrate,	THC lb/hr	
No.	Date	Time	Time	(wet)	SCFM	as CH4	
1	11/09/22	10:20	11:19	16.4	23,763	0.98	
2	11/09/22	11:41	12:40	18.2	23,675	1.09	
3	11/09/22	13:00	13:59	19.6	23,215	1.15	
	Aver	age		18.1	23,551	1.07	

#### Billerud Quinnesec LLC Quinnesec Mill November 8 and 9, 2022 Smelt Dissolving Tank Stack

#### **TRS Data Summary**

								TRS as
Run					Flowrate,	BLS	TRS as	H2S
No.	Time	$C_{TRS}$ lbs/dscf	TRS PPM	Oxygen %	dscfm	tons/hr	H2S lbs/hr	lbs/TBLS
1	8:37-11:37	5.13824E-07	5.81	20.00	13,934	91.0	0.43	0.0047
2	13:00-16:00	4.56901E-07	5.17	20.00	14,352	91.0	0.39	0.0043
3	10:20-13:20	5.80279E-07	6.56	20.00	15,602	91.0	0.54	0.0059
	Avera	ge	5.85	20.00	14,629	91.0	0.45	0.0050

Billerud Quinnesec LLC Quinnesec, Michigan Smelt Dissolving Tank Gaseous Summary											
Test No.	Date	Start Time	End Time	SO₂ ppmvd wet	O₂ % (dry)	Moisture, %	Flowrate, DSCFM	Flowrate, SCFM	Liquor Solids (BLS) tons/hr	SO₂ lb/hr	SO <sub>2</sub> Ib/ton BLS
1	12/14/22	15:25	16:27	0.6	20.8	47.8	12,450	23,101	91.0	0.14	0.0015
2	12/14/22	17:25	18:27	1.6	20.5	45.7	12,496	23,013	91.0	0.37	0.0041
3	12/14/22	18:55	19:57	0.0	20.5	46.8	12,489	23,193	91.0	0.00	0.0000
	Aver	rage		0.7	20.6	46.8	12,478	23,102	91.0	0.17	0.0019

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## 4.0 CERTIFICATION

Mostardi Platt is pleased to have been of service to Billerud Quinnesec LLC. If you have any questions regarding this test report, please do not hesitate to contact us at 630-993-2100.

As project manager, I hereby certify that this test report represents a true and accurate summary of emissions test results and the methodologies employed to obtain those results, and the test program was performed in accordance with the methods specified in this test report.

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**Program Manager** 

Daniel Kossack

and

Scott W. Banach

**Quality Assurance** 

## APPENDICES

### Appendix A - Plant Operating Data

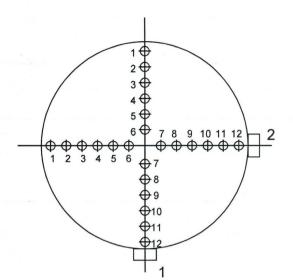
#### Smelt Dissolving Tank - Operating/Process Data

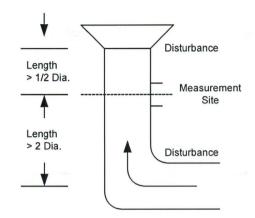
		Smelt Dissolving T				Carebbas Elau
TRS EPA 16A	Run Start	Run End	BLS tons/hr	Steaming rate kpph	Fan Amperage %	Scrubber Flow gpm
Run 1	11/8/22 8:37	11/8/22 11:37	91.0	600.76	93.84	223.05
Run 2	11/8/22 13:00	11/8/22 16:00	91.0	601.24	93.61	223.16
Run 3	11/9/22 10:20	11/9/22 13:20	91.0	601.36	91.97	223.35
TPM EPA 5 + 202	Run Start	Run End	BLS tons/hr	Steaming rate kpph	Fan Amperage %	Scrubber Flow gpm
Run 1	11/8/22 8:37	11/8/22 9:40	91.0	603.43	93.33	223.17
Run 2	11/8/22 10:20	11/8/22 11:22	91.0	597.55	94.25	222.96
Run 3	11/8/22 13:02	11/8/22 14:04	91.0	600.38	94.38	223.08
VOC EPA 25A	Run Start	Run End	BLS tons/hr	Steaming rate kpph	Fan Amperage %	Scrubber Flow gpm
Run 1	11/9/22 10:20	11/9/22 11:20	91.0	603.09	92.36	223.30
Run 2	11/9/22 11:41	11/9/22 12:41	91.0	600.06	91.75	223.40
Run 3	11/9/22 13:00	11/9/22 14:00	91.0	601.81	91.72	223.33
SO2 EPA 6C	Run Start	Run End	BLS tons/hr	Recovery Steam Flow kpph	Fan Amperage %	Scrubber Flow gpm
Run 1	12/14/22 15:25	12/14/22 16:27	91.0	607.75	89.74	222.36
Run 2	12/14/22 17:25	12/14/22 18:27	91.0	606.02	90.15	222.22
Run 3	12/14/22 18:55	12/14/22 19:57	91.0	603.67	90.12	222.35

Smelt Dissolving Tank Run Time/Process Data

### Appendix B - Test Section Diagrams

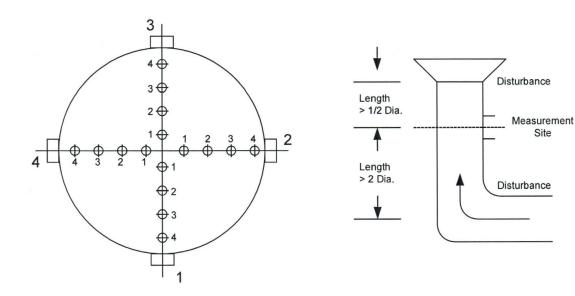
## EQUAL AREA TRAVERSE FOR ROUND DUCTS





Job:	Billerud Quinnesec LLC Quinnesec, Michigan
Date:	November 8 and 9, 2022
Test Location:	Smelt Dissolving Tank Stack
Stack Diameter:	4.0 Feet
Stack Area:	12.566 Square Feet
No. Points Across Diameter:	24
No. of Ports:	2
Port Length:	10 Inches

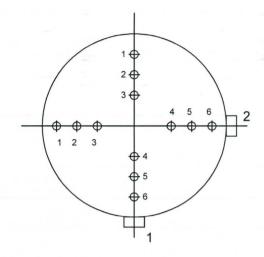
## EQUAL AREA TRAVERSE FOR ROUND DUCTS

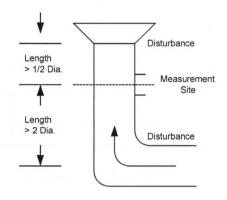


Job: Billerud Quinnesec, LLC Quinnesec, Michigan

- Date: November 8,9 and Dec.14,2022
- Test Location: Smelt Dissolving Tank
- Stack Diameter: 4.0 Feet
  - Stack Area: 12.566 Square Feet
- No. Sample Points Across Diameter: 8
  - No. of Ports 2
  - Port Length: 10 Inches

## GASEOUS STRATIFICATION TEST TRAVERSE FOR ROUND DUCTS





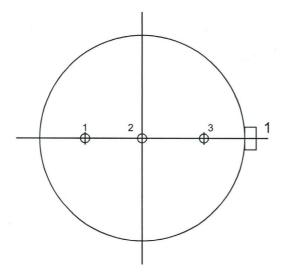
Job:	Billerud Quinnesec LLC Quinnesec, Michigan
Date:	December 14, 2022
Test Location:	Smelt Dissolving Tank Stack
Stack Diameter:	4.0 Feet
Stack Area:	12.566 Square Feet
No. Sample Points:	12
Upstream:	Approximately 13.5 Feet
Downstream:	Approximately 86.5 Feet

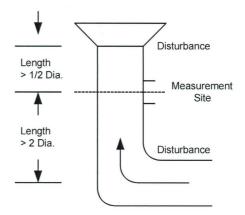
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## GASEOUS TRAVERSE FOR ROUND DUCTS



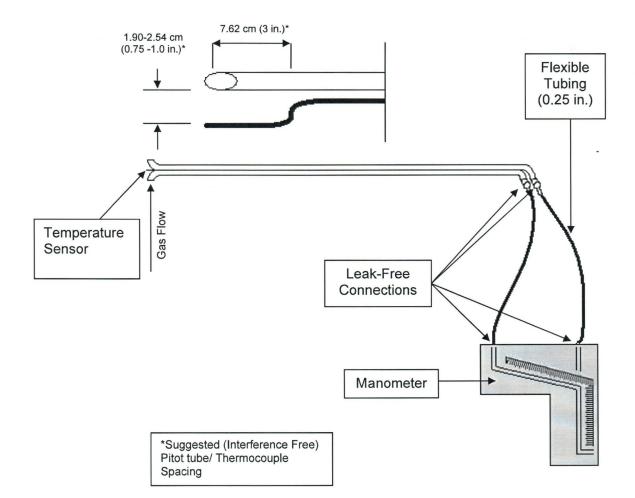


- Job: Billerud Quinnesec LLC Quinnesec, Michigan
- Date: November 8 and 9, 2022
- Test Location: Smelt Dissolving Tank Stack
- Stack Diameter: 4.0 Feet
  - Stack Area: 12.566 Square Feet
- No. Sample Points: 3
  - Upstream: approximately 13.5 feet
  - Downstream: approximately 86.5 feet

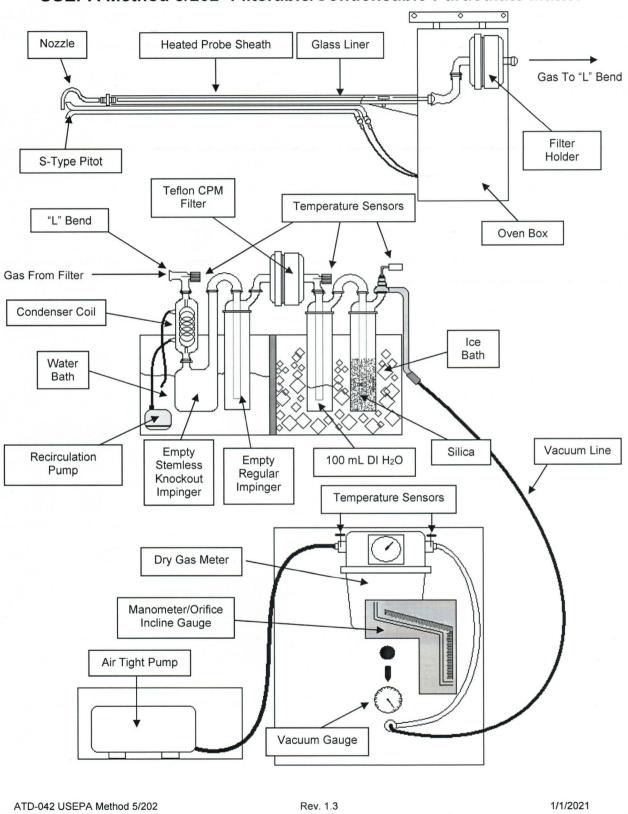
#### Distance from Inside Wall To Traverse Point:

- 1. 83.3 % of diameter
- 2. 50.0 % of diameter
- 3. 16.7 % of diameter

### Appendix C - Sample Train Diagrams

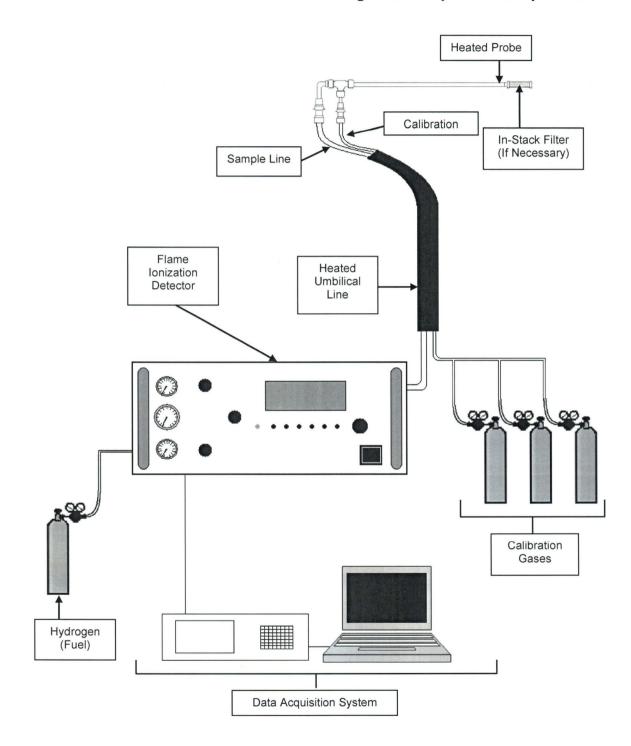


# USEPA Method 2 – Type S Pitot Tube Manometer Assembly



### USEPA Method 5/202- Filterable/Condensable Particulate Matter

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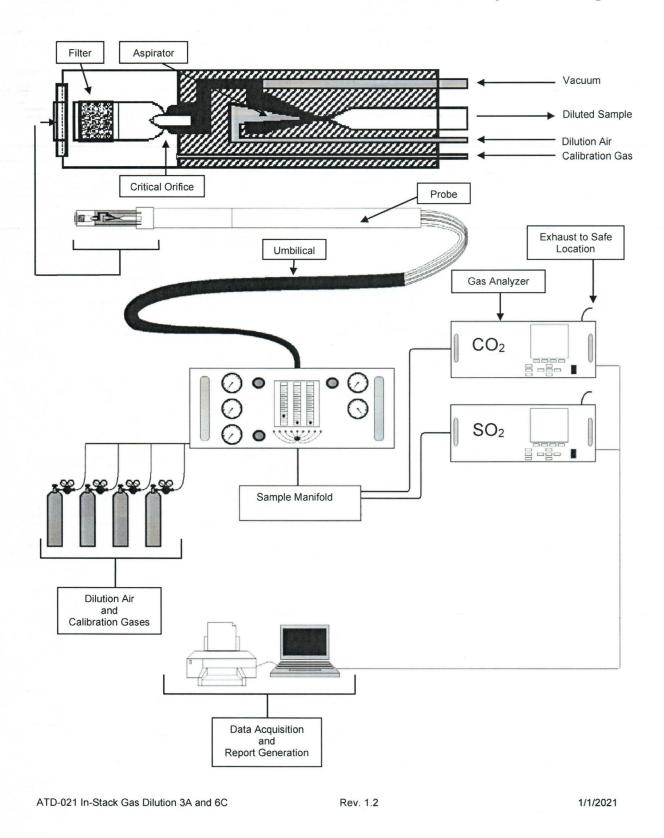


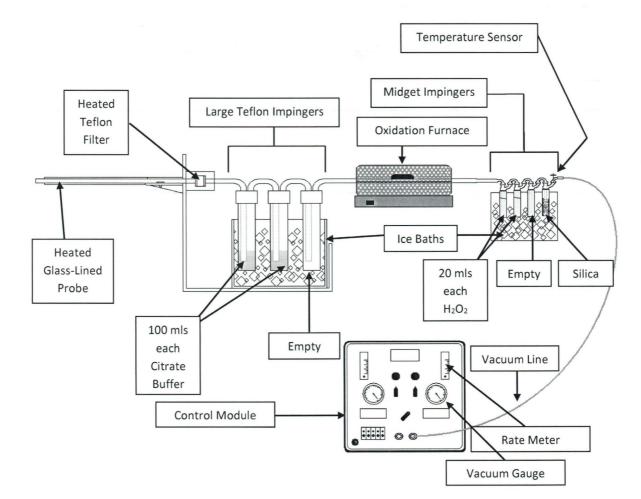
# USEPA Method 25A – Total Gaseous Organic Compound Sample Train

ATD-063 USEPA Method 25A

Rev. 1.2

### USEPA Methods 3A and 6C - Dilution Probe Gaseous Sample Train Diagram





### USEPA Method 16A – Total Reduced Sulfur Sample Train Diagram

Rev. 1.2