
Gas-Fired Steam Generator— Test Report Site C

Characterization of Fine Particulate Emission Factors and Speciation Profiles from Stationary Petroleum Industry Combustion Sources

Regulatory and Scientific Affairs

PUBLICATION NUMBER 4712
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PREPARED UNDER CONTRACT BY:

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Section 1

PROJECT DESCRIPTION

PROJECT OVERVIEW

In 1997, the United States Environmental Protection Agency (EPA) promulgated new ambient air standards for particulate matter, including for the first time particles with aerodynamic diameters smaller than 2.5 micrometers (PM_{2.5}). There are few existing data regarding emissions and characteristics of fine aerosols from petroleum industry combustion sources, and the information that is available is old. Traditional stationary source air emission sampling methods tend to underestimate or overestimate the contribution of the source to ambient aerosols because they do not properly account for primary aerosol formation, which occurs after the gases leave the stack. This issue was extensively reviewed by the American Petroleum Institute (API) in a recent report (England *et al.*, 1997), which concluded that dilution sampling techniques are more appropriate for obtaining a representative sample from combustion systems. These techniques have been widely used in research studies (Hildemann *et al.*, 1994; McDonald *et al.*, 1998) and use clean ambient air to dilute the stack gas sample and provide 80-90 seconds residence time for aerosol formation prior to sample collection for determination of mass and chemical speciation.

As a result of the API review, a test protocol was developed based on the dilution sampling system described in this report. The dilution sampling protocol was used to collect particulate emissions data from petroleum industry combustion sources, along with emissions data obtained from conventional sampling methods. This test program is designed to provide reliable source emissions data for use in assessing the contribution of petroleum industry combustion sources to ambient PM_{2.5} concentrations. The goals of this test program were to:

- Develop emission factors and speciation profiles for emissions of fine particulate matter, especially organic aerosols; and
- Identify and characterize PM_{2.5} precursor compound emissions.

This test report describes the results of tests performed on a gas-fired steam generator at Site C on October 21, 22 and 25, 1999.

PROJECT OBJECTIVES

The specific objectives of this test were to:

Primary Objectives

- Compare PM_{2.5} mass measured using an in-stack filter and an iced impinger train (EPA Method 201A/202), and mass measured using a dilution tunnel;
- Develop emission factors and speciation profiles for organic aerosols and PM_{2.5} mass;
- Characterize sulfate, nitrate, ammonium, inorganic elements, elemental carbon (EC) and organic carbon (OC) in particulate matter collected on filter media in the dilution sampler;
- Characterize key secondary particle precursors in stack gas samples: volatile organic compounds (VOC) with carbon number of 7 and above; sulfur dioxide (SO₂); and oxides of nitrogen (NO_x); and
- Document the relevant process design characteristics and operating conditions during the test.

Secondary Objective

- Characterize ions (sulfate, nitrate and ammonium), OC, and EC in particulate collected on filter media in stack gas sampling trains.

TEST OVERVIEW

The scope of testing is summarized in Table 1-1. The emissions testing included simultaneous collection and analysis of both in-stack and diluted stack gas samples. All emission samples were collected from the stack of the unit. The samples were analyzed for the compounds listed in Table 1-2. Process data and fuel gas samples were collected during the tests to document operating conditions.

Source Level (In-stack) Samples

In-stack sampling and analysis for filterable (total, PM₁₀ and PM_{2.5}) and condensible particulate matter (CPM), NO_x, oxygen (O₂), carbon dioxide (CO₂), carbon monoxide (CO) and SO₂ was performed using traditional EPA methods. In-stack cyclones and filters were used for filterable particulate matter. Sample analysis was expanded to include OC, EC and organic species on the in-stack quartz filters.

Table 1-1. Overview of Sampling Scope.

Sampling Location	Number of Samples		
	Fuel Gas Header	Stack	Ambient Air
EPA Method 201A/202 train	--	3	--
EPA Method 6 train	--	3	--
Dilution tunnel Teflon [®] filter TIGF/PUF/XAD-4 Quartz filter Tenax	--	3	1
Fuel sample	3	--	--
NO _x , CO, O ₂ , CO ₂	--	Continuous	--
Process monitoring	--	Hourly	--

TIGF - Teflon[®]-impregnated glass fiber filter

PUF - polyurethane foam

XAD-4 - Amberlite[®] sorbent resin

Dilution Stack Gas Samples

Dilution sampling was used to characterize PM_{2.5} including aerosols formed in the near-field plume. The dilution sampler extracted a sample stream from the stack into a mixing chamber, where it was diluted approximately 21:1 with purified ambient air. Because PM_{2.5} behaves aerodynamically like a gas at typical stack conditions, the samples were extracted nonisokinetically. A slipstream of the mixed and diluted sample was extracted into a residence time chamber where it resided for approximately 80 seconds to allow time for low-concentration aerosols, especially organics, to condense and grow. The diluted and aged sample then passed through cyclone separators sized to remove particles larger than 2.5 microns, after which samples were collected on various media: high-purity quartz, Teflon[®] membrane filters (TMF), and Teflon[®]-impregnated glass fiber (TIGF) filters; a polyurethane foam (PUF)/Amberlite[®] sorbent resin (XAD-4)/PUF cartridge to collect gas phase semivolatile organic compounds; and a Tenax cartridge to capture VOCs. Three samples were collected on three sequential test days.

Table 1-2. Summary of Analytical Targets.

Parameters	In-Stack				Dilution Tunnel				
	Cyclones	Quartz Filter	Impingers	Gases	Quartz Filter	TIGF/XAD-4	TMF	Tenax	Gases
Total PM mass	X	X							
PM10 mass	X	X							
PM2.5 mass	X	X					X		
Condensible particulate mass			X						
Sulfate			X		X				
Chloride			X		X				
Ammonium			X						
Nitrate			X		X				
Elements			X				X		
Organic carbon		X			X				
Elemental carbon		X			X				
Semivolatile organic compounds		X				X			
Volatile organic compounds*								X	
NO _x				X					
SO ₂			X						
CO				X					
O ₂				X					
CO ₂				X					
Moisture or relative humidity			X						X
Velocity				X					
Temperature				X					X

TMF - Teflon® membrane filter

TIGF - Teflon®-impregnated glass fiber filter

*Carbon number of 7 or greater

An ambient air sample was collected to establish background concentrations of measured substances. The same sampling and analysis procedures used for the dilution tunnel were applied for collecting ambient air samples.

Process Samples

A sample of the fuel gas burned in the steam generator was collected on each day of testing and analyzed for specific gravity, heating value, and hydrocarbon speciation.

KEY PERSONNEL

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Section 2

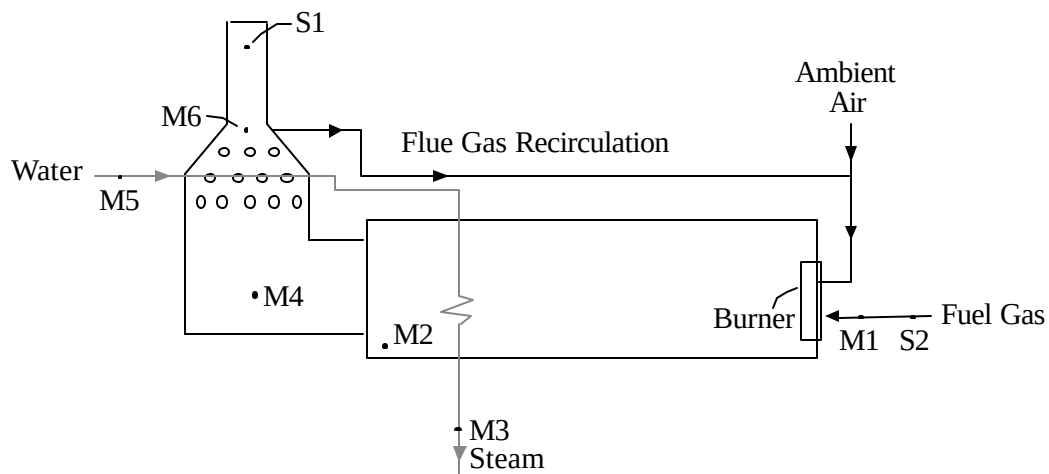
PROCESS DESCRIPTION

The tests were performed on a gas-fired steam generator at Site C. The generator has a maximum heat input of 62.5 MMBtu/hr with an average rate of approximately 50 MMBtu/hr. The unit is an oil field steam generator with a single burner and retrofitted with flue gas recirculation. The generator was designed to fire both crude oil and natural gas, but is now only fired on natural gas. The generator appeared to be in good working condition during the test. Operating conditions during the test are given in Section 4. Process parameters monitored during testing include: burner gas rate; inlet water rate; steam quality; radiant section, steam and stack temperature; and excess oxygen.

SAMPLING LOCATIONS

Figure 2-1 provides an overview of the generator process and the sampling and monitoring locations. Flue gas samples were collected from the stack. The single stack is equipped with a 3 feet by 10.5 feet rectangular sampling platform located approximately 25 feet above the ground, which is accessible via a ladder. There are two 4-inch diameter sampling ports on the stack which are at 45° to one another. The ports are threaded with a 4-inch nipple. The stack diameter at this elevation is 36 inches. The sample ports are located 16 and 29 inches (0.4 and 0.9 diameters) downstream and 104 and 91 inches (2.9 and 2.5 diameters) upstream of flow disturbances. Following velocity and O₂ traverses to check for uniform mixing, all sampling was performed at a single point in the center of the stack to facilitate co-location of the dilution tunnel and EPA Method 201A/202 probes.

Fuel gas samples were collected from the gas supply fuel-sampling manifold. Ambient air samples were collected at ground-level close to the air inlet for the steam generator.



<u>Sampling location</u>	<u>Description</u>	<u>Parameters</u>
S1	Stack	See Table 3-1
S3	Fuel gas feed	
M1	Fuel gas feed	Burner gas rate
M2	Radiant section	Radiant temperature
M3	Steam feed	Steam temperature, steam quality
M4	Radiant section outlet	Excess oxygen
M5	Water feed	Inlet water rate
M6	Stack	Stack temperature

Figure 2-1. Generator Process Overview and Sampling/Monitoring Locations.

Section 3

TEST PROCEDURES

An overview of the sampling and analysis procedures is given in Table 3-1. Figure 3-1 shows the testing chronology for the dilution tunnel and in-stack methods. The time of day for the start and finish of each measurement run is shown on the figure. For example, Method 201A/202 Run 1 began at 09:30 hours and finished at 15:30 hours on Thursday, October 21. Dilution tunnel testing and in-stack testing were performed concurrently. All samples were collected at approximately the same point in the center of the stack; the dilution tunnel and in-stack test method probes were co-located. Testing during Run 3 was halted before the 6-hour sample time due to a process upset. A change in fuel quality caused the oxygen levels in the flue gas to drop, and the unit was not able to automatically adjust to the low levels.

STACK GAS FLOW RATE, MOISTURE CONTENT AND MOLECULAR WEIGHT

An S-type Pitot tube (EPA Method 2) was used to determine the average stack gas velocity and volumetric flow rate. Stack gas molecular weight was calculated in accordance with EPA Method 3. Moisture content of the sample was determined based on weight gain of the impingers used in the Method 201A/202 train according to EPA Method 4. A full velocity traverse of the stack was performed before and after each test to determine total stack gas flow rate.

O₂, CO₂, CO, AND NO_x

Major gases and pollutant concentrations in the stack sample were measured using a continuous emission monitoring system (CEMS), illustrated schematically in Figure 3-2. Table 3-2 lists the CEMS specifications. The sample was collected from a single traverse point in the stack after verifying that the gas concentration profile deviated by less than 10 percent of the mean concentration. Sample gas was passed through a primary in-stack sintered metal filter, a heated stainless steel probe, a heated Teflon[®] transfer line, a primary moisture removal system (heat exchanger impingers in an ice bath), a heated secondary filter, a diaphragm pump, and a heated back-pressure regulator to a thermoelectric water condenser. The condenser's heat exchangers are specially designed impingers that separate the condensate from the gas sample with a

Table 3-1. Summary of Test Procedures.

Sampling Location	Measurements	Sampling Approach	Sample Analyses	Reference
S1 (Stack)	Total PM, PM10, PM2.5 and composition	In-stack series cyclones and filter	Mass; organic species	U.S. EPA Method 201A (modified)
	Condensible PM and composition	Impingers	Mass (organic and inorganic), sulfate, chloride, nitrate, ammonium, elements	U.S. EPA Method 202 (modified)
	Gaseous PM2.5 Precursors	Continuous	NO _x (O ₂ , CO ₂ , CO also measured)	U. S. EPA Methods 3A/6C/7E/10
		Impingers	SO ₂	U.S. EPA Method 6; SCAQMD Method 6.1
S1 (Stack)	PM2.5 mass and chemical composition	Dilution tunnel and filters	Mass, organic carbon, elemental carbon, elements, sulfate, nitrate, chloride, ammonium	U.S. EPA, 1999a; Hildemann <i>et al.</i> , 1989
	VOC	Dilution tunnel and Tenax	Speciated VOC	Zielinska <i>et al.</i> , 1996; Hildemann <i>et al.</i> , 1989
	SVOC	Dilution tunnel and filter/PUF/XAD-4/PUF	Speciated SVOC	U.S. EPA Method TO13; Hildemann <i>et al.</i> , 1989
S2 (Ground level – ambient air)	PM2.5 and chemical composition	Filters	Mass, organic carbon, elemental carbon, organic species, elements, chloride, sulfate, nitrate, ammonium	U.S. EPA, 1999a
	VOC	Tenax	Speciated VOC	Zielinska <i>et al.</i> , 1996
	SVOC	PUF/XAD-4/PUF	Speciated SVOC	U.S. EPA Method TO13
S3 (Fuel gas feed to generator)	Fuel gas composition	Integrated grab sample (bag or canister)	Hydrocarbon speciation, CHON*, sulfur content and heating value	ASTM D3588-91

* carbon, hydrogen, oxygen, nitrogen

	Time	Generator Stack					Process Samples
		Velocity	Method 201/202	Method 6	CEMS	Dilution Tunnel	Fuel Sample
10/19/99 Tues.	9:00						
	10:00						
	11:00						
	12:00						
	13:00						
	14:00						
	15:00						
	16:00						
	17:00				Stratification		
	18:00				18:26		
	19:00				19:40		
	20:00						
10/21/99 Thurs.	8:00	8:19-8:33	Run 1		Run 1	Run 1	
	9:00		9:30		9:55	9:30	
	10:00			Run 1			
	11:00			11:17	12:00		
	12:00				12:25		12:49
	13:00						
	14:00			14:17	14:27		
	15:00		15:30		14:55-15:30	15:30	
	16:00	16:50					
	17:00	17:17					
	18:00						
10/22/99 Fri.	8:00	8:05-8:31	Run 2	Run 2		Run 2	
	9:00		9:15	9:47		9:15	9:55
	10:00				Run 2		
	11:00				11:38		
	12:00			12:47			
	13:00				13:18		
	14:00				13:49		
	15:00		15:15		15:15	15:15	
	16:00	16:46-17:13					
	17:00						
	18:00						
	19:00						
10/25/99 Mon.	8:00						
	9:00	9:54					
	10:00	10:26	Run 3			Run 3	
	11:00		11:00	Run 3	Run 3	11:00	
	12:00			12:17	12:00		
	13:00				14:28		13:31
	14:00				15:03		
	15:00			15:17	16:20		
	16:00		16:20			16:20	
	17:00						
	18:00	18:25-18:53					
	19:00						

Figure 3-1. Chronology for Gas-Fired Steam Generator Tests (Site C).

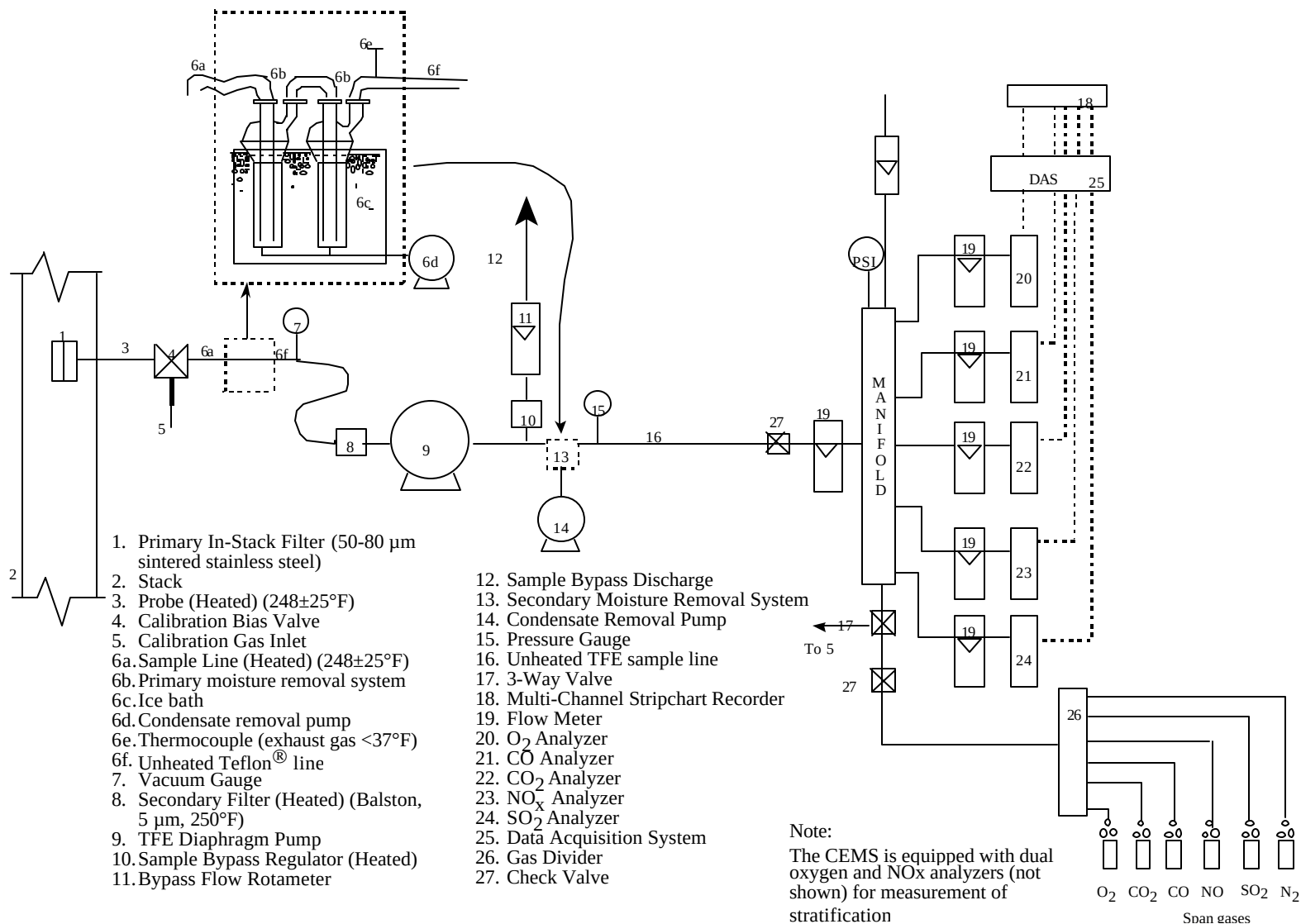


Figure 3-2. Continuous Emissions Monitoring System.

Table 3-2. CEMS Instrumentation Used For Gas-Fired Steam Generator Test (Site C).

Instrument/ Specification	Oxygen (O ₂)	Oxides of Nitrogen (NO _x)	Carbon Monoxide (CO)	Carbon Dioxide (CO ₂)
Instrument Manufacturer	Taylor- Servomex	Thermo- Electron	Thermo- Electron	ACS
Model Number	Model 1400	Model 10AR with molybdenum NO ₂ converter	Model 48H	Model 3300
Detection Principle	Paramagnetism	Chemi- luminescence	Gas filter correlation infrared absorption	Non-dispersive infrared absorption (NDIR)
Units Measured	%	ppmv	ppmv	%
Detection Limit	0.10%	1 ppm	0.5 ppm	0.10%
Ranges Used	0-10	0-100	0-100	0-20

minimum of contact area to avoid loss of the water soluble gas fraction. The condensate was removed with a peristaltic pump through the bottom of the heat exchanger. All components in contact with the sample were constructed of inert materials such as glass, stainless steel, and tetrafluoroethylene (TFE). All components preceding the condenser (probe, sample line, sample bypass regulator, and pump) were heated to 248° F to prevent condensation. The sample was conducted from the chiller outlet through the TFE line to a tertiary filter preceding the sample manifold. Samples were analyzed for O₂ and CO₂ using instrumental methods according to EPA Method 3A. O₂ was measured using a paramagnetic analyzer and CO₂ was measured using a non-dispersive infrared (NDIR) analyzer. Samples were analyzed for NO_x using a low-pressure chemiluminescence analyzer with a molybdenum nitrogen dioxide (NO₂)-to-nitric oxide (NO) converter according to EPA Method 7E. CO was determined using a NDIR analyzer following EPA Method 10.

SULFUR DIOXIDE (SO₂)

A modified version of SCAQMD Method 6.1 was used to measure SO₂ in the stack gas due to expected low concentrations. Flue gas was extracted non-isokinetically and passed through impingers containing hydrogen peroxide. Figure 3-3 shows a schematic of the Method 6 sampling train.

IN-STACK METHOD TESTS

Total particulate, PM₁₀ and PM_{2.5} filterable at stack temperature were determined using in-stack methods. CPM, defined as the material collected in chilled impingers, also was measured for the in-stack samples.

In-Stack Total Filterable PM, PM₁₀ and PM_{2.5}

Two in-stack cyclones followed by an in-stack filter (Figure 3-4) were used to measure total particulate and particulate matter with nominal aerodynamic diameters less than or equal to 10 µm (PM₁₀) and 2.5 µm (PM_{2.5}). EPA Method 201A, modified to accommodate the second cyclone, was used following the constant-rate sampling procedure. Sampling time was six hours for each of the three runs. The sample recovery field procedure is summarized in Figure 3-5. Sampling was performed as published except for the following modifications and clarifications:

- A PM₁₀ cyclone and a PM_{2.5} cyclone (Andersen Model Case-PM₁₀ and Case-PM_{2.5}) were attached in series to the filter inlet. Sample recovery procedures were modified accordingly;
- The sample was collected from a single traverse point near the center of the stack to preserve the integrity of the dilution tunnel method comparison. It was assumed that any particulate present was small enough to mix aerodynamically in the same manner as a gas; therefore, the magnitude of the particle concentration profile was assumed to be no greater than the gas concentration profile. Quartz filters were used. The filters were preconditioned in the same manner as those used in the dilution tunnel, as described below; and
- A modified filter assembly was employed in an effort to improve the precision of the gravimetric analysis for low particulate concentration. An o-ring, a filter and a filter support were all placed together in an aluminum foil pouch and weighed as a unit. Post-test all three components were recovered together into the same foil pouch to prevent negative bias due to filter breakage.

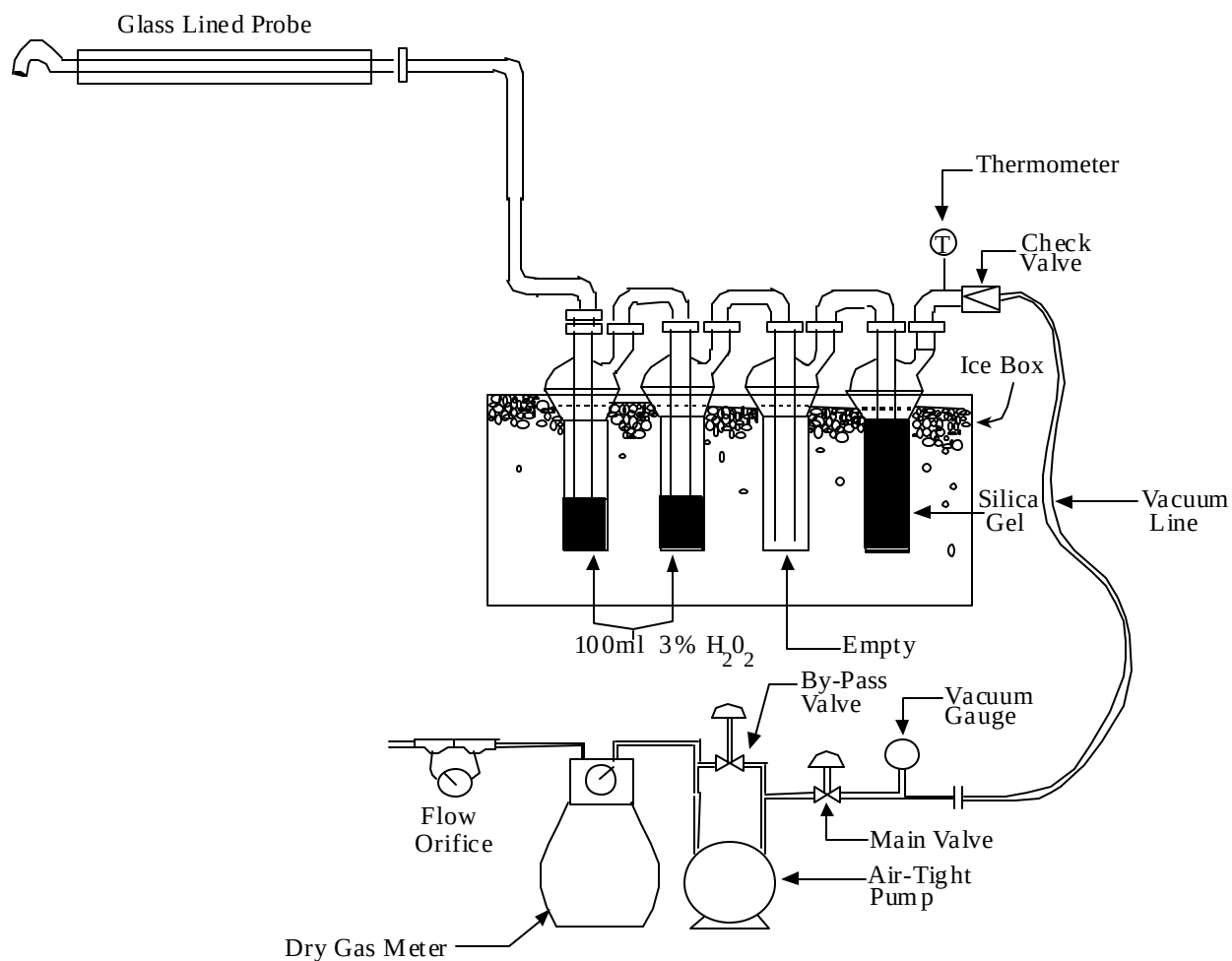
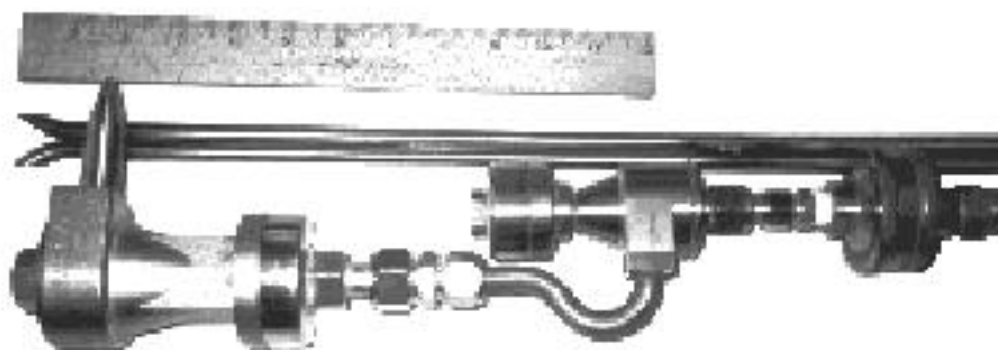
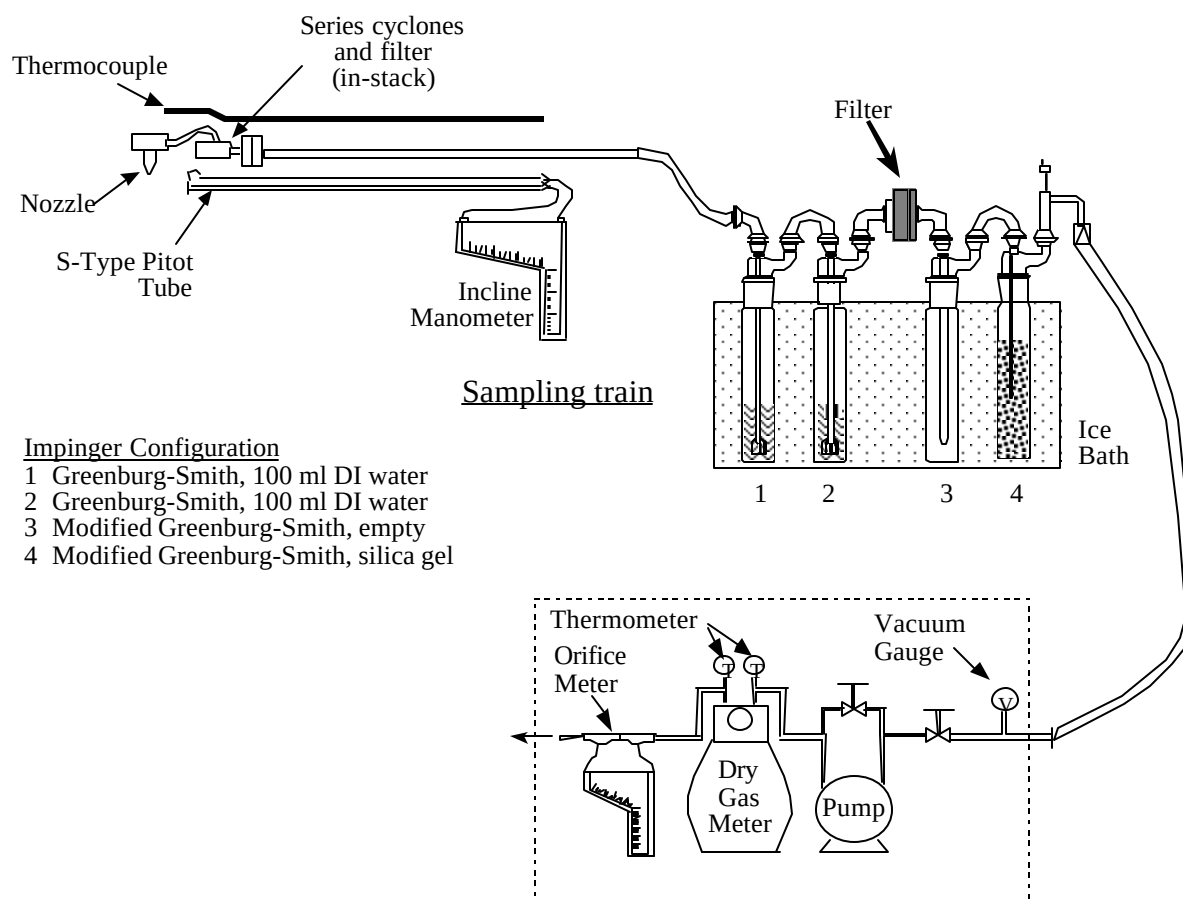


Figure 3-3. Modified SCAQMD Method 6.1 – Sulfur Oxides Sampling Train.



Series cyclone and filter assembly

Figure 3-4. PM10/PM2.5 Train Configuration for Method 201A/202.

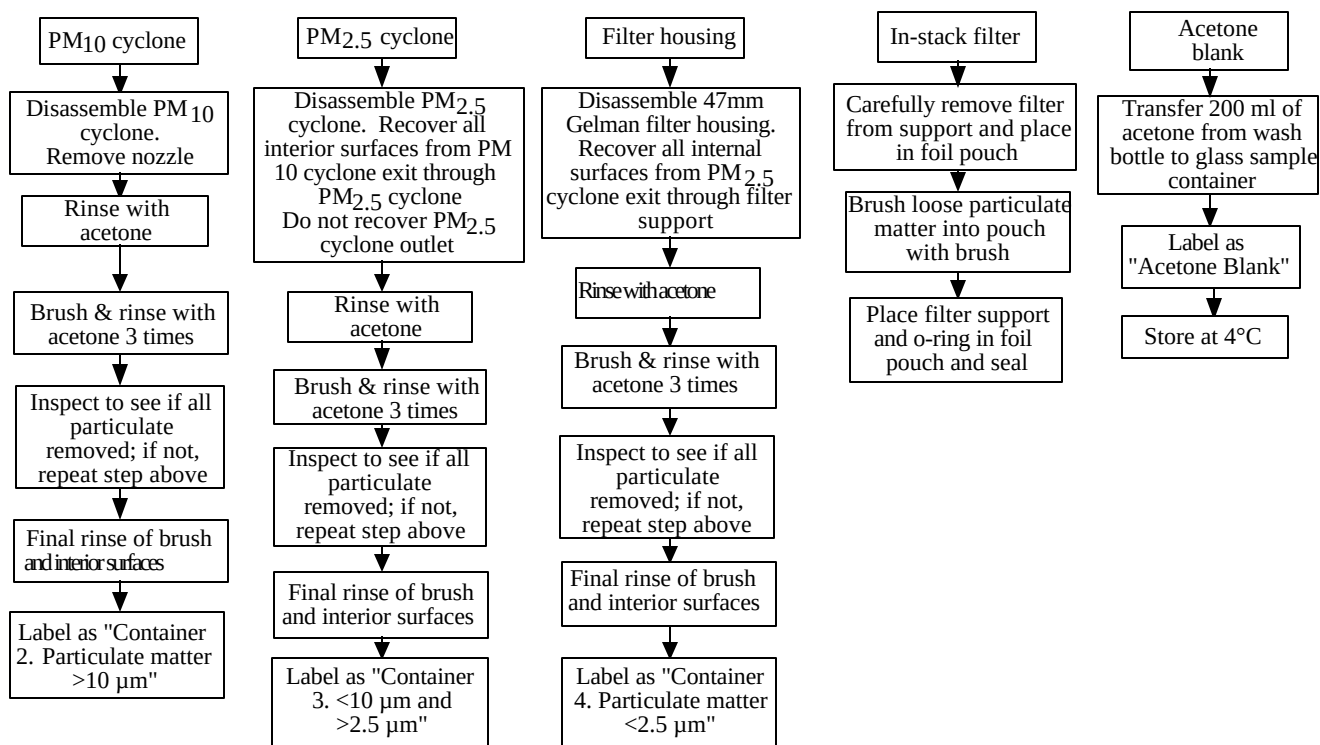


Figure 3-5. Method 201A (Modified) Sample Recovery Procedure.

The particulate mass collected in the two cyclones and on the filter was determined gravimetrically (Figure 3-6). The filters (Pallflex No. 51575) were weighed before and after testing on an analytical balance with a sensitivity of 10 micrograms. In an effort to improve the accuracy and precision of the gravimetric results, the filters, filter support and Viton O-ring seals were weighed together to minimize post-test loss of filter matter during sample recovery. Pre- and post-test weighing was performed after drying the filters in a dessicator for a minimum of 72 hours; repeat weighings were then performed at a minimum of 6-hour intervals until constant weight was achieved. Probe and cyclone acetone rinses were recovered in glass sample jars for storage and shipment, then transferred to tared Teflon[®] beaker liners for evaporation and weighing. Acetone and filter blanks also were collected and analyzed. See Section 4 for discussion of data treatment.

Subsequent to the planning of these tests, EPA published preliminary method PRE-4, entitled "Test Protocol PCA PM10/PM2.5 Emission Factor and Chemical Characterization Testing" (U.S. EPA, 1999b). This protocol, developed by the Portland Cement Association (PCA), is intended for use by Portland cement plants to measure PM10 and PM2.5 emission factors applicable to a variety of particulate sources. Method PRE-4 describes substantially the same sampling equipment and sample collection procedures used in these tests. The analytical procedures differ in the scope of chemical analyses performed: Method PRE-4 cites Method 202 for measurement of CPM, which includes analyses for inorganic and organic CPM mass, sulfate and chloride only. The analyses performed in these tests go beyond the requirements of Method 202 to further speciate the CPM by analysis for anions, cations, metals and VOCs, as described below.