
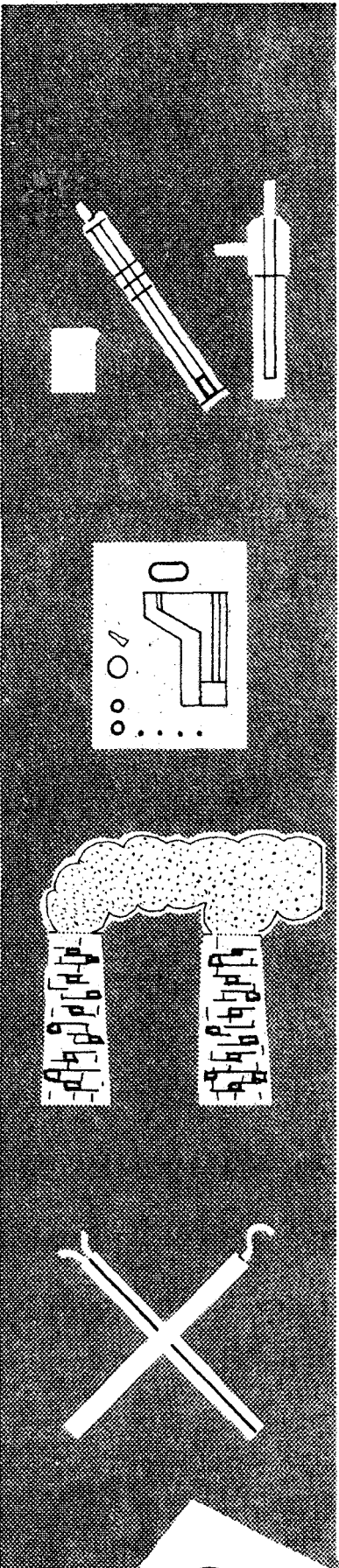


AIR POLLUTION EMISSION TEST

STAUFFER CHEMICAL COMPANY

DELAWARE CITY, DELAWARE



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air and Waste Management
Office of Air Quality Planning and Standards
Emission Measurement Branch
Research Triangle Park, North Carolina

environmental science and engineering, inc.

AN EQUAL OPPORTUNITY EMPLOYER

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SOURCE TEST REPORT

EMISSIONS FROM SULFUR RECOVERY PLANT
Stauffer Chemical Company
Delaware City, Delaware
for

The Environmental Protection Agency
United States Government
Report No. 74-SRY-4

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SOURCE TEST REPORT

REPORT NO: 74-SRY-4

PLANT TESTED: Stauffer Chemical Company
Delaware City, Delaware

EMISSIONS FROM: Sulfur Recovery Plant

TESTOR: Environmental Science and Engineering, Inc.
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CONTRACT NO: 68-02-1402, Task Order No. 1

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1.0 INTRODUCTION

Under Section III of the Clear Air Act of 1970, as amended, the Environmental Protection Agency is charged with the establishment of standards of performance for new stationary sources which may contribute significantly to air pollution. A performance standard is based on the best emission reduction systems which have been shown to be technically and economically feasible.

In order to set realistic performance standards, accurate data on pollutant emissions is normally gathered from the stationary source category under consideration.

The sulfur recovery system at Stauffer Chemical Company's Carbon Disulfide Plant at Delaware City, Delaware, was designated as a well controlled stationary source and was thereby selected by the Office of Air Quality Planning and Standards (OAQPS) for an emission testing program. Tests were conducted on the IFP sulfur recovery unit during June 10-12, 1974. The tests were performed by personnel from Environmental Science and Engineering, Inc., Gainesville, Florida, and the Environmental Protection Agency, Emission Measurement Branch, OAQPS, Research Triangle Park, North Carolina.

The sulfur recovery system consists of a 395 long tons per day (LT/D) Claus Sulfur Recovery Unit followed by a IFP-1500 tail gas plant for sulfur removal. The IFP-1500 unit treats the tail gas from the Claus Unit to remove additional sulfur before the gas is released to the atmosphere.

Tests were conducted at a point downstream from the IFP unit in the duct just before the incinerator. The tests were designed to determine the average emission rates during four-hour sampling periods on each of three days. The emissions measured were: sulfur compounds (hydrogen sulfide, carbonyl sulfide, carbon disulfide, sulfur dioxide), carbon dioxide, oxygen, carbon monoxide, nitrogen oxides, and visible emissions.

2.0 SUMMARY AND DISCUSSION OF RESULTS

- 2.1 A complete summary of all data collected for the three four-hour tests completed at Stauffer Chemical Company is included as Table 1. To facilitate a comparison of the results all concentrations are presented as ppmv dry (except % dry for CO₂ and O₂), and all emission rates are standardized as grams per hour (gm/hr). Sample calculations and conversion factors are presented in Appendix A.
- 2.2 Sulfur compound concentrations were determined by gas chromatography (COS, SO₂, H₂S and CS₂), DuPont Analyzer (total sulfur and SO₂), Meloy Analyzer (Total Sulfur), EPA Method 6 (SO₂), and EPA Method 11 (H₂S).
- 2.2.1 Average sulfur dioxide concentrations, as obtained by the various methods, ranged from 48 ppmv to 400 ppmv. General agreement was obtained between the gas chromatography values and those determined by EPA Method 6 (78 ppmv versus 48 ppmv). The SO₂ concentration obtained by the DuPont Analyzer is much higher (400 ppmv) and can be attributed to the fact that (1) the system tested contains elemental sulfur; and (2) the analysis method (UV) does not discriminate between sulfur dioxide and elemental sulfur.
- 2.2.2 The average results obtained for total sulfur (as SO₂) ranged from 2230 ppmv to 2330 ppm. When the results are compared for each

Table 1¹

DATA SUMMARY - OUTLET FROM IFP-1500 SULFUR RECOVERY UNIT
STAUFFER CHEMICAL COMPANY
Delaware City, Delaware

Date	Location	Flow Rate DM ³ /Min	% Moisture	Orsat Data			NDIR/PARAMAGNETIC			Emission Concentrations, ppmv, dry											
				% CO ₂	% O ₂	% CO	% CO ₂ (1)	% O ₂ (2)	CO ppmv (1)	Gas Chromatograph					Moloy TS(7)	DuPont TS(4)	NO _x (8)	H ₂ S(9)	Visible Emissions(10)		
10 June 1974	IFP Outlet	421	30.9	1.3	0.28	---	1.5	0.1	3240	59	420	82	132	1150	460	2310	2390	2380	7.8	1715	0
11 June 1974	IFP Outlet	431	37.7	1.1	0.35	---	1.6	---	2450	42	430	72	77	1410	180	1920	2590	2540	1.0	3800	0
12 June 1974	IFP Outlet	414	39.0	1.1	0.31	---	1.8	0.1	3140	42	360	80	133	1950	300	2760		2070	4.0	2403	--
12 June 1974	Incinerator Outlet	---	31.6	---	---	---	---	---	---	--	---	--	---	---	---	---	---	---	---	1652	--
	AVERAGE (IFP OUTLET)	422	35.9	1.2	0.31		1.6	0.1	2940	48	400	78	114	1520	310	2330	2490	2330	4.3	2640	0

MASS EMISSION RATES, gm/hr

Date	CO(1)	SO ₂ (3)	SO ₂ (4)	SO ₂ (5)	COS(5)	H ₂ S(5)	CS ₂ (5)	TS(6)	TS(7)	TS(4)	NO _x (8)	H ₂ S(9)
10 June 1974	9.37 x 10 ⁴	3.90 x 10 ³	2.78 x 10 ⁴	5.42 x 10 ³	8.17 x 10 ³	4.15 x 10 ⁴	3.60 x 10 ⁴	1.49 x 10 ⁵	1.58 x 10 ⁵	1.57 x 10 ⁵	3.71 x 10 ²	6.00 x 10 ⁴
11 June 1974	7.25 x 10 ⁴	2.84 x 10 ³	2.91 x 10 ⁴	4.87 x 10 ³	4.88 x 10 ³	5.04 x 10 ⁴	1.44 x 10 ⁴	1.30 x 10 ⁵	1.75 x 10 ⁵	1.72 x 10 ⁵	0.49 x 10 ²	13.6 x 10 ⁴
12 June 1974	8.93 x 10 ⁴	2.73 x 10 ³	2.34 x 10 ⁴	5.20 x 10 ³	8.09 x 10 ³	6.70 x 10 ⁴	2.31 x 10 ⁴	1.11 x 10 ⁵	1.35 x 10 ⁵	1.35 x 10 ⁵	1.87 x 10 ²	8.26 x 10 ⁴
AVERAGE	8.52 x 10 ⁴	3.16 x 10 ³	2.68 x 10 ⁴	5.16 x 10 ³	7.04 x 10 ³	5.30 x 10 ⁴	2.45 x 10 ⁴	1.30 x 10 ⁵	1.56 x 10 ⁵	1.55 x 10 ⁵	2.02 x 10 ²	9.29 x 10 ⁴

1. NDIR
2. Paramagnetic
3. EPA Method 6
4. DuPont Analyzer (Includes Elemental Sulfur)
5. Gas Chromatographic
6. Gas Chromatographic Sulfur Data Summarized as SO₂
7. Moloy Total Sulfur Analyzer
8. EPA Method 7
9. EPA Method 11
10. Visible Emissions By EPA Method 9

individual test, the total sulfur values obtained by the Meloy Analyzer and the DuPont Analyzer are in good agreement. Individual values obtained by the gas chromatograph are less by several hundred ppmv for two of the three tests. The difference between gas chromatography and the other two methods was expected due to the fact that the other methods also respond to elemental sulfur. No explanation is offered for the increase in total sulfur as determined by gas chromatography on the third test.

2.2.3 Hydrogen Sulfide, as determined by the gas chromatograph, varied from 1190 to 1950 ppmv, with the average value for the test computed at 1520 ppmv. Hydrogen sulfide determined by EPA Method 11 varied from 1715 to 3800 ppmv. The 3800 ppmv value appears to be in error since it is much greater than either the total sulfur or the hydrogen sulfide as obtained by other methods. Other hydrogen sulfide values obtained by EPA Method 11 are higher than the corresponding values obtained by gas chromatography but are in the same general range. General interferences with Method 11 are positive in nature and would tend to explain the differences.

2.2.4 Carbonyl Sulfide and Carbon Disulfide, as analyzed by gas chromatography, varied from 77 to 133 ppmv and 180 to 460 ppmv, respectively. No comparative methods were available for these components.

2.2.5 Carbon dioxide was determined by NDIR and gas chromatography (thermal conductivity). The results obtained varied from 1.1 to

1.3 (G.C.) and 1.5 to 1.8% (NDIR). Agreement between the two methods is within experimental error.

2.2.6 Oxygen was determined by paramagnetic analyzer and gas chromatography. A comparison of the results indicated that the values obtained by G.C. were slightly higher than those obtained by the paramagnetic analyzer (0.31% vs. 0.1%, Avg.).

2.2.7 Nitrogen oxides were determined by EPA Method 7. The results obtained varied from a low of 1.0 ppmv to a high of 7.8 ppm. Comparative tests by alternate methods were not made for nitrogen oxides.

2.2.8 Carbon monoxide was determined by NDIR and varied from 2450 to 3240 ppmv. Comparative results were not obtained for carbon monoxide by alternate methods.

2.2.9 Visible Emissions were determined by qualified observers in accordance with EPA Method 9. This phase of the test program was discontinued by the EPA project officer after the first two tests resulted in an average visible emissions of zero.

2.2.10 Hydrocarbons were not determined during the testing period due to a malfunction of the analyzer.

2.3 The overall results obtained from the various tests (SO_2 , H_2S , COS , SO_2 , CO , CO_2 , O_2 , etc.) seem to be in agreement with the results expected from the IFP Sulfur Removal Process.

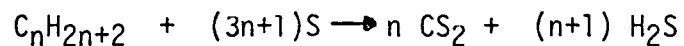
Agreement between comparative methods, where available, tends to substantiate the fact that the results are in the correct range and typify an operation of this type.

3.0 PROCESS DESCRIPTION

3.1 Process Description - Sulfur Recovery at Stauffer Chemical Company's Carbon Disulfide Plant

Figure 1 is a process schematic of carbon disulfide production and sulfur recovery similar to that found at the Stauffer Chemical Company carbon disulfide production facility at Delaware City, Delaware. The Stauffer plant produces CS_2 from the reaction between natural gas and sulfur known as the catalytic hydrocarbon-sulfur process.

The reaction is:



where $n = 1, 2, 3, \dots$

After the product carbon disulfide is absorbed from the process gas stream, the absorber vent gas, containing nearly 93 percent hydrogen sulfide, is directed to a sulfur recovery facility--a three stage, 395 LT/D Claus plant. The Claus plant operates at approximately 95 percent sulfur removal efficiency. Tail gas from the Claus plant goes to an IFP-1500 tail gas plant (Figure 2) which went onstream in December 1973. Carbonyl sulfide (COS) and CS_2 are not recovered by the IFP process but their presence is minimized back in the Claus by a reducing catalyst of cobaltmolybdenum and alumina in the first catalytic stage.

The IFP process reaction is a continuation of the gas phase Claus reaction in the liquid phase:

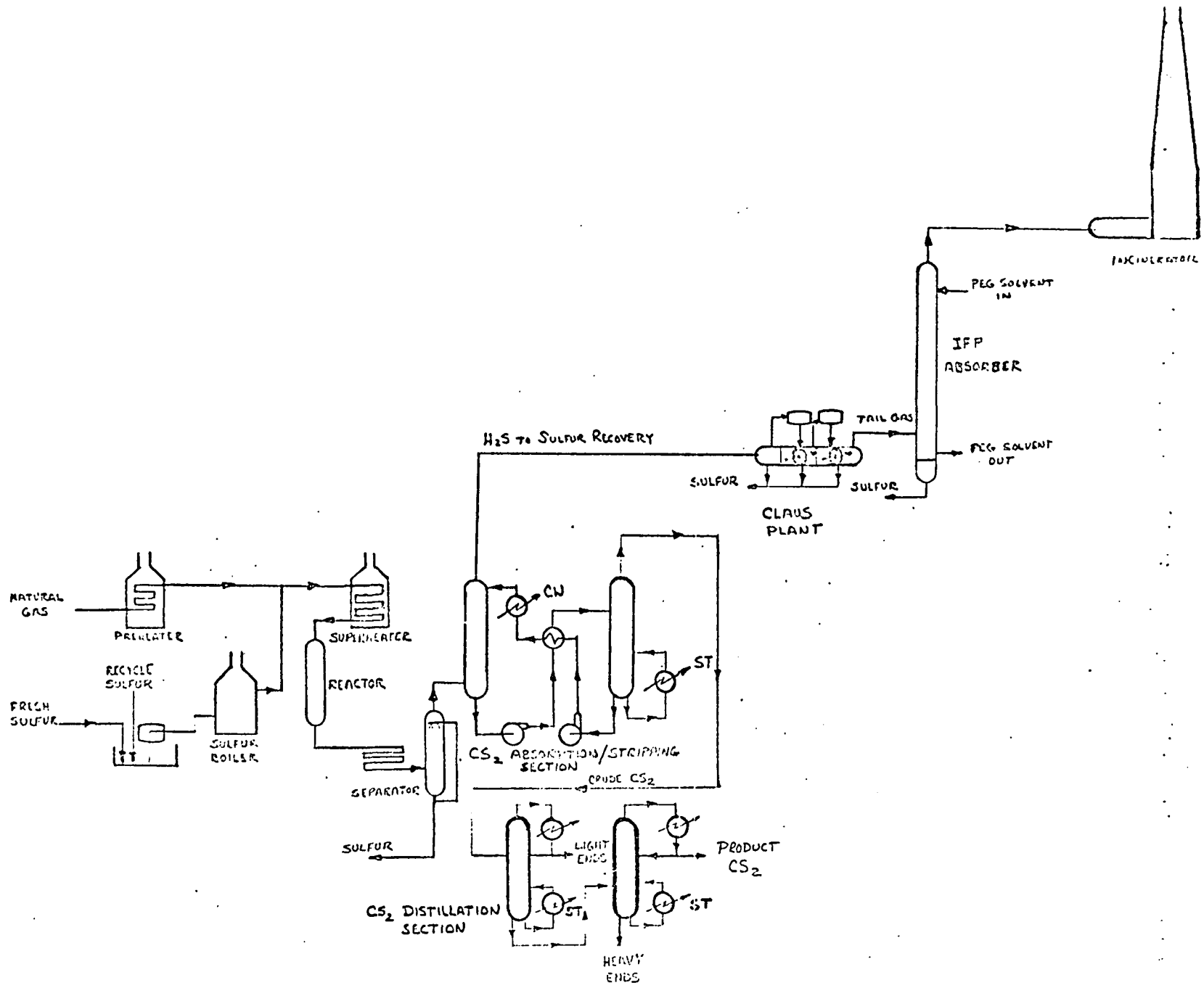


Figure 1. Process Schematic of Sulfur Recovery in CS₂ Production.

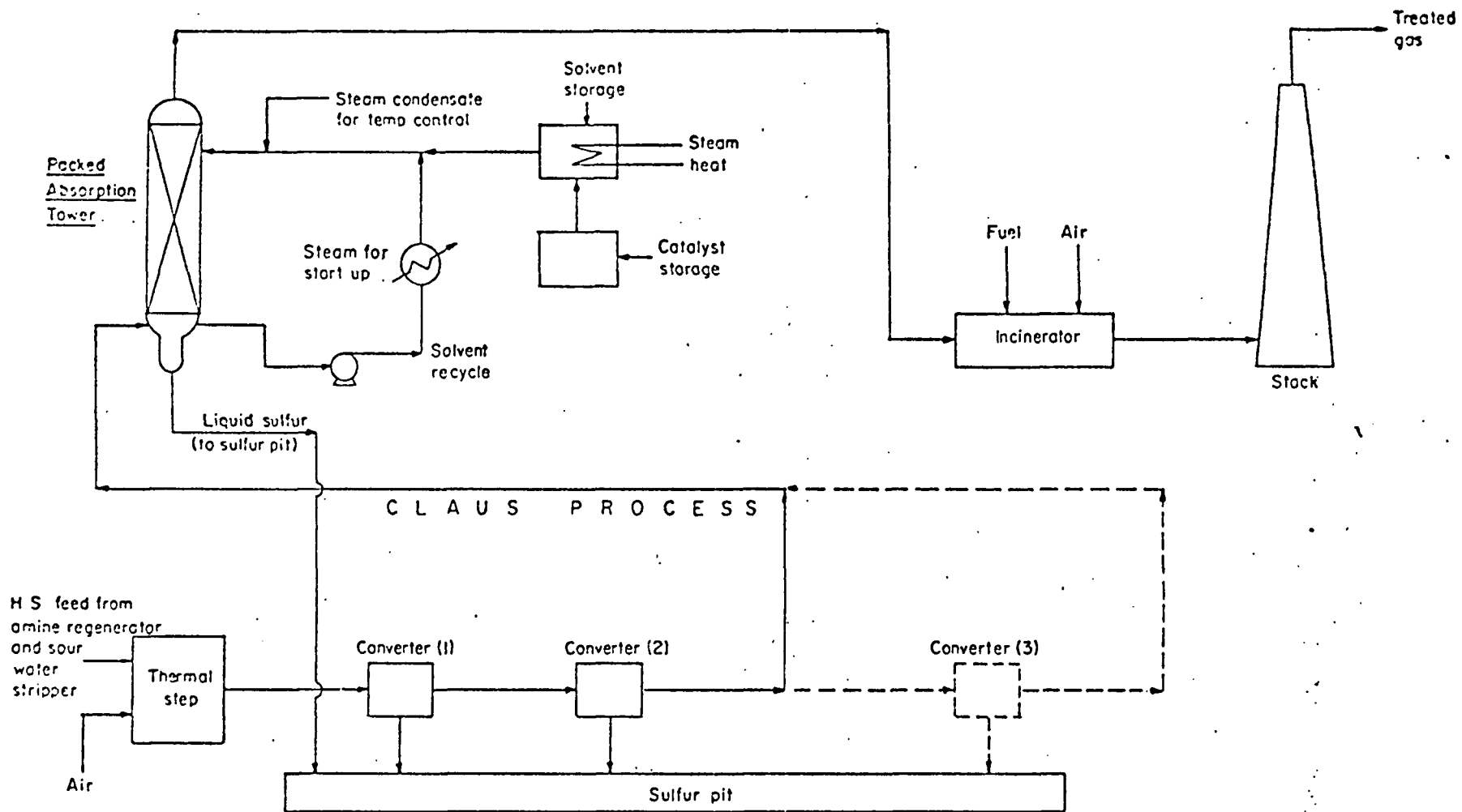


Figure 2. Flow Diagram for IFP (TGT-1500) Claus Tail-Gas Clean-Up Process.



Tail gas which exits the Claus unit is induced directly into the IFP reactor without cooling the gas. The reactor is essentially a packed column with a specially designed boot for collecting sulfur. Mixed alkali metal salts of an organic acid serve to catalyze the reaction which takes place in a high boiling polyglycol solvent above the melting point of sulfur--generally in the range 250-260°F. The metal salts form a complex with H₂S and SO₂ in the feed gas, which in turn reacts with more of the two sulfur compounds to form elemental sulfur and regenerate the catalyst complex. The sulfur coalesces and settles into the boot of the reactor, from which it is drawn as a molten product. In the reactor, the water of reaction is vaporized and carried out with the cleaned tail gas. To maintain the heat balance in the reactor, condensate is injected and vaporized together with the water of reaction. The solvent is generally a polyalkylene glycol, although alcohols, glycols, glycol ethers, or glycol polyethers may also be used.

3.2 Plant Operation During Emission Measurements

During the test period, process variables were monitored to assure that emissions measured would be representative of normal process operation. For the Stauffer Chemical test the monitored variables included:

- (1) tail gas H₂S and SO₂
- (2) SO₂ from incinerator stack

- (3) IFP inlet gas temperature
- (4) IFP inlet solvent temperature
- (5) IFP solvent flow rate
- (6) acid gas feed rate to Claus

As shown in the process data sheets (Tables 2, 3 and 4), the process was very steady during testing. The entire sulfur recovery system (Claus + IFP) ran at normal rates. For the three tests, sulfur recovery was calculated at 336 long tons per day (LT/D), versus normal operation of 341 LT/D. Plant design is 395 LT/D.

The emission results of the tests performed should be representative of IFP tail gas technology.

4.0 LOCATION OF SAMPLING POINTS

The sampling points selected for emission tests at Stauffer Chemical Company are shown in Figure 3.

Port B, downstream from the IFP Sulfur Removal Unit, was used to obtain samples for hydrogen sulfide (EPA Method 11), sulfur dioxide (EPA Method 6), nitrogen oxides (EPA Method 7), oxygen (paramagnetic), carbon monoxide (NDIR-EPA Method 10), and carbon dioxide (NDIR). Velocity traverses and moisture determinations were also made at this sampling point. The sampling port at this point consisted of a gate valve with a 1 1/4" opening.

The EPA sampling port consisted of a 2" gate valve located downstream from the IFP Unit and the Port B sampling location. Heated probes were installed at this point to extract sample gases for analysis by the Dupont Analyzer, the Meloy Analyzer and the gas chromatographic system.

Port A, as shown in Figure 3, was a 3" opening in the post-incinerator breeching. Moisture and hydrogen sulfide were determined at this sampling location.

The major problems presented by the location of the sampling points were the small size of Port B and the accumulation of moisture and elemental sulfur at the EPA sampling port. The problem at the EPA location was diminished by backflushing the sampling lines between samples to minimize the accumulation of elemental sulfur and moisture.

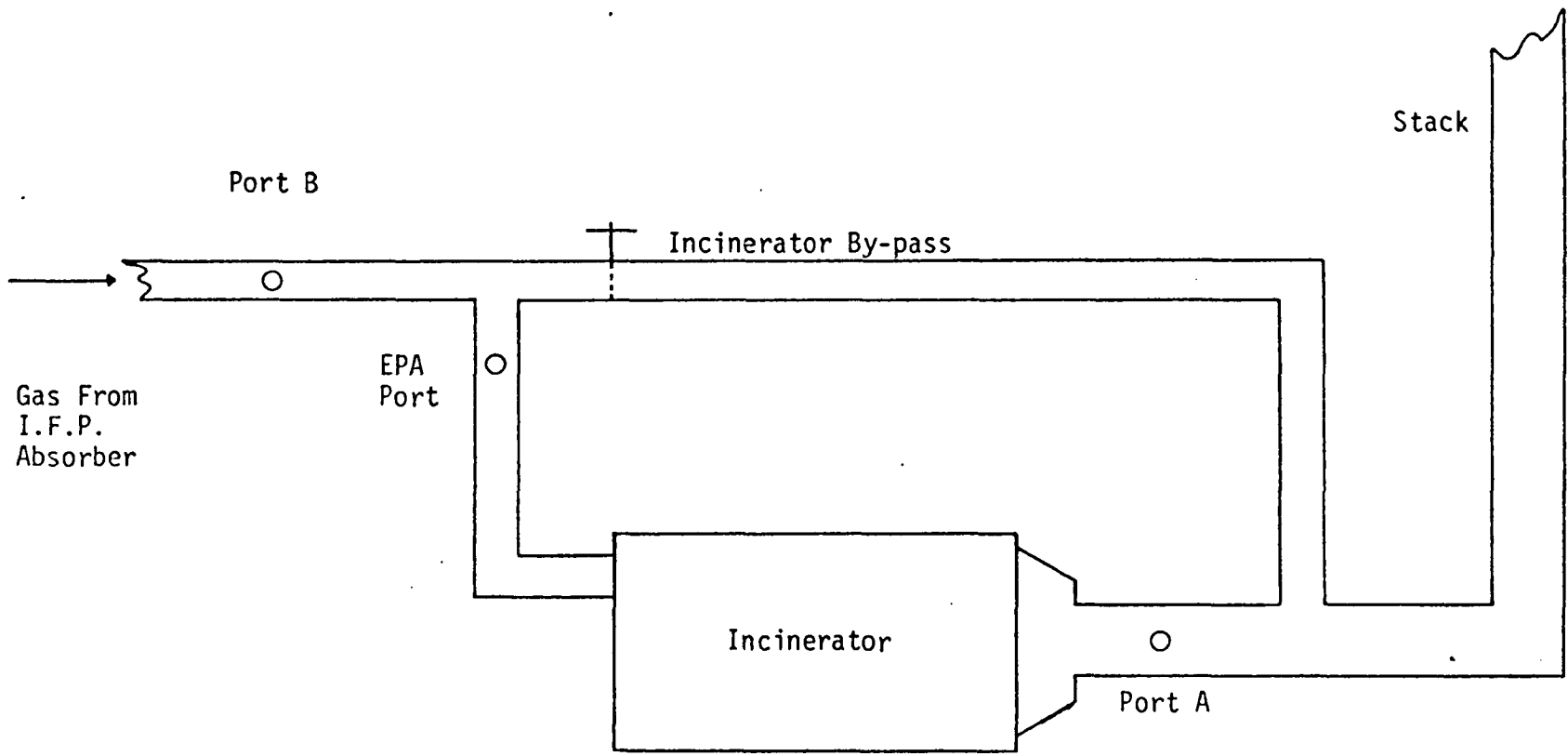


Figure 3. Location of Sampling Points

5.0 ANALYTICAL PROCEDURES

A brief summary of each procedure is presented in this section.

5.1 Sampling Procedures

Sample gases were extracted from the emission source for the determination of sulfur-containing compounds using a 3/16" FEP Teflon tubing sample line, heated and maintained at 121°C (250°F). This sample line terminated in the dilution manifold in the EPA mobile laboratory. Either direct or diluted sample could be withdrawn from the system as dictated by the analytical range of the instruments. The samples were simultaneously analyzed for total sulfur, carbon disulfide, sulfur dioxide, hydrogen sulfide and carbonyl sulfide. FEP Teflon parts or Teflon coated parts (including the sample pump heads) were used throughout the system to take advantage of the minimum reactivity of the Teflon to low level concentrations of sulfur compounds. Figure 4 is a diagram of the sample dilution system in the EPA mobile laboratory.

Sample gases were extracted from the emission source for the determination of carbon monoxide, carbon dioxide and oxygen using a three foot, 1/4" stainless steel sample probe with Swagelok connectors to polyethylene tubing. The sample passed through a dry impinger, immersed in an ice bath, before passing through the pump. A diagram of the sampling system and the sample dilution system is shown in Figure 5.

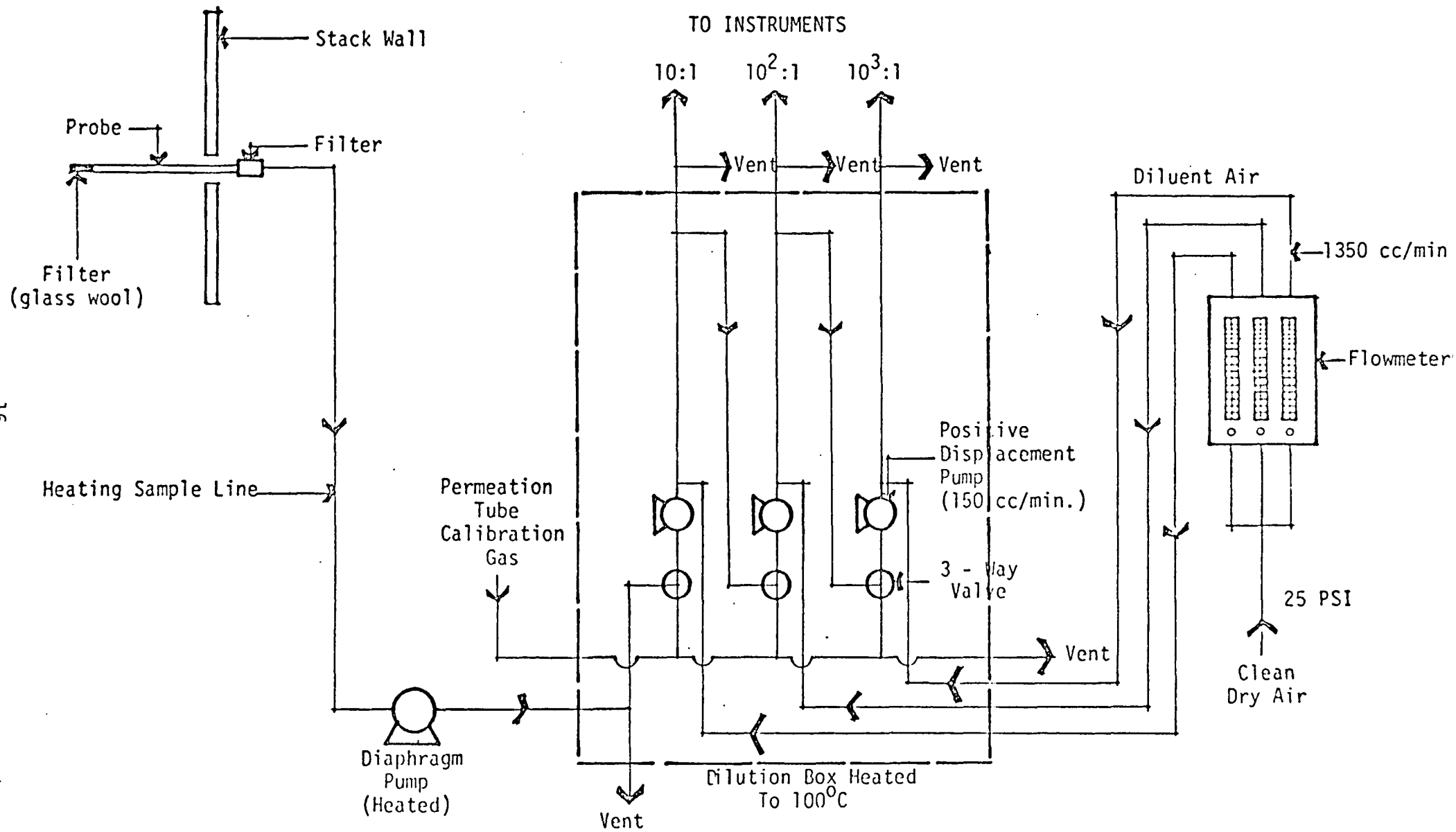


Figure 4.0. Sample Dilution System in EPA Mobile Laboratory

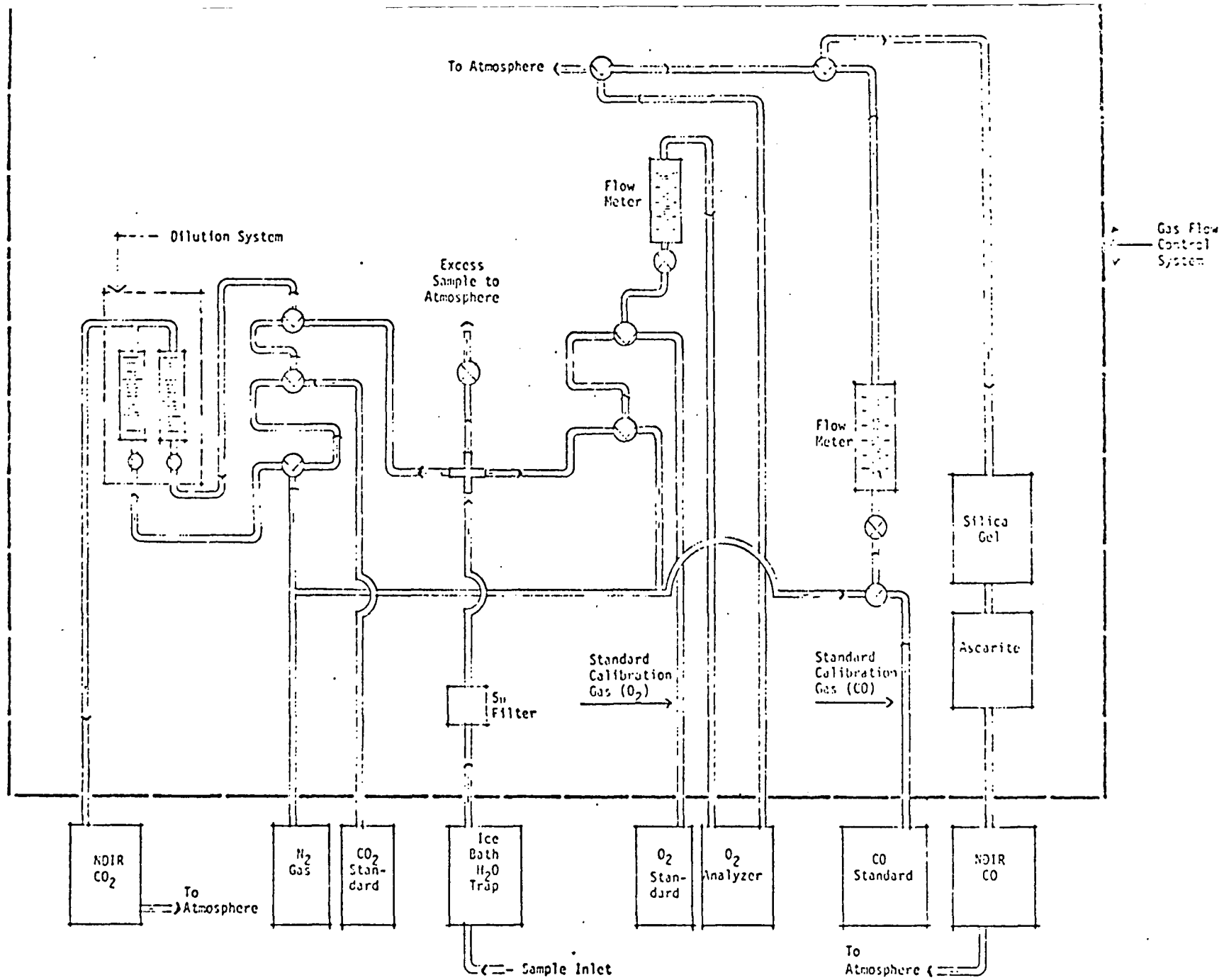


Figure 5.0. Schematic of sampling system for CO₂, CO, and O₂.

5.2 Analytical Procedures for Sulfur Compounds

Sulfur compounds were measured by three instrumental methods and two wet chemical methods in order to obtain a comparison of methods. The analytical methods for the various sulfur compounds are described in the following paragraphs.

5.2.1 Sulfur compounds by Gas Chromatography

Sulfur compounds, when introduced into a hydrogen-rich flame, produce strong luminescent emissions between 300 and 423 nm. Through the use of a narrow band optical filter that permits transmission at 394 nm, a flame photometric detector (FPD) can measure the chemiluminescent emissions produced by the S_2 species and can differentiate between sulfur containing and non-sulfur containing compounds. Through the use of a gas chromatograph (GC) equipped with the appropriate analytical columns, it is possible to separate and quantify the various sulfur compounds.

5.2.1.1 Applicability of Method

The compounds of interest in emissions from sulfur recovery systems are hydrogen sulfide (H_2S), carbon disulfide (CS_2), sulfur dioxide (SO_2) and carbonyl sulfide (COS).

The two GC/FPD systems available in the EPA mobile laboratory are capable of the separation and quantification of all of the compounds

of interest with the exception that COS and H₂S could not be determined simultaneously on any one system due to the relatively small difference in the retention times and the relatively large differences in concentration which results in the overlap of peaks. The difficulty presented by the H₂S - COS separation and determination was overcome through the use of a scrubbing system which effectively removes one component (H₂S) from the sample. Silver wool, which reacts readily with H₂S, was installed in one of the GC systems between the sample loop and the analytical column. Removal of the H₂S makes possible the determination of COS while the other system determines H₂S + COS. The difference in response observed between the two systems is used to calculate the H₂S concentration.

5.2.1.2 Instrumentation and Standards

GC/FPD System - The system provided in EPA mobile laboratory was assembled from components available from various commercial sources.

Sulfur Compound Permeation Tubes - Provided by EPA and gravimetrically calibrated by EPA personnel.

5.2.1.3 Analysis of Samples

The sample gas was extracted from the test source and diluted with clean, dry, sulfur-free air in the dilution system. Diluted sample was continuously flowed through the sample loop and injected at fifteen minute intervals throughout the test. The fifteen minute interval was selected due to the retention time of CS₂.

Responses obtained from each compound were compared to the standard curve for that component and the concentrations were determined. In the series of tests conducted, two GC/FPD systems were utilized. GC#1 was equipped with a scrubbing system and was used for a direct determination of COS concentration. GC#2 was used to determine SO₂, CS₂, and H₂S. H₂S was determined on GS#2 by subtracting the COS concentration found with GC#1 from the H₂S + COS concentration. The analytical methods used for the analysis of sulfur compounds in this series of tests were the same as described in an EPA preliminary draft method entitled "Semicontinuous Determination of Malodorous Reduced Sulfur Emissions From Stationary Sources".

5.2.2 Total Sulfur by the Meloy Analyzer

The detection system of this instrument is the same as for the gas chromatograph. The major difference between the two systems is that no analytical column is present to effect a separation of the various sulfur compounds. Another difference between the systems is that sulfur compounds in the sample gas are oxidized to sulfur dioxide by passage through a tube furnace maintained at 1500°C in the presence of excess oxygen before entering the Meloy Analyzer. The principal of the method and the applicability, however, are the same for the two systems.

5.2.2.1 Instrumentation and Standards

Detector System - Meloy Sulfur Analyzer, Model 160SA

Oxidation System - Lindberg Hevi - Duty Tube Furnace, Model 55035.

Calibration Standards - Permeation tubes provided by and gravimetrically calibrated by EPA personnel.

5.2.2.2 Analysis of Samples

The sample was obtained from the test source and diluted with clean, dry, sulfur-free air in the dilution system. Diluted sample was flowed through the tube furnace and into the Meloy Analyzer. A continuous readout of total sulfur concentration was displayed on a strip chart recorder. Comparison of the recorder response with the analytical curve obtained by plotting response versus concentration gave the total sulfur (as SO₂) concentration presence in the sample stream at any given point in time.

5.2.3 Total Sulfur and Sulfur Dioxide by the DuPont Analyzer

The detection system for this method is based on the absorption of ultraviolet radiation by sulfur species, in particular by sulfur dioxide. A sample is pumped into a cell where the sulfur dioxide is determined by a change in the intensity of ultraviolet radiation reaching the photocell. The cell is subsequently filled with oxygen and all the reduced and elemental sulfur species are converted to sulfur dioxide. After the oxidation step, the concentration is again determined by the change in the intensity of the ultraviolet radiation reaching the photocell.

5.2.3.1 Instrumentation and Standards

Detector System - DuPont Sulfur Analyzer

Calibration Standards - Provided by DuPont Personnel

5.2.3.2 Analysis of Samples

The sample was obtained from the test source as described above. DuPont personnel calibrated and monitored the instrument throughout the test period. The data were presented on a strip chart recorder and the responses of the individual samples (this method is semi-continuous) were compared to the standard calibration curve in order to obtain concentrations.

5.2.4 Titrimetric Method for the Determination of Sulfur Dioxide

Sulfur dioxide was oxidized to sulfate in the presence of hydrogen peroxide according to EPA Method 6 as outlined in the Federal Register, 36, No. 59, Part II, August 17, 1971. The sulfate which was formed and collected was subsequently titrated with a standardized solution of barium perchlorate in the presence of thorin indicator, and the sulfur dioxide concentration was calculated.

5.2.5 Titrimetric Method for the Determination of Hydrogen Sulfide

Hydrogen Sulfide was determined by EPA Method 11 as outlined in the Federal Register, 39, No. 47, Part II, March 8, 1974. The sample gas was extracted from the source and reacted with a basic solution containing cadmium ions. The cadmium sulfide formed was reacted with an excess of standard iodine solution and titrated with a standard solution of sodium thiosulfate. From the amount of iodine which reacted with the cadmium sulfide, the concentration of sulfide in the original sample was calculated.

5.3 Analytical Procedures for Carbon Monoxide, Carbon Dioxide and Oxygen
Carbon monoxide, carbon dioxide and oxygen were monitored continuously from the source during the three four-hour tests. The sample was obtained as described in section 5.1.

5.3.1 Instrumentation and Standards

Carbon Dioxide - Beckman Model 315A NDIR configured for 0-5% carbon dioxide.

Carbon Monoxide - Beckman Model 315B NDIR configured for 0-1000 ppm carbon monoxide.

Oxygen - Beckman Model F-3, paramagnetic oxygen analyzer capable of measuring 0-25% oxygen.

Standard Gases - Obtained from Matheson Gas Products, Inc., Cucamonga, California. All standards were either primary or certified and were analyzed by Matheson Gas Products, Inc. All standards consisted of the component of interest with the balance of the mixture as nitrogen.

5.3.2 Analysis of Samples

The sample gas was extracted from the test source and flowed through the instruments. In the case of the carbon monoxide the sample was diluted with clean, dry, carbon-monoxide free nitrogen in order to maintain the concentration within the operating range of the instrument. A schematic of the instrument flow system is presented as Figure 5.

Responses obtained from the instrument were displayed on a strip chart recorder and these responses were compared to the appropriate standard curve to obtain the concentrations of the different constituents.

EPA Method 10 as outlined in the Federal Register 39, No. 47 on Friday, March 8, 1974 was used as a guideline in the determination of carbon monoxide concentrations.

5.4 Analytical Procedure for Nitrogen Oxides

Nitrogen oxides were measured according to EPA Method 7 which appeared in Federal Register, 39, No. 47, 8 March 1974. A portion of the contents of the integrated bag sample was collected in an evacuated flask which contained sulfuric acid and hydrogen peroxide. After the oxides of nitrogen had been oxidized to nitrate, the nitrate was reacted with phenoldisulfonic acid and a spectrophotometric method was used to determine concentration.

5.5 Procedure for the Determination of Moisture

The moisture content was measured at a location downstream from the IFP Unit in accordance with EPA Method 4 which appeared in Federal Register, 36, No. 59, Part II, August 17, 1974.

5.6 Procedure for the Determination of Stack Gas Velocity

The stack gas velocity was determined at a location downstream from the IFP Unit after sample and velocity traverse points were located. EPA Method 1 and 2 as they appeared in Federal Register, 36, No. 59, Part II, August 17, 1974 were used as guidelines for these determinations.

5.7 Procedure for the Determination of Visible Emissions

EPA Method 9, as outlined in the Federal Register, 36, No. 247, Part II,

December 23, 1971 was used as the guideline in the determination of visible emissions. A certified observer was used to observe emissions from the sample source.

5.8 Carbon Dioxide and Oxygen by Orsat

A Carle gas chromatograph with a thermal conductivity detector was substituted for the Orsat Method in the determination of carbon dioxide and oxygen. This substitution was approved by the EPA Project Officer.