

OFFICE OF ENFORCEMENT

EPA-330/2-78-018

*SO<sub>2</sub> Emission Testing*  
*at the*  
*Bunker Hill Company*  
*Kellogg, Idaho*

(MAY 12-26, 1978)

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
DENVER, COLORADO  
AND  
REGION X, SEATTLE

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[May 12-26, 1978]

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National Enforcement Investigations Center - Denver  
and  
Region X - Seattle

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## I. INTRODUCTION

The Bunker Hill Company, a subsidiary of Gulf Resources, Incorporated, operates a lead smelter and zinc plant with annual production capacities of 127,000 and 100,000 m. tons (140,000 and 110,000 tons), respectively, at Kellogg, Idaho. On November 19, 1975, Region X of the Environmental Protection Agency (EPA) disapproved Regulation "S" of Idaho's State Implementation Plan (SIP), which limited sulfur dioxide ( $\text{SO}_2$ ) emissions from the Bunker Hill complex and substituted a replacement regulation<sup>1</sup>. The replacement regulation established  $\text{SO}_2$  concentration limits (6-hour averages) on the acid plants and the lead smelter main stack of 2,600 and 2,000 ppm, respectively. In addition, the regulation established a 7-day emission limitation of 617 m. tons (680 tons) of  $\text{SO}_2$  from the entire complex. Compliance with the concentration limits was to be determined either by continuous measurement systems or Method 8<sup>2</sup>. The continuous monitoring systems were required to meet performance specifications delineated in Appendix D of 40 CFR 52<sup>3</sup>.

The Bunker Hill Company challenged the disapproval of Regulation "S" and substitution of the above limitations with the result that the U.S. Court of Appeals for the Ninth Circuit remanded the replacement regulation to EPA for further consideration of its technological feasibility. Region X subsequently requested that the National Enforcement Investigations Center (NEIC) aid in the remand by conducting tests to determine  $\text{SO}_2$  concentrations and flow rates at the following locations [Figures 1 and 2], which are considered the major sources

of SO<sub>2</sub> emissions from the Bunker Hill complex:

1. Tailgas from Acid Plant 1 (Stations 48901 and 48907)
2. Tailgas from Acid Plant 2 (Stations 48902 and 48903)
3. Tailgas from Acid Plant 3 (Station 48904)
4. Sinter Machine weak stream (Station 48905)
5. Blast Furnace exhaust stream (Station 48906)\*

Reconnaissance inspections of the Bunker Hill complex were conducted by NEIC during December 12 and 13, 1977, and March 27 to 30, 1978, to evaluate sampling sites and to identify process data that would be collected in conjunction with testing. Region X subsequently informed the Company of necessary sampling site modifications and process data requirements [Appendix A]. An additional visit to the plant was made by NEIC on May 3, 1978 to verify completion of the requested site modifications.

Sampling at Bunker Hill commenced May 12 and ended May 26, 1978. With few exceptions, all measurements were conducted according to the procedures detailed in the Project Plan [Appendix B]. Sulfur dioxide concentrations were measured using both Method 8 and a Thermo Electron Corporation (TECO) pulsed-fluorescence analyzer. Only TECO data were collected at the blast furnace because of the adverse working environment at that location. Flow rates were measured using Method 2<sup>2</sup> with traversing according to Method 1<sup>2</sup>.

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\* Bunker Hill has two blast furnaces, but operate only 1 at a time.

## II. SUMMARY AND CONCLUSIONS

From May 12 to May 26, the National Enforcement Investigations Center (NEIC) measured gas stream flow rates and sulfur dioxide (SO<sub>2</sub>) concentrations at selected sites within the Bunker Hill lead smelter and zinc plant at Kellogg, Idaho. During this time, the lead smelter was operating near capacity, while the zinc plant was operating at about 70% of capacity. Operating conditions at both the smelter and zinc plant were reported to be normal; no unusual conditions (upsets or malfunctions) occurred which would have greatly affected SO<sub>2</sub> emissions.

Gas stream velocities were measured intermittently using Method 2 and traversing according to Method 1. Based on these velocities and the measured moisture content of the gas streams, the following gas flows were calculated for the outlets of the three acid plants and the Sinter Machine weak stream:

Location	Flow rate (dry @ standard conditions)*	
	Average	Range
Acid Plant 1	444 (15,700)	393-475 (13,900-16,800)
Acid Plant 2	408 (14,400)	297-450 (10,500-15,900)
Acid Plant 3	659 (23,300)	552-736 (19,500-26,000)
Sinter Machine weak stream	475 (16,800)	371-594 (13,100-21,000)

\* Standard conditions are 20°C (68°F) and 760 mm (29.92 in) Hg.



Sulfur dioxide concentration data were collected at the above locations by 1-hour Method 8 tests. Results of these tests, which characterized the average SO<sub>2</sub> concentrations in the acid plant tailgas streams and the sinter machine weak stream during the testing program, are summarized below:

Location	Hours	1-hour Average SO <sub>2</sub> Concentration (ppm)
Acid Plant 1	11	3,580
Acid Plant 2	10	2,270
Acid Plant 3	9	1,920
Sinter Machine weak stream	9	8,700

No method 8 or velocity data were collected at Blast Furnace 1 because of the adverse working environment caused by fugitive emissions from the furnace tapping and charging areas.

Sulfur dioxide concentrations were also measured at all locations using a TECO pulsed-fluorescence analyzer. The TECO measured SO<sub>2</sub> concentrations on a continuous basis for 2 to 9 hours/day and provided information regarding SO<sub>2</sub> fluctuations. The performance of the TECO analyzers at the acid plants and sinter machine were evaluated against the following performance specifications of 40 CFR 52, Appendix D:

Accuracy . . . . .	≤20% of reference mean value
Calibration Error . . . . .	≤5% of calibration gases
Zero Drift (2-hours)* . . . . .	≤2% of emission standard
Calibration Drift (2-hours)* . . . . .	≤2% of emission standard
Response Time . . . . .	15 min

\* The 2-hour zero and calibration drift specifications were evaluated using the proposed emission standard (2,600 ppm) at the acid plants and the average concentration measured by Method 8 (8,700 ppm) at the Sinter Machine.

These performance specifications were used as the criteria for assessing the quality of the TECO data and its relationship to Method 8. Because of time restrictions, the purpose of the testing was limited to characterization of the gas stream  $\text{SO}_2$  concentrations and not certification of the TECO analyzer. Only those specifications pertaining to data quality -- accuracy, calibration error, zero and calibration drift, and response time -- were, therefore, addressed. A conditioning period for the instrument was not provided and the 168-hour operational period specification was not addressed because these requirements measured instrument durability and not data quality. Because the monitors were only operated for periods of 8 to 10 hours at a time, the 24-hour drift specifications were also not addressed.

The TECO did satisfy the zero drift and response time specifications at all locations. The TECO was also able to meet the accuracy specifications of  $\pm 20\%$  at Acid Plant 3, but not at the other locations. The analyzer was unable to consistently meet the calibration error and calibration drift specifications; i.e., the calibration error specification was satisfied at two of the four locations.

The failure of the TECO analyzer to consistently meet the accuracy specification prevents the unqualified use of these data. However, for the purpose of additional data analysis, the Method 8/TECO comparisons can be used to bring the TECO data into closer agreement with the Method 8 results. For example, the average  $\text{SO}_2$  concentrations measured by Method 8 at Acid Plant 1 (3,220 ppm) was 15% greater than the average concentration measured by the TECO (2,800 ppm) during nine hours of concurrent sampling. The TECO data was, therefore, multiplied by 1.15 which, on the average, brings it into closer agreement with the Method 8 results. Similar adjustments were also made on the data for Acid Plants 2 and 3 and the Sinter Machine weak stream using the results of the concurrent Method 8/TECO sampling conducted at these locations.

TECO data, both unadjusted and adjusted, are summarized below:

Location	Hours of Data	Average SO <sub>2</sub> Concentration (ppm)	
		Unadjusted	Adjusted
Acid Plant 1	27	2,720	3,130
Acid Plant 2	29	2,800	2,100
Acid Plant 3	35	1,690	1,770
Sinter Machine			
weak stream	29.5	6,970	8,080
Blast Furnace 1	6	1,840 <sup>a</sup>	

a Since no Method 8 tests were conducted at the Blast Furnace 1 the TECO results could not be adjusted.

Adjusted TECO data were interpreted in terms of running 6-hour averages for those days during which at least 6 hours of concentration data were obtained. Six-hour average SO<sub>2</sub> concentrations in excess of the proposed 2,600 ppm limit were measured at Acid Plants 1 and 2 with maximum 6-hour concentrations of 4,400 and 3,500 ppm, respectively. The maximum 6-hour average SO<sub>2</sub> concentration at Acid Plant 3 was 2,080 ppm.

Average SO<sub>2</sub> emission rates based on NEIC measured flow rates and adjusted TECO SO<sub>2</sub> concentration data are listed below:

Location	Average SO <sub>2</sub> Emission Rate	
	m. tons (tons)/day	
Acid Plant 1	5.4	(5.9)
Acid Plant 2	3.3	(3.6)
Acid Plant 3	4.4	(4.9)
Sinter Machine		
weak stream	14.8	(16.2)

No emission rate was calculated for the blast furnace because no gas flow rates were measured for this service.

Company flow data was extracted from the operating logs for Acid Plants 1 through 3 for the period of May 12 through 25 and the average for each sampling period was compared (after adjustment for SO<sub>2</sub> content) to NEIC-measured flow data for the same period. The comparison indicated that Company flow measurements at Acid Plant 2 averaged approximately 7.0% low compared to the NEIC data, while Company measurements at Acid Plants 1 and 3 were higher by 24% and 30%, respectively.

The NEIC flow data were based on average gas velocities measured using Method 2 and traversing by Method 1 and have an expected accuracy of approximately  $\pm 10\%$ . The accuracy of Company flow data is unknown, but the use of single point velocity measurements makes this data questionable.

Both adjusted TECO data and concurrent Reich test results were used to calculate a daily average SO<sub>2</sub> concentration for each acid plant tailgas stream which reflected a 2 to 11 hour sampling period. The overall average SO<sub>2</sub> concentrations, based on TECO and Reich test results, agreed to within 17% for all three acid plants. Less agreement was noted when the average concentrations for a single day were considered. No direct comparison was made between Method 8 and Reich test results because of the difficulty associated with identifying concurrent pairs of data.

Company measured SO<sub>2</sub> concentrations for the Sinter Machine weak stream were in wide disagreement with NEIC measurements. During testing at this location from May 13 to 17, the maximum Company measured SO<sub>2</sub> concentrations was 3,500 ppm, while the NEIC SO<sub>2</sub> concentrations data (adjusted TECO) averaged 8,080 ppm.

### III. PROCESS AND SO<sub>2</sub> EMISSION SOURCE DESCRIPTION

The Bunker Hill facilities in Kellogg, Idaho, consist of a lead smelter, a zinc plant, and a phosphoric acid-ammonium phosphate fertilizer plant jointly owned with Stauffer Chemical Company. The phosphate fertilizer complex uses some of the sulfuric acid produced by the three acid plants operated as SO<sub>2</sub> controls at the lead smelter and zinc plant. Byproducts recovered from the metallurgical facilities include silver, gold, copper, cadmium, antimony, zinc oxide and sulfuric acid.

#### LEAD SMELTER

The Bunker Hill smelter [Figure 1] is a custom smelter; it handles concentrate from several outside sources, as well as those produced by Company-owned mines. As many as 30 different concentrates are handled during a year. Production capacity is reported to be 127,000 m. tons (140,000 tons)/year of metallic lead.

Lead concentrates are received primarily by rail and unloaded to receiving bins. Individual concentrates are then blended and sent to the bedding plant for preparation of a charge consisting of lead concentrates, lime and silica fluxes and recycled materials from the smelter and zinc plant. After a bed of material has been brought within specifications, it is removed from the bedding plant, mixed with recycled sinter, pelletized and sent to a sintering machine.

The sintering process removes sulfur by oxidation and allows agglomeration of the charge. In the first stage of sintering, a high strength (>5%) SO<sub>2</sub> gas stream is produced which is sent to a sulfuric



acid plant (No. 3). The conventional Lurgi updraft sinter machine used by Bunker Hill also produces a weak ( $\sim 1\%$ )  $\text{SO}_2$  stream which is blended with the exhaust gases from the blast furnaces and lead refining area, treated for particulate removal (baghouse) and sent to the smelter stack.

Material leaving the Sinter Machine is separated into coarse and fine fractions with the oversized material sent to a blast furnace and the undersized material recycled to the Sinter Machine. Bunker Hill has two oxygen-enriched blast furnaces (Nos. 1 and 3) but operates only one at a time. Lead sinter and coke are fed to the blast furnace in approximately a 9:1 ratio. The lead oxide ( $\text{PbO}$ ) in the sinter is reduced to an impure metallic form called bullion which is tapped from the furnace and sent through a series of purification steps -- drossing, softening, degolding, desilvering and dezincing. The end product of these refining steps is pure lead. Byproducts recovered during the purification steps include copper, antimonial lead (hard lead), silver, and dore metal (silver-gold alloy). The blast furnace slag is sent to the zinc fuming furnace where it is treated to recover zinc and lead oxides.

Major process sources of  $\text{SO}_2$  emissions from the lead smelter are the weak and strong gas streams from the Sinter Machine and the blast furnace exhaust. The weak  $\text{SO}_2$  stream from the Sinter Machine, the blast furnace gas stream and assorted exhaust streams from the lead refinery are treated for particulate removal in the main baghouse and then emitted to the atmosphere from the 217 m (715 ft) smelter stack. The strong  $\text{SO}_2$  stream from the Sinter Machine is treated in a separate baghouse and is then sent to a 270 m. tons (300 tons)/day single-contact acid plant (No. 3).

Acid Plant 3 was designed and built by Monsanto and began operation in 1972. It was designed to handle gas flows up to  $910 \text{ m}^3$

(32,000 scf)/min with an inlet  $\text{SO}_2$  concentration of 5%. Designed conversion efficiency for the acid plant was 97.5%. Tailgas from the acid plant is combined with the gas stream from the main baghouse and vented to the smelter stack.

Other possible sources of  $\text{SO}_2$  emissions from the lead smelter include the exhaust from the zinc fuming furnace, the silver refinery, the lead softening furnace, the tapping floor ventilation and the dross reverb furnace. The zinc fuming furnace emissions are treated by a baghouse and then emitted to the atmosphere from the zinc fuming furnace stack. Exhaust gases from the silver refinery, the lead softening furnace and the tapping floor are combined with the blast furnace gases prior to the main baghouse [Figure 1] while the dross reverb gases are treated in a separate baghouse, and then combined with the above streams after the main baghouse. All of these streams vent to the smelter stack.

### ZINC PLANT

The zinc plant [Figure 2] treats concentrates purchased from outside sources as well as concentrates from Company-owned mines. Production capacity has been reported as 100,000 m. tons (110,000 tons)/year. Zinc concentrates are received primarily by rail and are unloaded to one of 14 receiving bins at the zinc plant. Each bin normally holds a separate concentrate. Processing begins with a sulfuric acid leach of those zinc concentrates (~33% of total) containing sufficient dolomitic material (magnesium-calcium carbonate) to require magnesium or calcium removal before zinc recovery. The concentrates (both treated and untreated) are then dried, blended and sent to the roasters to remove sulfur prior to the electrolytic reduction process.



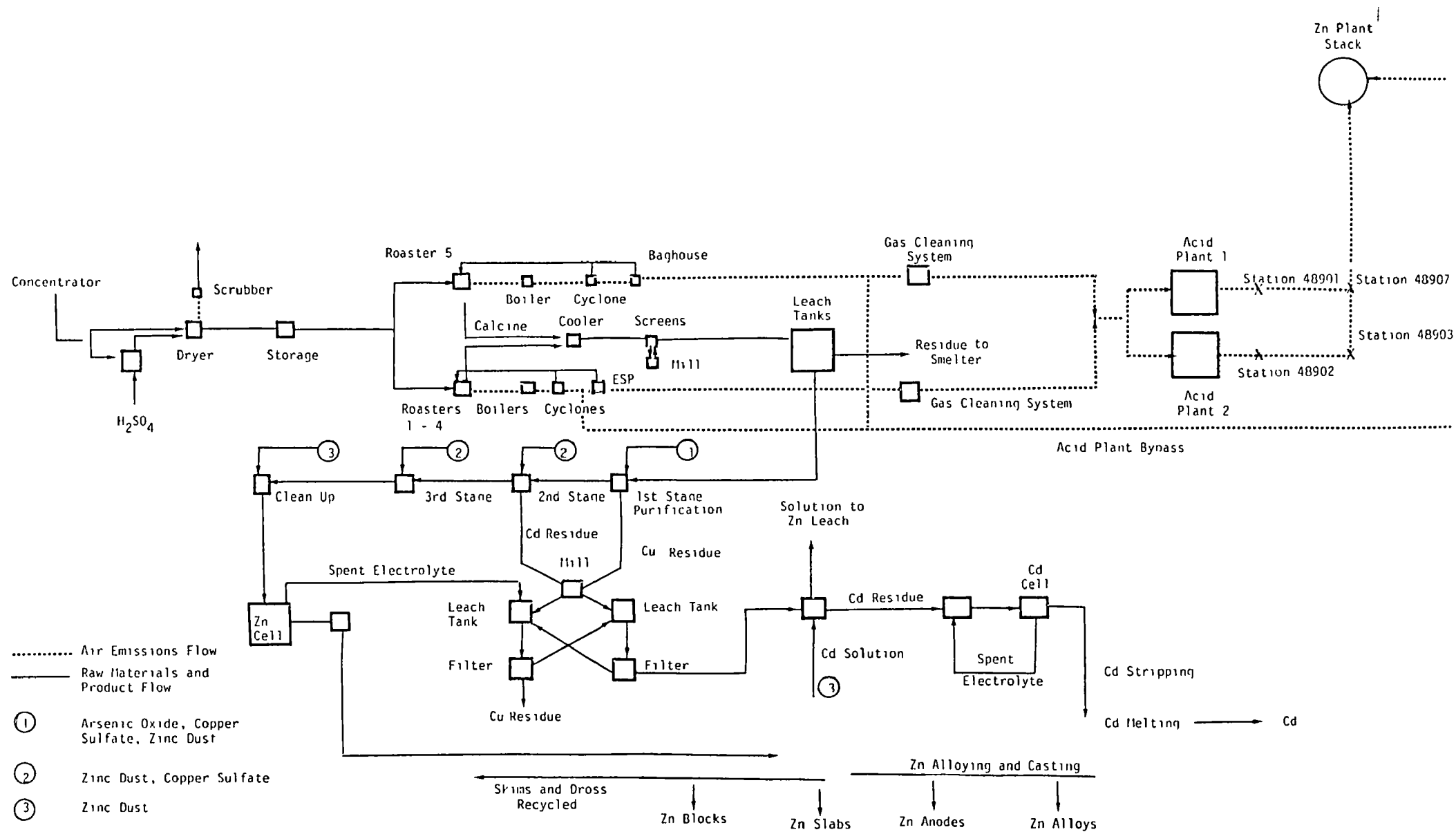


Figure 2 Simplified Process Flow Sheet of Zinc Plant  
Bunker Hill Company  
Kellough, Idaho

Bunker Hill has five flash roasters (Nos. 1-5) but reportedly operates a maximum of four at any one time. Roaster 5 is the largest with a rated capacity of 320 m. tons (350 tons)/day. Each of Roasters 1-4 has a rated capacity of 110 m. tons (120 tons)/day. Exhaust gases from the roasters, containing approximately 6%  $\text{SO}_2$ , are normally sent to two sulfuric acid plants (Nos. 1 and 2).

Calcine from the roasters is cooled and then leached with spent electrolyte from the zinc electrolytic cells. Residue from the leach tanks is sent to the lead smelter for recovery of the lead, gold and silver content while the solution, which contains the soluble zinc, is sent through a series of purification steps.

Purification consists of the sequential addition of arsenic oxide, copper sulfate and/or zinc dust to the zinc sulfate solution to precipitate cadmium, cobalt, nickel, copper and arsenic impurities. Following each purification step, the solution is filtered and the filtrates are either sent to the cadmium plant for recovery of cadmium and copper or recycled to the first stage of the purification sequence.

The purified solution is pumped to the cell room where zinc metal is recovered by electrolysis on aluminum cathodes. The cathodes are removed from the cells, and the zinc is stripped and sent to melting furnaces for casting.

The most significant process sources of  $\text{SO}_2$  emissions at the zinc plant are the offgases from the five flash roasters. The gas stream from Roasters 1-4 are passed through waste heat boilers for heat recovery, combined and sent to an electrostatic precipitator (ESP) for particulate removal. The gas stream which exits the ESP is sent to a wet cleaning system (Peabody scrubber and two mist precipitators), combined with the cleaned gases from Roaster 5 and sent to Acid Plants 1 and 2, operated in parallel.

The exhaust gases from Roaster 5 undergo treatment similar to that described for Roasters 1-4; they pass through a waste heat boiler, a baghouse and a wet gas cleaning system (Peabody scrubber and mist precipitator) prior to mixing with the gases from the other roasters. Any of the roaster gas streams can bypass the acid plants on the way to the main stack.

Acid Plant 1 was installed in 1954 by Monsanto. It is a single-contact acid plant designed for a flow rate of  $620 \text{ m}^3$  (24,000 scf)/min and an inlet  $\text{SO}_2$  concentration of 7.0%. Designed conversion efficiency at the above operating conditions is 95%. Acid Plant 2, also single-contact, was designed and built by Chemico and started operation in 1964. The design flow rate and inlet  $\text{SO}_2$  concentration are  $710 \text{ m}^3$  (25,000 scf)/min and 7.5%, respectively. Designed conversion efficiency is 97.5%. Tailgases from the two acid plants are combined and sent to the 185 m (610 ft) zinc plant stack. The tailgases can also be vented to the atmosphere directly from the absorption towers.

#### IV. TESTING AND PROCESS OBSERVATION PROCEDURES

A copy of the Project Plan [Appendix B] was provided to the Bunker Hill Company prior to the time NEIC entered the plant. During the testing program, Bunker Hill was provided all pilot tube, dry gas meter and orifice calibration data; span gas concentrations; and copies of all data collected by NEIC. Aliquots of all Method 8 samples and blanks were provided to Alsid, Snowden and Associates, a consulting firm retained by Bunker Hill. Alsid, Snowden and Associates was also provided access to all sampling locations and field laboratories.

##### NEIC SAMPLING METHODOLOGY

Sulfur dioxide concentrations were measured by both Method 8 and TECO pulsed-fluorescent analyzers. Method 8 was used to measure  $\text{SO}_2$  concentrations at all locations except Blast Furnace 1. No Method 8 tests were conducted at the blast furnace because of the adverse working environment at this site caused by fugitive emissions from the tapping and charging areas.

The purposes of the Method 8 testing were to characterize the  $\text{SO}_2$  concentrations of the gas streams by the reference method and to provide a measurement of the relative accuracy of the TECO data. Nine Method 8 tests were required at each site to allow an evaluation of the relative accuracy of the TECO; that is, the results of the Method 8 test were compared to the average  $\text{SO}_2$  concentrations measured by the TECO over the same 1-hour periods.

Since only SO<sub>2</sub> concentrations were to be measured by the Method 8 tests, it was not necessary to perform isokinetic sampling. Sampling was conducted at a constant rate of approximately 0.014 m<sup>3</sup> (0.50 ft<sup>3</sup>)/min at the center point of the duct.

The Scientific Glass, Inc. Model AP 5000 sampling train [Appendix C] was used for all Method 8 sampling and was arranged as follows:

Stainless steel (316) nozzle

Glass-lined probe

First impinger - Greenburg-Smith with 100 ml of 80%  
isopropanol solution

Glass fiber filter (5.1 cm diameter)

Second Impinger - modified Greenburg-Smith with 100 ml of 5%\*  
hydrogen peroxide solution

Third Impinger - Greenburg-Smith with 100 ml of 5%\* hydrogen  
peroxide solution

Fourth Impinger - modified Greenburg-Smith with approximately  
200 grams of silica gel.

Sample recovery and cleanup was according to the procedure listed below:

1. All impingers were weighed to determine the amount of moisture collected, and the contents of Impingers 1 and 4 were then discarded; as was the 5.1 cm filter.
2. Contents of Impingers 2 and 3 were transferred to a 1,000 ml graduated cylinder. The impingers and all connecting glassware between the filter holder and the silica gel (fourth) impinger were washed with deionized distilled

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\* 15% hydrogen peroxide solution was used at the Sinter Machine weak stream.

water and this washwater was added to the graduated cylinder. Deionized distilled water was then added to bring the volume to 1,000 ml. This sample was transferred to a 1,000 ml polypropylene container\* and the container was sealed. Following completion of a day's testing, the container was opened and an aliquot provided to representatives of the Bunker Hill Company. The container was then resealed, the liquid level in the container was marked, and the sample was stored for shipment to Denver.

3. The nozzle, probe, first impinger and all connecting glassware between the probe and the filter were washed with 80% isopropanol and the washings discharged.

All Method 8 samples were returned to the NEIC laboratories for analyses according to the procedures described in Method 8. Sample Chain-of-Custody was maintained at all times and sample blanks were obtained for the hydrogen peroxide and distilled water used during the testing [Appendix D]. All peroxide-distilled water samples saved as blanks were first used as a second wash for Impingers 2 and 3 of the Method 8 train. As noted previously, NEIC furnished an aliquot of all samples and blanks to Alsid, Snowden and Associates, a consulting firm retained by Bunker Hill.

TECO analyzers [Appendix E] were also used to measure  $\text{SO}_2$  concentrations at all locations. A gas sample was continuously removed from the duct of interest, the sample was mixed with clean dry air at a predetermined ratio, and the diluted sample was sent to the analyzer [Figure 3]. The purpose of diluting the sample prior to analysis was threefold:

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\* Two 500 ml containers were used for some samples at acid plants 1 and 2 (Stations 48901 and 48902).

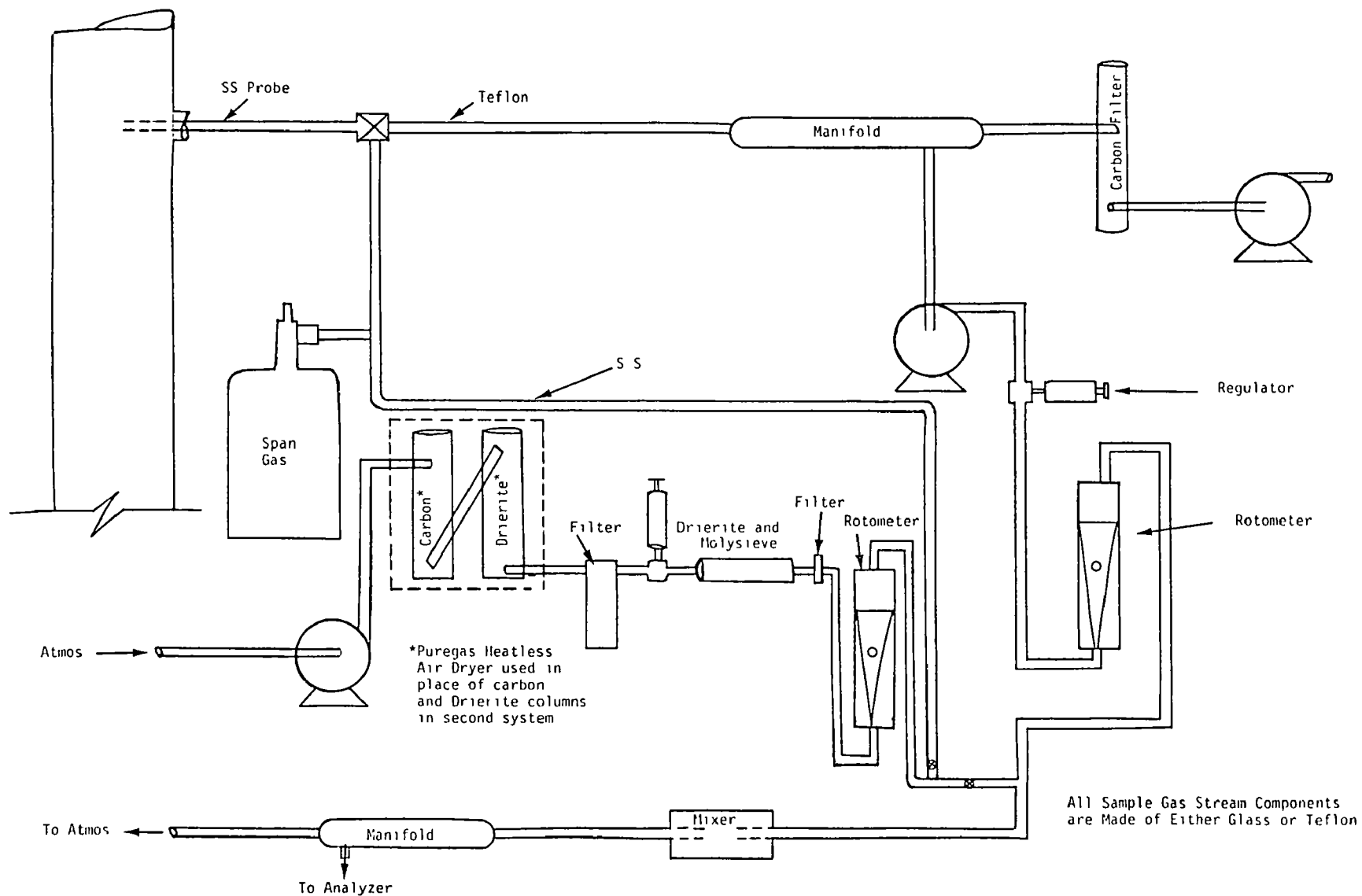


Figure 3 Sampling and Dilution System for Continuous Monitor  
Bunker Hill Company  
Kellogg, Idaho

1. Reduce the moisture content of the gas sent to the analyzer, thereby lowering the dewpoint and avoiding condensation.
2. Provide a sample which is essentially  $\text{SO}_2$  in the air (i.e., ~0%  $\text{CO}_2$  and 21%  $\text{O}_2$ ) so that the quenching effect\* of  $\text{CO}_2$  is eliminated and that of  $\text{O}_2$  can be compensated for during calibration.
3. Bring the  $\text{SO}_2$  concentration of the sample within the upper limit of the analyzer (5,000 ppm maximum).

The TECO analyzer measures the fluorescence excited at the 2100<sup>0</sup>Å (ultraviolet) region of the  $\text{SO}_2$  spectrum. In the absence of interfering gases ( $\text{CO}_2$  and  $\text{O}_2$ ), the fluorescence is linearly proportional to the concentration of  $\text{SO}_2$ .<sup>4</sup> Both carbon dioxide and oxygen reduce the fluorescent signal by quenching the excited  $\text{SO}_2$  molecule, but this was compensated for by diluting the sample and using a  $\text{SO}_2$  in air calibration gas.

At the acid plant and Sinter Machine sites, the TECO analyzers were evaluated against the following performance specification of 40 CFR 52, Appendix D:

Parameter	Specification
Accuracy <sup>a</sup>	< 20% of reference mean value
Calibration Error <sup>a</sup>	≤ 5% of each (50% and 90% span) calibration gas mixture
Zero Drift (2-hours) <sup>a</sup>	< 2% of emission standard
Calibration Drift (2-hours) <sup>a</sup>	≤ 2% of emission standard
Response Time	15 minutes maximum

a Expressed as sum of absolute mean value plus 95% confidence interval of a series of tests.

These performance specifications were used as the criteria for assessing the quality of the TECO data and its relationship to Method 8. Because of time restrictions, the purpose of the testing was limited to

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\* Excited  $\text{SO}_2$  molecules can transfer energy to  $\text{CO}_2$  and  $\text{O}_2$  molecules by collision (quenching), thereby reducing the intensity of the fluorescence.



characterization of the gas stream  $\text{SO}_2$  concentration and not certification of the TECO analyzer. Only those specifications pertaining to data quality -- accuracy, calibration error, zero and calibration drift, and response time -- were addressed. A conditioning period for the instrument was not provided, and the 168-hour operational period specification was not addressed because these requirements measured instrument durability and not data quality.

The promulgated acid plant emission standard of 2,600 ppm was used to evaluate 2-hour zero and calibration drift specifications at the three acid plant sites; 8700 ppm, the average  $\text{SO}_2$  concentration measured by Method 8, was used for the Sinter Machine weak stream. Because the monitors were only operated for periods of 8 to 10 hours at a time, the 24-hour drift specifications were not addressed in the sampling program.

In conjunction with the Method 8 and continuous  $\text{SO}_2$  monitoring, gas flow rates were determined throughout the day, initially at approximately 1-hour and later at 2-hour intervals. Method 2 (using an isolated S-type pitot tube) with traversing according to Method 1 was used to measure gas velocities. Temperature measurements were collected with a thermocouple-potentiometer arrangement at the same time as the velocity measurements.

A gas sample was collected (Method 3<sup>2</sup>, grab) concurrent with the velocity measurements and analyzed with Fyrite analyzers (approximately every third sample was analyzed using an Orsat analyzer) for calculation of gas molecular weight. Moisture contents of the gas streams were determined from the Method 8 sampling train (weight gain of Impingers 1-4).

All pitot tubes, dry gas meters and orifices used during the survey were calibrated both prior to the start of the survey and at its completion [Appendix F]. Thermocouples used to measure stack gas

temperatures were checked against ASTM\* thermometers prior to the start of the survey and at its completion. The TECO analyzers were operated in accordance with the calibration and quality control procedures of 40 CFR 52, Appendix D. All span gases were analyzed by Method 6<sup>2</sup> procedures for SO<sub>2</sub> concentrations both when the survey began and at its completion [Appendix F]. The average measured concentration, which differed from the reported value by as much as 10%, was used for all calculations. In addition, a span check was performed at each site using a gas of unknown concentration.

### NEIC SAMPLING LOCATIONS

#### Acid Plant 1 (Stations 48901 and 48907)

Sulfur dioxide concentrations in the tailgas from Acid Plant 1 were measured at a sampling location (48901) in the 91 cm (36 in) diameter stainless steel duct which connects the absorption tower with the downcomer to the fiberglass-reinforced plastic (FRP) ductwork which leads to the stack [Figure 4]. Two 10 cm (4.0 in) diameter ports are located on one side of the duct, with a 0.46 m (1.5 ft) separation between the two ports. The distances between this sampling location and the nearest upstream and downstream flow disturbances are approximately 11 and 3.3 m (35 and 11 ft), respectively. The Method 8 train was operated at the upstream port, because of space limitations, while the TECO sample was collected from the downstream port. Sulfur dioxide concentrations were measured at the midpoint of the duct. Station 48901 was not used for velocity measurements because only one port was available at the Method 8 sampling location.

Velocity measurements were conducted at a sampling site (48907) in the downcomer section of ducting using two 10 cm (4.0 in) ports, with a 90° separation, located approximately 1.8 m (6.0 ft) downstream

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\* American Society for Testing and Materials

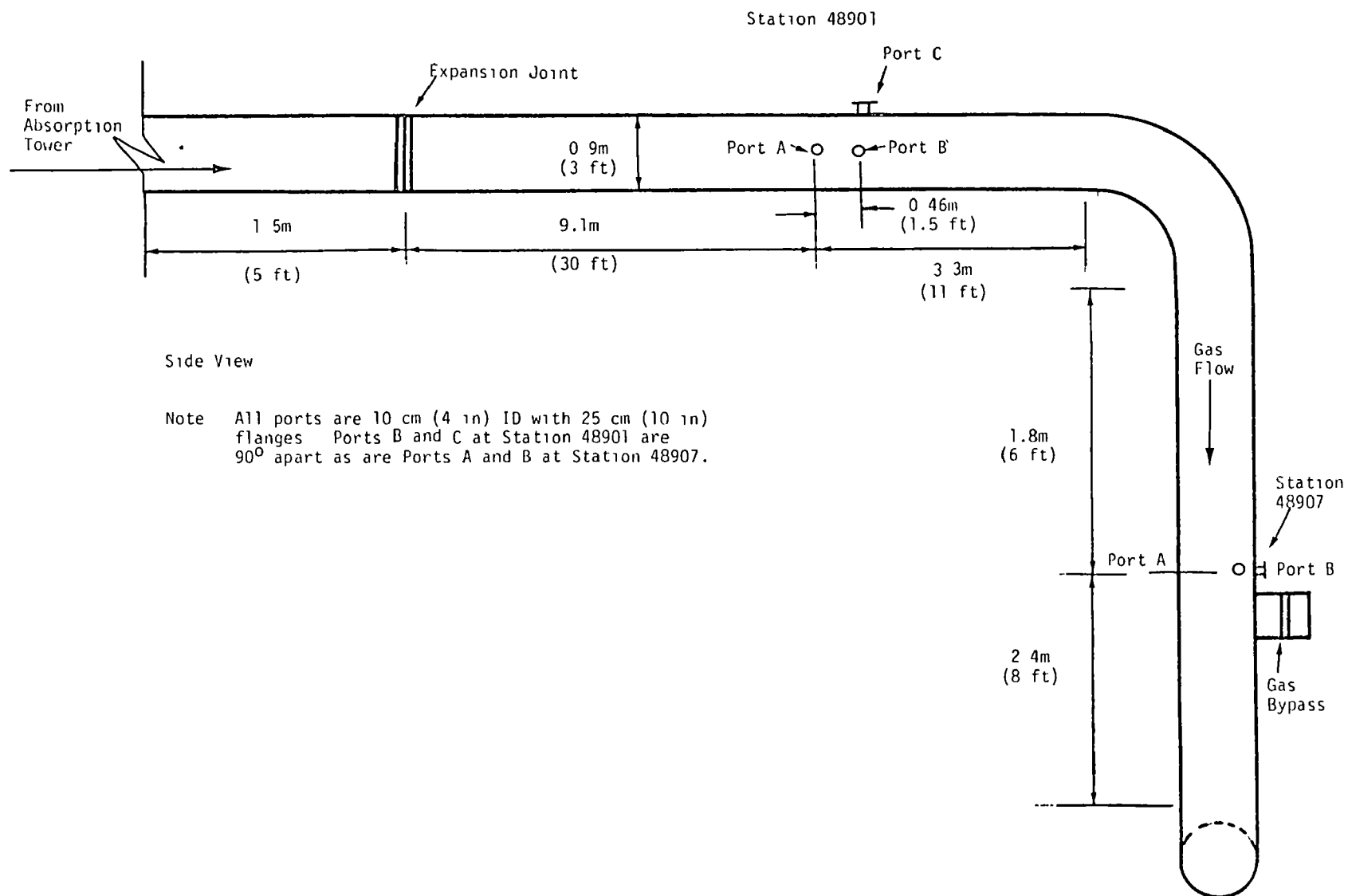


Figure 4. Sampling Locations at Acid Plant 1  
(Stations 48901 and 48907)  
Bunker Hill Company  
Kellogg, Idaho

and 2.4 m (8 ft) upstream of the nearest flow disturbances. Twenty-four sample points were used to measure average gas velocity. Although this site does not satisfy the minimum downstream separation required by Method 1, results of simultaneous velocity measurements conducted at Stations 48901\* and 48907 showed that, on the average, the velocity measured at Station 48907 was only 2.9% greater than that measured at Station 48901. Station 48907 was, therefore, deemed acceptable and used for all future measurements.

#### Acid Plant 2 (Stations 48902 and 48903)

A sampling site (48902) is located in the 107 cm (42 in) diameter horizontal stainless steel duct which connects the absorption tower with the downcomer to the FRP duct [Figure 5]. This site is similar to the one at Acid Plant 1; two 10 cm (4.0 in) sampling ports are installed on one side of the duct, with approximately a 0.61 m (2.0 ft) separation between them. This sampling location is less than 1.2 m (4.0 ft) downstream of a constriction in the ducting; therefore, no velocity measurements were collected at this site. Both ports were used to measure  $\text{SO}_2$  concentrations; a TECO was used to sample the downstream port while a Method 8 train was used at the upstream port. The Method 8 sampling was done at the upstream part because of space limitations at the downstream location. Sulfur dioxide concentrations were measured at the midpoint of the duct.

Two 10 cm (4.0 in) ports with a 90° separation are located in the downcomer to the FRP duct approximately 7.6 m (25 ft) downstream of Station 48902. This location (48903) is approximately 6.1 m (20 ft) downstream and 2.4 m (8.5 ft) upstream of the nearest flow disturbances. Twenty-four sample points were used to measure velocity.

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\* Station 48901 satisfied the requirements of Method 1

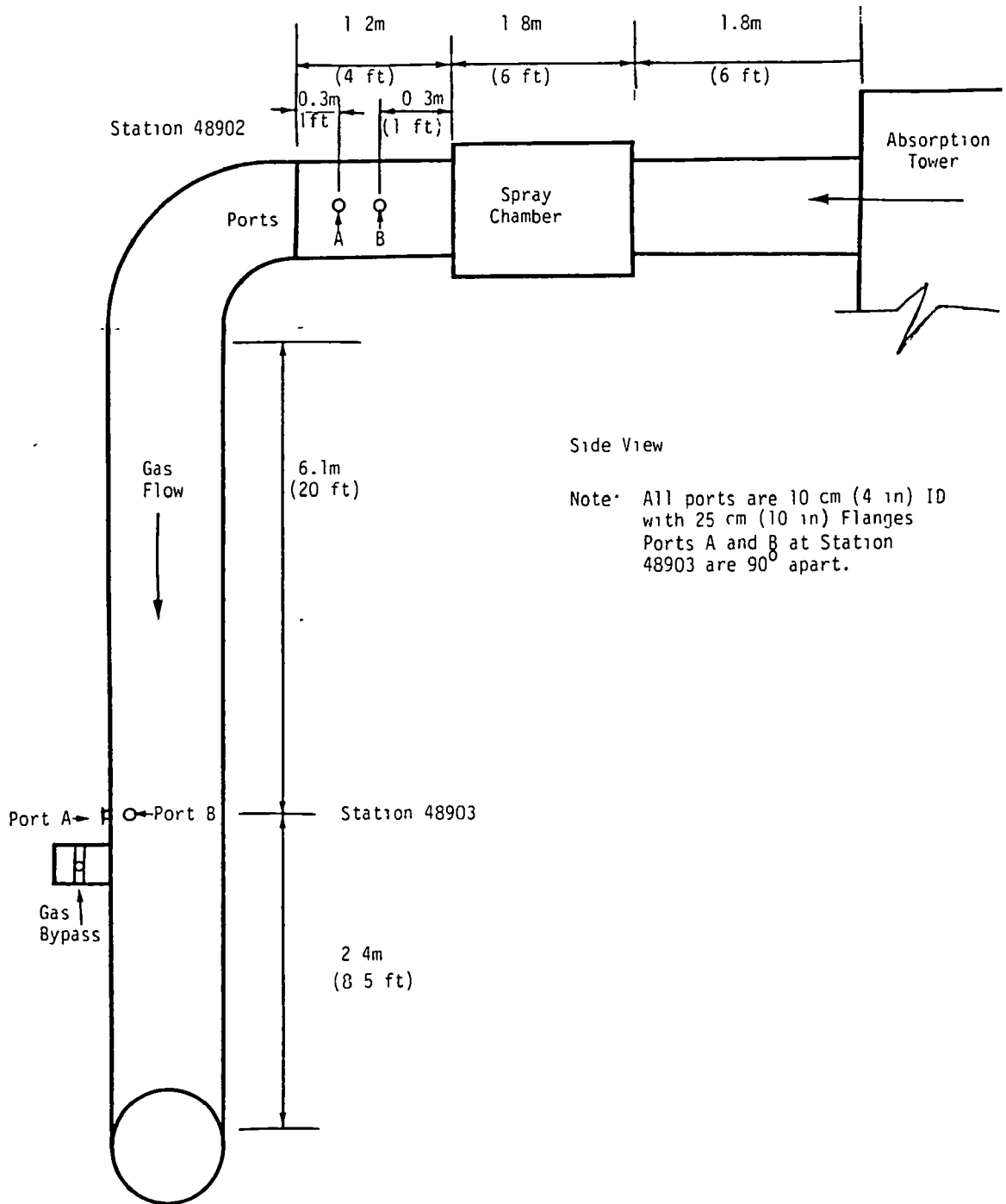


Figure 5 Sampling Locations at Acid Plant 2  
(Stations 48902 and 48903)  
Bunker Hill Company  
Kellogg, Idaho

### Acid Plant 3 (Station 48904)

A sampling site is located in the 140 cm (54 in) diameter FRP downcomer which connects the outlet from the acid plant to the downstream fan [Figure 6]. Two 10 cm (4.0 in) diameter ports (Ports B and D) are installed on the front side (with respect to the platform) of the duct, one port 0.61 m (2.0 ft) below the other. Two additional downstream ports (A and C) are installed at the same level as Port B, each at 45° to that port.

The sampling location is approximately 3.7 m (12 ft) and 7.6 m (25 ft), respectively, from the nearest downstream and upstream flow disturbances. The upstream port (Port D) was used for the TECO while Port B was used for the Method 8 sampling. The two remaining downstream ports (Ports A and C) were used for velocity measurements using 24 sample points.

### Sinter Machine Weak Stream (Station 48905)

The Sinter Machine weak stream was sampled about 3.0 m (10 ft) upstream of the point where it connects with the high-velocity flue leading to the main baghouse [Figure 7]. Two 10 cm (4.0 in) diameter ports (Ports A and B) are installed on the side and top of the 110 cm (42 in) diameter duct, about 3.0 m (10 ft) upstream of the connection with the high-velocity flue. This site is approximately 2.1 m (7.0 ft) and 3.0 m (10 ft), respectively, from the nearest upstream and downstream disturbances. Port A was used for Method 8 sampling, while both ports were used for velocity measurements using 28 points. A third 10 cm port (Port C) is located 0.70 m (2.4 ft) upstream of Port A. The TECO was installed in Port C.

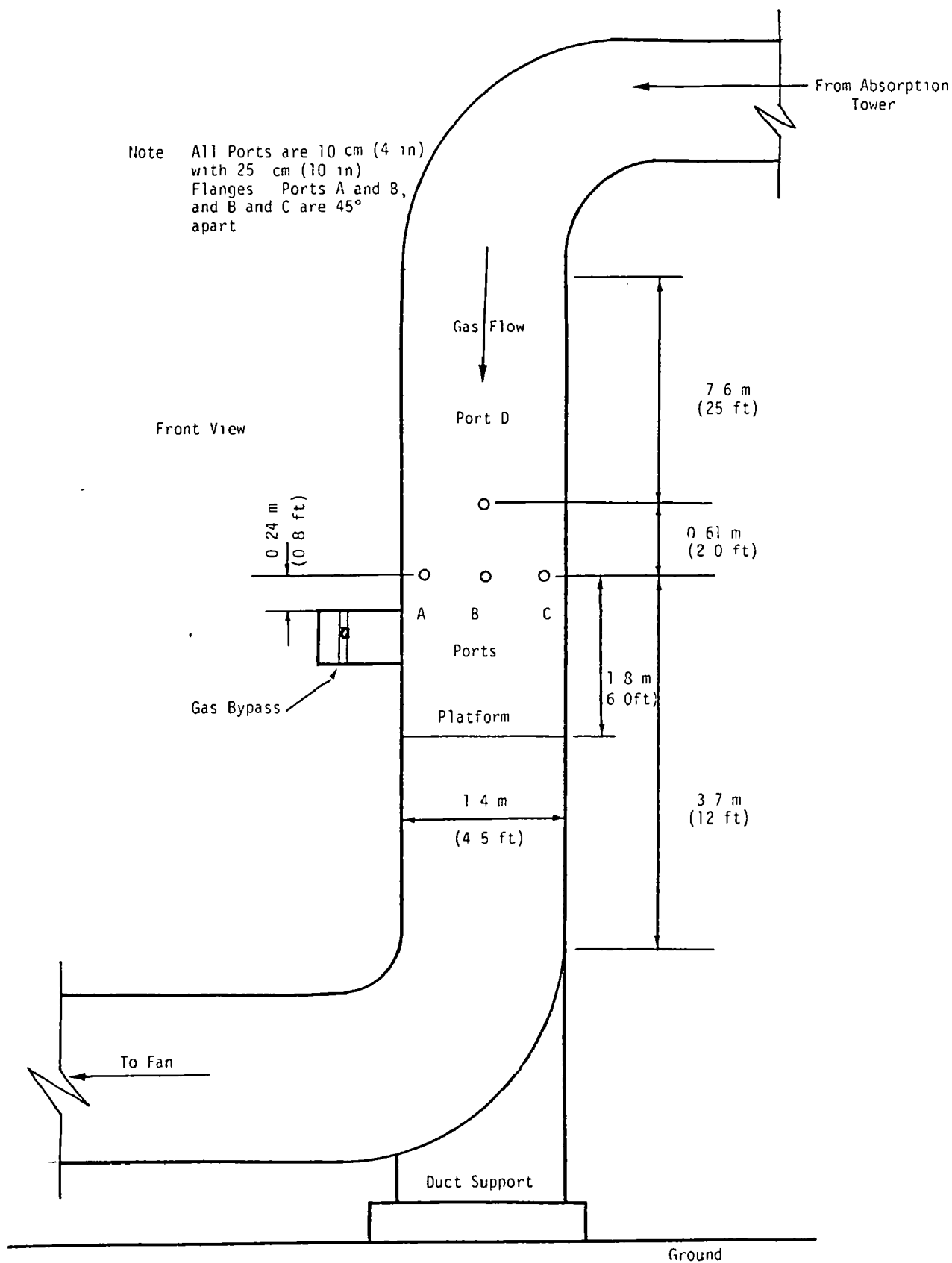


Figure 6 Sampling Location at Acid Plant 3 (Station 48904)  
Bunker Hill Company  
Kellogg, Idaho

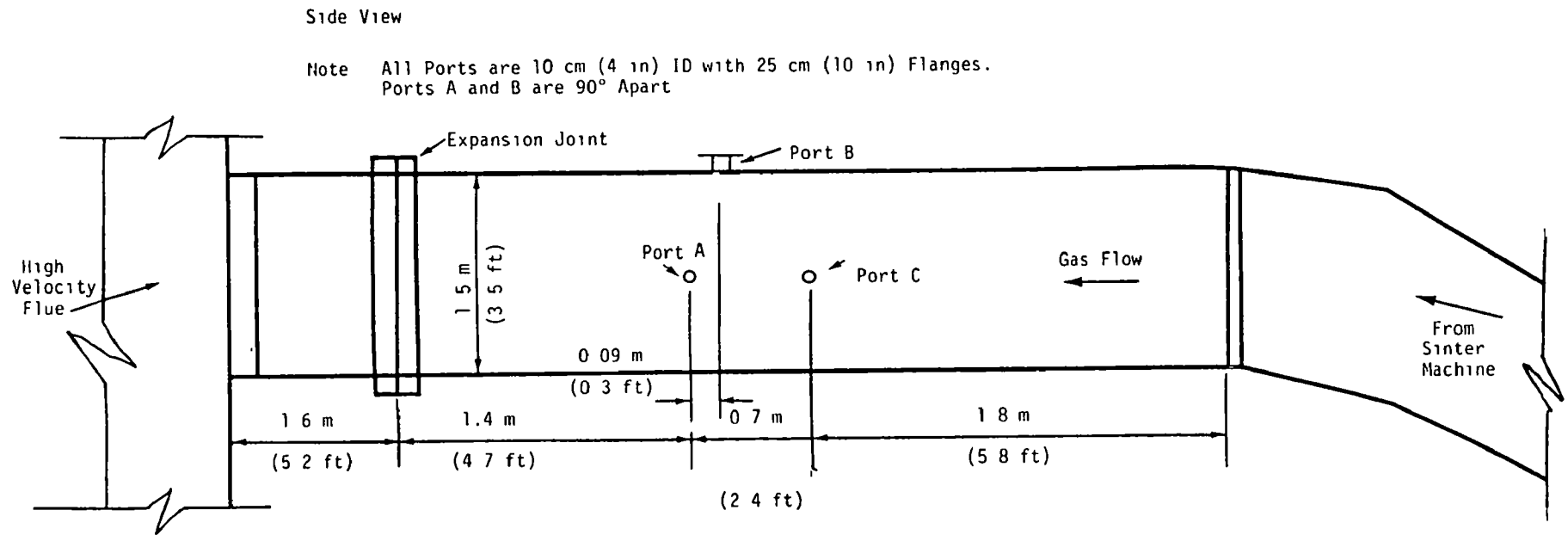


Figure 7 Sampling Location at Sinter Machine Weak  $\text{SO}_2$  Stream (Station 48905)  
Bunker Hill Company  
Kellogg, Idaho



### Blast Furnace 1 (Station 48906)

Blast Furnace 1 was sampled in the gooseneck duct which connects the furnace with the brick flue which leads to the high-velocity flue [Figure 8]. Five 10 cm (4.0 in) ports are installed on the top of the duct in a line perpendicular to the duct's axis, with an equal separation between ports. Sulfur dioxide concentrations were measured at approximately the middle of the duct using a TECO monitor installed in the center port. No velocity data was collected at this site because of the poor working environment.

### COMPANY SAMPLING METHODOLOGY AND LOCATIONS

Bunker Hill presently monitors both SO<sub>2</sub> concentration and gas flow rate at the following locations:

- Smelter main stack
- Zinc plant main stack
- Inlets to Acid Plants 1 through 3
- Roaster 5 exhaust

The instrumentation at the smelter and zinc plant main stacks is the same; a DuPont Model 460 (UV spectrophotometer) SO<sub>2</sub> monitor and an Annubar velocity sensor. Sulfur dioxide concentration and gas flow are measured at the 53 m (175 ft) level of the 217 m (715 ft) smelter stack and at the 71 m (200 ft) level of the 185 m (610 ft) zinc plant stack.

Inlet SO<sub>2</sub> concentrations to the acid plants are measured between the drying tower and the blower of each plant by both the Reich test\* (grab sample taken once/2 hrs) and a thermal conductivity monitor (continuous sample). Flow rates to the acid plants are measured at a location between the drying tower and the blower using either a pitot

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\* The Reich test, using a starch indicator, measures the volume of gas required to completely react with a known amount of iodide reagent.

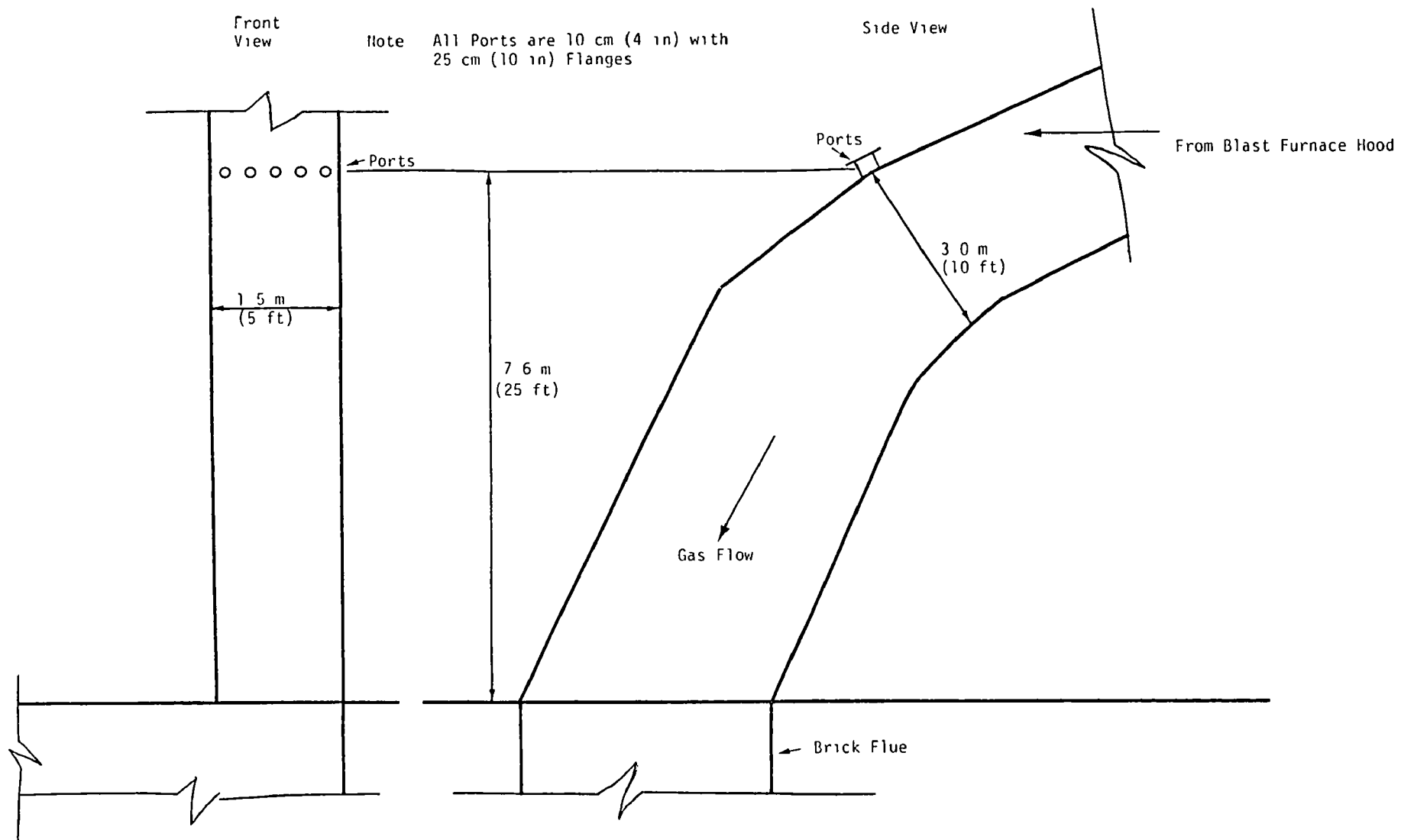


Figure 8 Sampling Location at Blast Furnace 1 (Station 48906)  
Bunker Hill Company  
Kellogg, Idaho

tube or pitot-tube-type device. More specific information regarding the thermal conductivity monitors and the velocity sensors was requested of the Company, but was not provided. No information was obtained regarding the flow and SO<sub>2</sub> instrumentation at Roaster 5.

Sulfur dioxide concentrations are also measured at the weak and strong streams off the Sinter Machine and the outlets of the acid plants. A DuPont Model 400 (UV spectrophotometer) measures the SO<sub>2</sub> concentration of the Sinter Machine strong stream at two locations; upstream of the spray chamber preceding the baghouse, and at the outlet of the No. 6 fan. The weak stream is also measured approximately 3.0 m (12 ft) off the Sinter Machine hood. The DuPont 400 monitors the three sites on a time-sharing basis, allocating a third to each site. Concentrations at the outlets of each acid plant are determined once-per-hour by the Reich test.

#### PROCESS OBSERVATION PROCEDURES

The NEIC collected the following logs, control sheets, etc., to characterize the operation of the Sinter Machine, blast furnace, zinc roasters and acid plants during the testing program:

##### Lead Smelter

1. Daily Operating Log Sheet (Acid Plant)
2. Sinter Plant Operation Process Control Sheet
3. Sinter Plant Process Control Sheet
4. Main Baghouse Operation Log
5. Lead Refinery Daily Report
6. Blast Furnace Process Control Sheet
7. Blast Furnace Schedule Control Sheet
8. Smelter Bedding Plant Work Sheet
9. Casting and Loading Schedule Control Sheet

10. Ore Preparation Schedule Control
11. Strip chart showing inlet SO<sub>2</sub> concentration at acid plant
12. Circular chart showing inlet gas flow at acid plant
13. Feederman Process Control Sheet
14. Circular chart showing SO<sub>2</sub> concentrations in gas streams from Sinter Machine
15. Shift Report, Smelter Division, Hi-Line Department
16. Strip chart showing SO<sub>2</sub> concentration and flow at smelter main stack<sup>\*</sup>

#### Zinc Plant

1. Daily Operating Report, No. 5 Roaster
2. Flash Roaster Daily Report (Roasters 1-4)
3. Daily Operating Log Sheets (Acid Plants No. 1 and 2)
4. Strip charts showing inlet SO<sub>2</sub> concentrations at acid plants
5. Materials Handling Sheet
6. Electrolytic Zinc Plant Pretreatment Sheet
7. Strip chart showing gas flow from No. 5 Roaster
8. Strip chart showing SO<sub>2</sub> concentration of exhaust from No. 5 Roaster
9. Strip chart showing SO<sub>2</sub> concentration and flow at zinc main stack<sup>\*</sup>

The Company provided copies of the preceding data only for those days during which NEIC was actually at a facility; lead smelter data were provided for the periods May 10 to 18 and May 26, while zinc plant data were provided for the period of May 20 to 25. All data were collected and retained by the Company until it was provided to NEIC in exchange for copies of NEIC test data, normally at the end of a test period.

During the testing program, an NEIC observer was allowed access (when accompanied by a designated Company representative) to the

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\* Strip chart shows SO<sub>2</sub> concentration and flow for both the smelter and zinc plant stack.

meteorological station and the following process control rooms:

Lead Smelter

1. Sinter Machine
2. Blast Furnace
3. Acid Plant 3

Zinc Plant

1. Acid Plants 1 and 2
2. Roaster 5
3. Roasters 1-4

Normally, 2 to 3 visits per day were made to the control rooms associated with the facility being tested, while one visit was made to the control rooms at the other facility. During these visits, the NEIC process observer: 1) reviewed the operating status of the process; 2) validated the content of the process control sheets and operating logs; 3) collected values for parameters not reported on the standard control sheets; and 4) became familiar with process operating and record keeping procedures. All questions regarding process operation, record keeping, etc., were directed to the Company's designated representative.

## V. TEST RESULTS

### SURVEY DATA

From 9 to 11 one-hour Method 8 tests [Appendix G] were conducted at each of the three acid plants and the Sinter Machine weak stream. The additional tests at Acid Plants 1 and 2\* were conducted because of suspected problems with the TECO analyzer during some Method 8 tests. Results of the Method 8 testing [Tables 1 through 4] are summarized below:

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Location	Hours Data	Method 8 Results <sup>a</sup>	
		SO <sub>2</sub> Concentration (ppm)	
		Average	Range
Acid Plant 1	11	3,580	1,680 - 6,840
Acid Plant 2	10	2,270	990 - 3,750
Acid Plant 3	9	1,920	1,440 - 3,260
Sinter Machine weak stream	9	8,700	3,870 - 15,300

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a One-hour average

Sulfur dioxide concentrations exhibited large fluctuations at all of the above sites with the results of individual one-hour tests varying by as much as 63% to 91% from the average concentration at a site.

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\* Ten Method 8 tests were conducted at Acid Plant 2 and eleven at Acid Plant 1.

Tables 1 and 2  
SUMMARY OF METHOD 8, SO<sub>2</sub> DATA  
BUNKER HILL COMPANY  
Kellogg, Idaho

Date	Start Time	Sample Volume <sup>a</sup> m <sup>3</sup>	SO <sub>2</sub> Collected mg	SO <sub>2</sub> Concentration ppmv
ACID PLANT 1 (STATION 48901)				
5/21	1208	0.792	7,580	3,590
	1605	0.800	9,570	4,510
5/22	0940	0.804	10,500	4,920
	1156	0.826	10,200	4,620
	1453	0.822	9,580	4,400
5/23	1013	0.817	3,820	1,760
	1310	0,849	4,800	2,120
	1612	0.820	4,580	2,100
5/24	1030	0.814	3,640	1,680
	1340	0.830	6,290	2,850
	1618	0.847	15,400	<u>6,840</u>
Avg.				3,580
ACID PLANT 2 (STATION 48902)				
5/21	1335	0.786	5,800	2,770
	1635	0.764	5,900	2,900
5/22	1053	0.809	7,010	3,250
	1311	0.766	4,720	2,310
	1638	0.822	3,310	1,510
5/23	1131	0.762	3,140	1,550
	1423	0.780	2,050	990
	1710	0.800	4,080	1,920
5/24	1034	0.824	3,990	1,820
	1415	0.811	7,990	<u>3,700</u>
Avg.				2,270

a Sample volume on dry basis at standard conditions of 20°C (68°F) and 760 mm (29.92 in) Hg.

Tables 3 and 4  
SUMMARY OF METHOD 8, SO<sub>2</sub> DATA  
BUNKER HILL COMPANY  
Kellogg, Idaho

Date	Start Time	Sample Volume <sup>a</sup> m <sup>3</sup>	SO <sub>2</sub> Collected mg	SO <sub>2</sub> Concentration ppmv
ACID PLANT 3 (STATION 48904)				
5/15	1146	0.822	4,190	1,920
	1550	0.809	7,030	3,260
5/16	1020	0.848	4,020	1,780
	1430	0.828	4,640	2,100
	1716	0.836	3,190	1,440
5/17	0943	0.832	3,440	1,550
	1158	0.844	5,170	2,300
	1418	0.830	3,340	1,510
	1717	0.854	3,300	<u>1,450</u>
Avg.				1,920
SINTER MACHINE WEAK STREAM (STATION 48905)				
5/15	1107	0.709	13,000	6,870
	1456	0.583	5,980	3,870
	1725	0.755	20,600	10,300
5/16	1025	0.867	18,300	7,920
	1427	0.744	19,400	9,800
	1750	0.736	13,600	6,950
5/17	1033	0.727	29,600	15,300
	1341	0.731	21,700	11,200
	1616	0.754	12,200	<u>6,080</u>
Avg.				8,700

a Sample volume on dry basis at standard conditions of 20°C (68°F) and 760 mm (29.92 in) Hg.



Sulfur dioxide concentrations were also measured using TECO pulsed-fluorescence analyzers [Appendix G]. From 27 to 35 hours of concentration data were collected at the acid plants and Sinter Machine, and 6 hours at Blast Furnace 1 using TECO analyzers [Table 5]. These data also indicate wide fluctuations in  $\text{SO}_2$  concentrations at individual sites with short-term (15 min) average concentrations varying by as much as 63% to 145% from the 1.5 to 9-hour average for the day. Daily averages differed by as much as 11% to 50% from the overall average  $\text{SO}_2$  concentration measured at a site.

The fluctuations observed at Acid Plant 3 reportedly reflect variations in the sulfur content of the Sinter Machine feed. Fluctuations in tailgas  $\text{SO}_2$  concentrations at Acid Plants 1 and 2 were normally associated with a roaster being placed in service or being shut down.

A comparison of Method 8 results with concurrent TECO data [Tables 6 through 9] show that the overall averages are in close agreement (~5%) at Acid Plant 3 but differ by 15% to 32% at the other three locations. These comparisons use only the first nine sets of Method 8 and concurrent TECO data to be consistent with the requirements of the accuracy performance specifications of 40 CFR 52, Appendix D. The TECO was able to meet the accuracy performance specifications only at Acid Plant 3 and did not consistently meet the calibration error and calibration drift specifications [Table 10]. The analyzer did satisfy the zero drift and response time specifications.

The stability and precision of the dilution system [Figure 3] used with the TECO are considered the primary factors responsible for the analyzer not consistently meeting all performance specifications. Voltage variations in the electrical power supply could also have affected analyzer response; however, no voltage data are available to allow this effect to be quantified.

Table 5  
SUMMARY OF TECO, SO<sub>2</sub> DATA  
BUNKER HILL COMPANY  
Kellogg, Idaho

Sampling Location (Station No.)	Date	Hours of Data	Daily Average SO <sub>2</sub> Concentration ppmv	Range of SO <sub>2</sub> Concentrations <sup>a</sup> ppmv
Acid Plant 1 (48901)	5/21	2.0	4,020	3,860-4,140
	5/22	6.75	3,750	2,480-5,350
	5/23	8.0	1,380	690-2,250
	5/24	8.25	2,880	1,140-4,070
	5/25	<u>2.0</u>	<u>2,590</u>	2,120-2,970
	Total	27	Avg. <sup>b</sup> 2,720	
Acid Plant 2 (48902)	5/21	3.0	2,920	2,310-3,220
	5/22	7.0	2,570	1,980-3,640
	5/23	6.0	2,220	1,430-4,810
	5/24	7.75	4,200	1,040-6,180
	5/25	<u>5.25</u>	<u>1,600</u>	1,550-2,320
	Total	29	Avg. <sup>b</sup> 2,800	
Acid Plant 3 (48904)	5/14	8.0	1,650	340-3,190
	5/15	9.0	1,880	880-3,130
	5/16	9.0	1,640	840-3,290
	5/17	<u>9.0</u>	<u>1,580</u>	600-2,490
	Total	35	Avg. <sup>b</sup> 1,690	
Sinter Machine Weak Stream (48905)	5/13	1.5	4,760	3,590-5,600
	5/14	4.0	3,450	1,930-7,490
	5/15	7.75	5,700	2,740-9,660
	5/16	8.25	7,650	3,590-17,600
	5/17	<u>8.0</u>	<u>9,680</u>	3,140-16,300
	Total	29.5	Avg. <sup>b</sup> 6,970	
Blast Furnace 1 (48906)	5/26	6.25	1,840	1,030-3,860

a Fifteen-minute average

b Time-weighted

Tables 6 and 7  
COMPARISON OF METHOD 8 AND TECO, SO<sub>2</sub> DATA  
BUNKER HILL COMPANY  
Kellog, Idaho

Date	Start Time	Average SO <sub>2</sub> Concentration (ppmv) <sup>a</sup>		Difference <sup>b</sup> (ppmv)
		Method 8	TECO	
ACID PLANT 1 (STATION 48901)				
5/21	1605	4,510	4,160	350
5/22	0940	4,920	4,380	540
	1156	4,620	4,240	380
	1453	4,400	3,070	1,330
5/23	1013	1,760	2,000	-240
	1310	2,120	1,440	680
	1612	2,100	790	1,310
5/24	1030	1,680	2,340	-660
	1340	2,850	2,820	30
	1618 <sup>c</sup>	<u>6,840</u>	<u>3,900</u>	<u>2,940</u>
	Averages	3,220	2,800	410
ACID PLANT 2 (STATION 48902)				
5/21	1335	2,770	2,560	210
	1635	2,900	3,110	-210
5/22	1053	3,250	3,550	-300
	1311	2,310	2,560	-250
	1638	1,510	2,250	-740
5/23	1131	1,550	2,580	-1,030
	1423	990	1,710	-720
	1710	1,920	2,950	-1,030
5/24	1034	1,820	3,940	-2,120
	<u>1415<sup>c</sup></u>	<u>3,700</u>	<u>5,710</u>	<u>-2,010</u>
	Averages	2,110	2,800	-690

a One-hour average

b Method 8 minus TECO results

c Extra test, results not used to  
calculate averages for comparison

Tables 8 and 9  
COMPARISON OF METHOD 8 AND TECO, SO<sub>2</sub> DATA  
BUNKER HILL COMPANY  
Kellogg, Idaho

Date	Start Time	Average SO <sub>2</sub> Concentration (ppmv) <sup>a</sup>		Difference <sup>b</sup> (ppmv)
		Method 8	TECO	
ACID PLANT 3 (STATION 48904)				
5/15	1146	1,920	1,670	250
	1550	3,260	2,860	400
5/16	1020	1,780	1,780	0
	1430	2,100	1,970	130
	1716	1,440	1,320	120
5/17	0943	1,550	1,610	-60
	1158	2,300	2,410	-110
	1418	1,510	1,490	20
	1717	<u>1,450</u>	<u>1,310</u>	<u>140</u>
Averages		1,920	1,820	100
SINTER MACHINE WEAK STREAM (STATION 48905)				
5/15	1107	6,870	6,120	750
	1456	3,870	3,550	320
	1725	10,300	8,150	2,150
5/16	1025	7,920	7,600	320
	1427	9,800	8,500	1,300
	1750	6,950	6,080	870
5/17	1033	15,300	12,400	2,900
	1341	11,200	9,820	1,380
	1616	<u>6,080</u>	<u>5,520</u>	<u>560</u>
Averages		8,700	7,530	1,170

a One-hour average

b Method 8 minus TECO results

Table 10  
EVALUATION OF TECO PERFORMANCE  
BUNKER HILL COMPANY  
KELLOGG, IDAHO

Parameter	Specification	Results of Performance Specification Check			
		Acid Plant 1 (48901)	Acid Plant 2 (48902)	Acid Plant 3 (48904)	Sinter Machine Weak Stream (48905)
Accuracy	≤20% of reference mean value	28.8	57.3	11.6	21.2
Calibration Error	≤ 5% of each (50% and 90% span) calibration gases	(50% gas) 2.4	6.7	7.0	2.0
		(90% gas) 1.5	1.0	8.6	1.1
Zero Drift (2-hour)	≤ 2% of emission standard	0.48/0.0 <sup>a</sup>	0.10	0.07	.19
Calibration Drift (2-hour)	≤ 2% of emission standard	3.6/5.6 <sup>a</sup>	1.3	1.6	1.5
Response Time	15 minutes maximum	2.30	0.84	0.68	1.90

a Two values calculated at this station because of change in span gases.

Inaccuracies in the TECO data can be mitigated by using the results of the Method 8/TECO comparisons conducted at each site to develop an appropriate correction factor.\* For example, the average SO<sub>2</sub> concentration measured by Method 8 (3,220 ppm) was 115% of that measured by the TECO (2,800 ppm) during 9 hours of valid concurrent sampling at Acid Plant 1 [Table 6]. The TECO data collected at Acid Plant 1 was, therefore, multiplied by 1.15 to bring it into closer agreement with the Method 8 results. Likewise, all TECO data were adjusted [Table 11] according to the Method 8/TECO correction factor calculated for that sampling site.

Since the EPA regulation which is the subject of the present remand contains a 6-hour average limitation of 2,600 ppm for the acid plants, the adjusted TECO data were interpreted in terms of 6-hour averages [Table 12]. For those days during which at least 6 hours of data were collected, running 6-hour average SO<sub>2</sub> concentrations were calculated using 1-hour intervals. As indicated in Table 12, 6-hour average concentrations in excess of 2,600 ppm were measured at Acid Plants 1 and 2 with maximum 6-hour averages of 4,400 and 3,500 ppm, respectively. The maximum 6-hour concentration measured at Acid Plant 3 was 2,080 ppm.

Gas flows were measured at the acid plants and the Sinter Machine at approximately 2-hour intervals over the same time period during which concentration data were collected [Appendix G]. Average flow

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\* No Method 8 testing was conducted at the blast furnace, therefore, no adjustment factor was developed for this data.

Table 11  
SUMMARY OF ADJUSTED TECO, SO<sub>2</sub> DATA  
BUNKER HILL COMPANY  
Kellogg, Idaho

Sampling Location (Station No.)		Hours of Data		Adjusted Daily Average SO <sub>2</sub> Concentration ppv
Acid Plant 1 (48901)	5/21	2.0		4,620
	5/22	6.75		4,310
	5/23	8.0		1,590
	5/24	8.25		3,310
	5/25	<u>2.0</u>		<u>2,980</u>
	Total	27	Avg <sup>a</sup>	3,130
Acid Plant 2 (48902)	5/21	3.0		2,190
	5/22	7.0		1,930
	5/23	6.0		1,700
	5/24	7.75		3,150
	5/25	<u>5.25</u>		<u>1,200</u>
	Total	29	Avg <sup>a</sup>	2,100
Acid Plant 3 (48904)	5/14	8.0		1,730
	5/15	9.0		1,970
	5/16	9.0		1,720
	5/17	<u>9.0</u>		<u>1,660</u>
	Total	35	Avg <sup>a</sup>	1,770
Sinter Machine Weak Stream (48905)	5/13	1.5		5,520
	5/14	4.0		4,000
	5/15	7.75		6,610
	5/16	8.25		8,870
	5/17	<u>8.0</u>		<u>11,200</u>
	Total	29.5	Avg <sup>a</sup>	8,080

a Time-weighted

Table 12  
 SIX-HOUR AVERAGE SO<sub>2</sub> CONCENTRATIONS  
 ADJUSTED TECO DATA, ACID PLANTS 1 THROUGH 3  
 BUNKER HILL COMPANY  
 Kellogg, Idaho

Location	Date	Time Period	Running 6-hour Average SO <sub>2</sub> Concentrations (ppmv)
Acid Plant 1	5/22	0855-1640	4400
	5/23	0905-1747	1840,1610,1350
	5/24	0852-1758	2970,3280,3580
Acid Plant 2	5/22	0926-1746	1960,1840
	5/23	1100-1804	1700
	5/24	0848-1756	3360,3500
Acid Plant 3	5/14	0950-1835	1460,1630,1760
	5/15	0841-1925	1670,1850,2060,2080
	5/16	0850-1855	1610,1720,1660,1690
	5/17	0844-1818	1830,1760,1700,1600



rates for these sites are summarized below:

Location	Flow Rate (STP, Dry) <sup>a</sup> m <sup>3</sup> (ft <sup>3</sup> )/min	
	Average	Range
Acid Plant 1	444 (15,700)	393-475 (13,900-16,800)
Acid Plant 2	408 (14,400)	297-450 (10,500-15,900)
Acid Plant 3	659 (23,300)	552-736 (19,500-26,000)
Sinter Machine weak stream	475 (16,800)	371-549 (13,100-21,000)

a Standard conditions are 20°C (68°F) and 760 mm (29.92 in) Hg.

Gas stream parameters (temperature, moisture, etc.) measured concurrently with velocities are summarized in Table 13.

The following SO<sub>2</sub> emission rates have been calculated for the Bunker Hill acid plants and sinter machine weak stream using the average flow rates and SO<sub>2</sub> concentrations (adjusted TECO) measured by NEIC during May 12-25, 1978:

Location	Average SO <sub>2</sub> Emission Rate m. tons (tons)/day
Acid Plant 1	5.4 (5.9)
Acid Plant 2	3.3 (3.6)
Acid Plant 3	4.4 (4.9)
Sinter Machine weak stream	14.8 (16.2)

No emission rate was calculated for the blast furnace because there were no measured gas flow rates for that source.

Table 13  
SUMMARY OF GAS STREAM PARAMETERS  
BUNKER HILL COMPANY  
Kellogg, Idaho

Parameter	Sampling Locations			
	Acid Plant 1 (Station 48907)	Acid Plant 2 (Station 48903)	Acid Plant 3 (Station 48904)	Sinter Machine (Station 48905)
Temp. °K (°R)				
Ave.	324 (584)	319 (575)	326 (587)	410 (738)
Range	306-334 (551-602)	306-329 (551-592)	316-335 (569-603)	370-461 (666-830)
% Moisture				
Ave.	2.3	10.4	1.4	6.5
Range	1.1-6.5	8.9-13.1	1.1-2.0	2.3-8.4
% O <sub>2</sub>				
Ave.	7.8	8.3	11.9	20.2
Range	5.3-10.3	6.5-10.9	9.0-15.7	18.7-21.0
% CO <sub>2</sub>				
Ave.	0.0	0.1	1.9	0.2
Range	0.0-0.6	0.0-1.0	0.0-2.9	0.0-1.2
Average Stack Pressure <sup>a</sup>				
mm Hg (in Hg)	695.4 (27.38)	692.6 (27.27)	700.3 (27.57)	695.7 (27.39)
Average Molecular Wt (Dry) <sup>a</sup>	28.1	28.3	28.7	28.8

a Individual readings varied from average by maximum of approximately 1%.

### COMPARISON OF NEIC AND COMPANY DATA

Company flow data extracted from the operating logs for Acid Plants 1 through 3 [Appendix H] for the period of May 12 through 25 and the average for each sampling period was compared (after adjustment for SO<sub>2</sub> content) to NEIC-measured flow data for the same period [Table 14]. The comparison indicated that Company flow measurements at Acid Plant 2 averaged approximately 7.0% low compared to the NEIC data, while Company measurements at Acid Plants 1 and 3 were higher by 24% and 30%, respectively.

The NEIC flow data were based on average gas velocities measured using Method 2 with traversing by Method 1 and have an expected accuracy of approximately  $\pm 10\%$ <sup>5</sup>. The accuracy of Company flow data is unknown, but the use of single point velocity measurements makes this data questionable.

Both adjusted TECO data and concurrent Reich test results were used to calculate an average SO<sub>2</sub> concentration (reflecting a 2- to 11-hour sampling period) for each day of sampling at the acid plant tailgas streams [Table 15]. The overall average SO<sub>2</sub> concentrations, based on the above daily averages agreed to within 17% for all three acid plants. Less agreement was noted when the average concentrations for a single day were considered. No direct comparison was made between Method 8 and Reich test results because the difficulty associated with identifying concurrent pairs of data.

Company measured-SO<sub>2</sub> concentrations for the Sinter Machine weak stream were in wide disagreement with NEIC measurements. During testing at this location from May 13 to 17, the maximum Company-measured SO<sub>2</sub> concentrations was 3,500 ppm, while the NEIC SO<sub>2</sub> concentrations data (adjusted TECO) averaged 8,080 ppm.

Table 14  
COMPARISON OF NEIC AND COMPANY FLOW DATA  
ACID PLANTS 1 through 3  
BUNKER HILL COMPANY  
Kellogg, Idaho

Location	Date	Time Period (hours)	Average Flow Rate <sup>a</sup> (STP, Dry) <sup>b</sup>	
			NEIC Data m <sup>3</sup> (ft <sup>3</sup> )/min	Company Data m <sup>3</sup> (ft <sup>3</sup> )/min
Acid Plant 1	5/21	1330-1730	408 (14,400)	518 (18,300)
	5/22	0900-1630	453 (16,000)	580 (20,500)
	5/23	0900-1630	436 (15,400)	546 (19,300)
	5/24	0900-1630	464 (16,400)	563 (19,900)
	5/25	0900-1100	453 (16,000)	546 (19,300)
Acid Plant 2	5/21	1430-1730	405 (14,300)	368 (13,000)
	5/22	0830-1600	419 (14,800)	402 (14,200)
	5/23	0830-1600	419 (14,800)	362 (12,800)
	5/24	0830-1600	399 (13,700)	416 (14,700)
	5/25	0830-1030	416 (14,700)	357 (12,600)
Acid Plant 3	5/12	1400-1800	690 (24,400)	787 (27,800)
	5/13	1000-1200	690 (24,400)	874 (30,900)
	5/14	1000-1430	620 (21,900)	872 (30,800)
	5/15	0900-1800	676 (23,900)	874 (30,900)
	5/16	0800-1630	642 (22,700)	857 (30,300)
	5/17	0800-1900	645 (22,800)	860 (30,400)

a Flow rates measured by Bunker Hill at inlets to acid plants have been adjusted to outlet conditions using the following inlet SO<sub>2</sub> concentrations: Acid plants 1 and 2 - 7.0%, Acid plant 3 - 5.0%.

b STP = Standard temperature 20°C (68°F) and 760 mm (29.92 in) Hg.

## VI. PROCESS EVALUATION

Company personnel indicated during the testing period that operation of the smelter and zinc plant were relatively normal. No upsets or malfunctions occurred which would have greatly affected SO<sub>2</sub> emissions. During the survey period, the lead smelter was operating near design capacity, and the zinc plant was operating at about 70% of capacity.

Operating conditions for major pieces of smelter and zinc plant process equipment are summarized in the Confidential Technical Appendix to this report (EPA-330/2-78-018A)\*. The Confidential Technical Appendix also contains all process data collected by NEIC during the testing program.

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\* A more detailed process evaluation will be presented in a future NEIC report.

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\* A more detailed process evaluation will be presented in a future NEIC report.

## REFERENCES

1. Code of Federal Regulations, Part 40, Title 52. Approval and Promulgation of Implementation Plans, Section 52.676, Control Strategy: Sulfur Oxides - Eastern Washington and Northern Idaho Interstate Region.
2. Code of Federal Regulations, Part 40, Title 60. Standards of Performance for New Stationary Sources, Appendix A, Reference Methods 1, 2, 3, 6 and 8.
3. Code of Federal Regulations, Part 40, Title 52. Approval and Promulgation of Implementation Plans, Appendix D, Determination of Sulfur Dioxide Emissions From Stationary Sources by Continuous Monitors.
4. Jahnke, J.A., Chevey, J.L. and Homolya, J.B. 1976. Quenching Effects in SO<sub>2</sub> Fluorescence Monitoring Instruments. Environmental Science and Technology 10:12, p 1246 - 1250.
5. Shigehara, R.T., Todd, W.F., and Smith, W.S. Significance of Errors in Stack Sampling Measurements. Paper presented at 63rd Annual Meeting of the Air Pollution Control Association, June 14-19, 1970 (APCA No. 70-35).

APPENDIX A  
REQUEST FOR SAMPLING SITE MODIFICATIONS



U.S. ENVIRONMENTAL PROTECTION AGENCY

REGION X

1200 SIXTH AVENUE

SEATTLE, WASHINGTON 98101



REPLY TO  
ATTN OF Mail Stop 514

APR 12 1978

Mr. E. Viet Howard  
President  
Bunker Hill Company  
P. O. Box 29  
Kellogg, Idaho 83837

Dear Mr. Howard:

In our letter of March 3, 1978 we advised the Company that the Environmental Protection Agency (EPA) planned to conduct a sampling and monitoring program at your facility in early May 1978. In Bill Boyd's letter of March 23 he noted that our proposed May visit appeared to be acceptable to Bunker Hill, but requested additional details and verification of the purpose of the visit.

At this time we would like to inform you of the details and purpose of the measurement program and to request the Company to make necessary ductwork modifications and other arrangements required to conduct the program prior to the commencement of sampling on May 8. We have attempted to specify as clearly and precisely as possible what must be accomplished, but we believe that a visit by Mr. Osag and Mr. Sims will be necessary to ensure that all the necessary arrangements have been made. They will plan to be at the Bunker Hill complex on or about May 2, 1978, for the purpose of a pre-test inspection.

DESCRIPTION OF MEASUREMENT PROGRAM

The measurement program itself will commence on May 8, 1978 and should extend to approximately May 28, 1978. During this time we plan to measure:

- 1) Acid plant #1, #2 and #3 offgas-SO<sub>2</sub> concentration and gas flow.

### DUCTWORK MODIFICATIONS

For each sample location Bunker Hill will be required to make certain ductwork modifications to conduct the sampling. These modifications are fully outlined in Enclosure 1 and must be completed on or before May 1, 1978.

### PROCESS DATA

To enable us to equate the results of the gas stream measurements to specific process operations during the period of sampling, certain process and production information will be needed. This information is described in Enclosure 2.

It has come to our attention that operating personnel are instructed to shut-off strip chart recorders during periods when process equipment is shutdown. We find this highly unusual and, for our purposes, an unacceptable practice, since the strip charts do not then portray an accurate record of plant operations. Therefore, it is requested that Bunker Hill immediately continue to operate all strip chart recorders continuously at all times.

### PARTICIPANTS

We are not yet able to specify exactly which EPA personnel will be involved in the sampling/monitoring program. However, we intend to have two sampling teams of at least four members each present in the facility. In addition, since it is necessary to do the sampling and to simultaneously observe the status of process and/or control equipment, at least two additional people will be observing equipment status. Lastly, one or two men will be present in each mobile laboratory. Therefore we expect a total of 13 to 16 EPA people to be present.

Please inform us immediately if any piece of the process or control equipment (i.e., blast furnaces, sinter machine, all three acid plants) we have set for testing is scheduled to be down during any of the proposed sampling days.

As you know, this test program is necessary to enable the EPA to properly conduct the SO<sub>2</sub> remand activity. Authority for conducting these activities and for the advance arrangements required by the Company is contained in Section 114(a) of the Clean Air Act.

- 2) Blast furnace offgas-SO<sub>2</sub> concentration and gas flow.
- 3) Sinter machine weak stream-SO<sub>2</sub> concentration and gas flow.

We will also need to conduct certain ancillary activities necessary to assess the validity of the measurements and the operating status of the relevant equipment.

The purpose of the program is to measure the tailgas SO<sub>2</sub> content of each acid plant to evaluate the current performance of each acid plant and to document the impact of the changes Bunker Hill has apparently performed on the acid plants in the last 2 years. As we have noted before several times, acid plant performance specifications must be included as part of any response to the remand. We need to determine whether Bunker Hill's acid plants are properly designed, operated and maintained and to evaluate whether the acid plant emission performance needs to be significantly improved. As you know, we have requested Bunker Hill to install continuous monitors on the acid plant tailgas but the Company has refused to do so. The EPA sampling activity is not a substitute for Company installed and operated monitors and we once again request that Bunker Hill install and operate continuous SO<sub>2</sub> monitors on the tail gas of each acid plant.

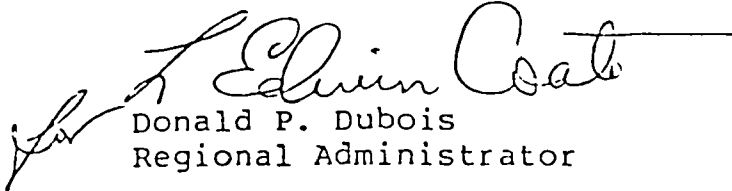
We also intend to measure the sinter machine weak stream and blast furnace SO<sub>2</sub> offgas concentrations and gas flows in order to determine what SO<sub>2</sub> total plant emission limit shall be included in the response to the remand and to determine the feasibility of scrubbing and other additional SO<sub>2</sub> controls for those streams. In our original total plant emission limit calculation we relied on data provided verbally by Bunker Hill personnel, which we presently believe to be incorrect.

#### SAMPLING LOCATIONS

Specific locations where samples will be drawn are fully detailed in Enclosure 1. We also request Bunker Hill to provide 110 vac electrical outlets at each sample location and to provide four parking sites, two close to the zinc plant acid plants and two as close as possible to the lead smelter acid plant. Each parking site will require 220 vac, single phase, 50 amp service for a mobile laboratory.

If you have questions regarding any of the above matters, please contact William T. Christian for legal matters or Larry L. Sims for technical matters by phone at (206) 442-1275 or (206) 442-1106, respectively. Thank you for your cooperation in this matter.

Sincerely,

Donald P. Dubois  
Regional Administrator

cc: William Boyd, Esq.  
Gene Baker

# ENCLOSURE 1

## SAMPLING LOCATIONS & DUCTWORK MODIFICATION

### A. References -

Location	Drawing No.	Title
No. 1 Acid Plant Outlet	B-1990 (REV E)	Plan & Elevation, Fan to Towers-Zinc Plant, 5'-0", 3'-6" and 3'-0" Diameter
No. 2 Acid Plant Outlet	(Same as for No. 1 Acid Plant)	
Smelter Acid Plant Outlet	D-1797 (REV A) B-2097 (REV C)	Duct, MK-S-39 and MK-S-40, 4'-6" Diameter Duct, Section View B-B & C-C, Lead Smelter 54" ID
Sinter Machine Weak Stream	90-10-003(REV 5) 90-10-037(REV 3)	New Flue & Fan System, General Arrangement, Blast Furnace to Baghouse New Flue & Fan System, Sinter Tail Gas Flue, Arrangement, Plan & Elevation
Blast Furnace	S-15-704(REV 3)	Sinter Blast Furnaces Dual Sinter Feed System, General Arrangement

### B. Sample Site Descriptions -

The following describes each location where measurements will be taken. Each description specifies certain modifications such as drilling new sampling ports and installing nipples which Bunker Hill is requested to perform.

Note - Reference to "Method #'s" denotes EPA source test methods contained in 40 CFR Part 60.

### No. 1 Acid Plant (Zinc plant)

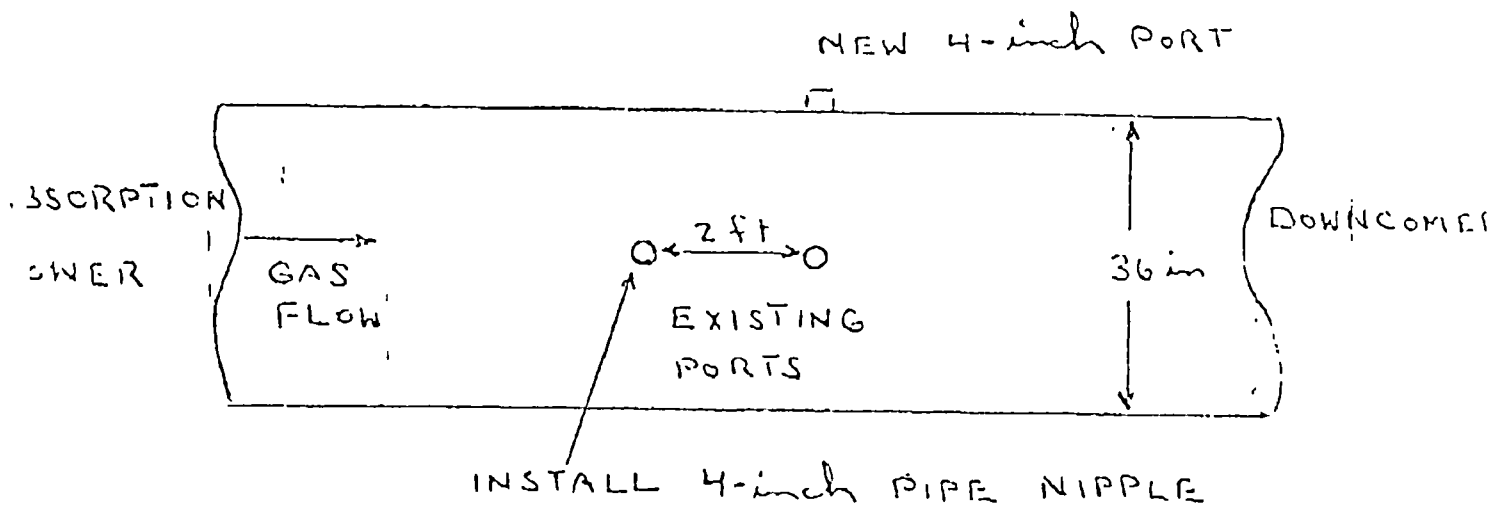
A sampling location for the tailgas is available in the 36 inch diameter stainless steel (SS) duct which connects the absorption tower with the downcomer to the fiberglass reinforced plastic (FRP) ductwork leading to the stack. Two 4.0 inch diameter ports are located on one side of this duct with a 2.0 ft. separation between the two ports. The distance between the sampling location and the nearest upstream and downstream flow disturbances are 40 and 10 ft. respectively.

It will be necessary to install an additional 4.0 inch diameter sampling port in the top of the duct at 90° to the existing downstream port (see figure 1). The two downstream ports will be used to measure velocity and SO<sub>2</sub> using a Method 8 train while the existing upstream port will be used to measure SO<sub>2</sub> concentration with the continuous monitor. The upstream port must be equipped with a 4.0 inch diameter pipe nipple with standard threads.

### No. 2 Acid Plant (Zinc plant)

An existing sampling site is located in the 42 inch diameter horizontal SS duct which connects the absorption tower with the downcomer to the FRP duct. This site is similar to the one at the No. 1 Acid Plant; i.e., two 4.0 inch diameter sampling ports are installed on one side of the duct with approximately a 2 ft. separation between them. The existing sampling location is less than 4 ft. downstream of a constriction in the ducting (duct changes from 52 to 42 inch diameter). No velocity measurements will be taken at this site. However, the existing ports will be used to monitor the SO<sub>2</sub> concentration of the acid plant tailgas. A continuous monitor will be installed in the upstream port while a Method 8 train will be used at the downstream port. The upstream port must therefore be equipped with a 4.0 inch diameter pipe nipple with standard threads. In addition, a 2 ft. section of the guardrail (top rail only) must be removed from the area directly behind the downstream port. The 2 ft. section should be centered around the centerline of the port.

Two 4.0 inch diameter ports with a 90° separation must be installed in the downcomer at a location 7.0 ft. above the platform. Both ports must be accessible from the platform (see figure 2). This location is approximately 20 ft. downstream of the SO<sub>2</sub> sampling site and will be used to measure velocities.



SIDE VIEW

Figure 1. Sampling Site at No. 1 Acid Plant

13/15

TOP VIEW

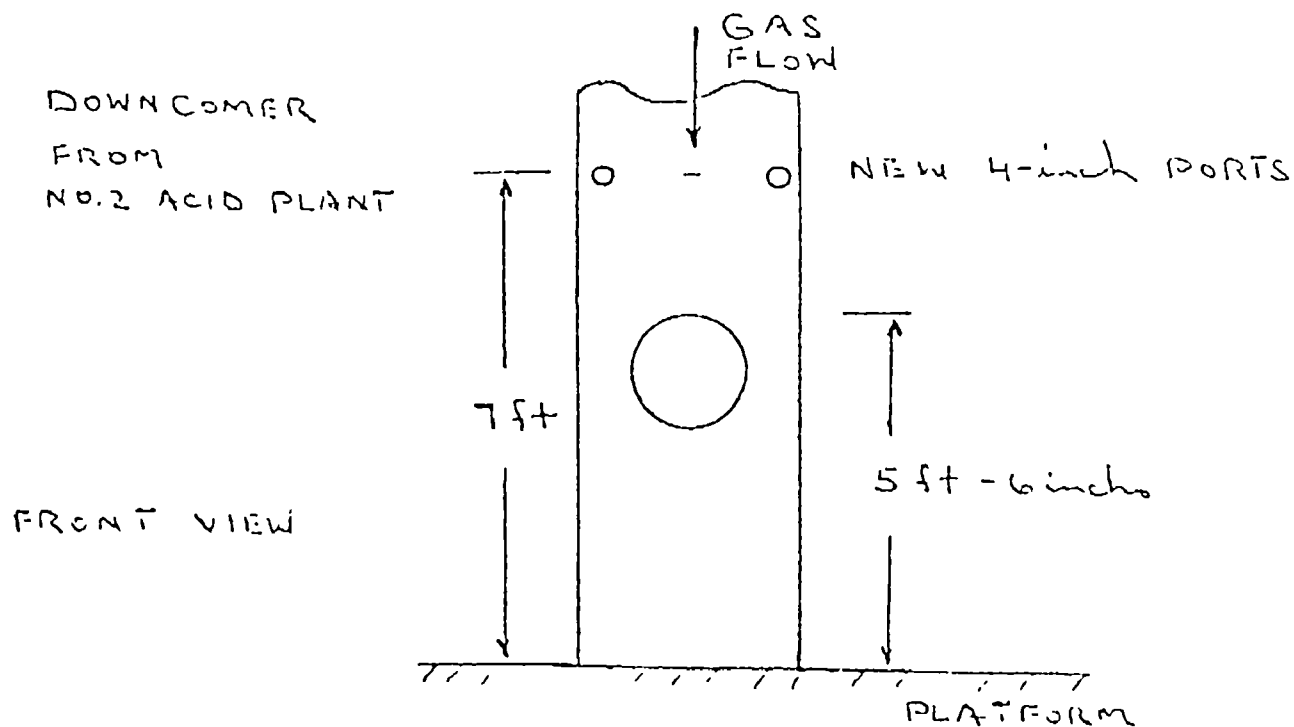
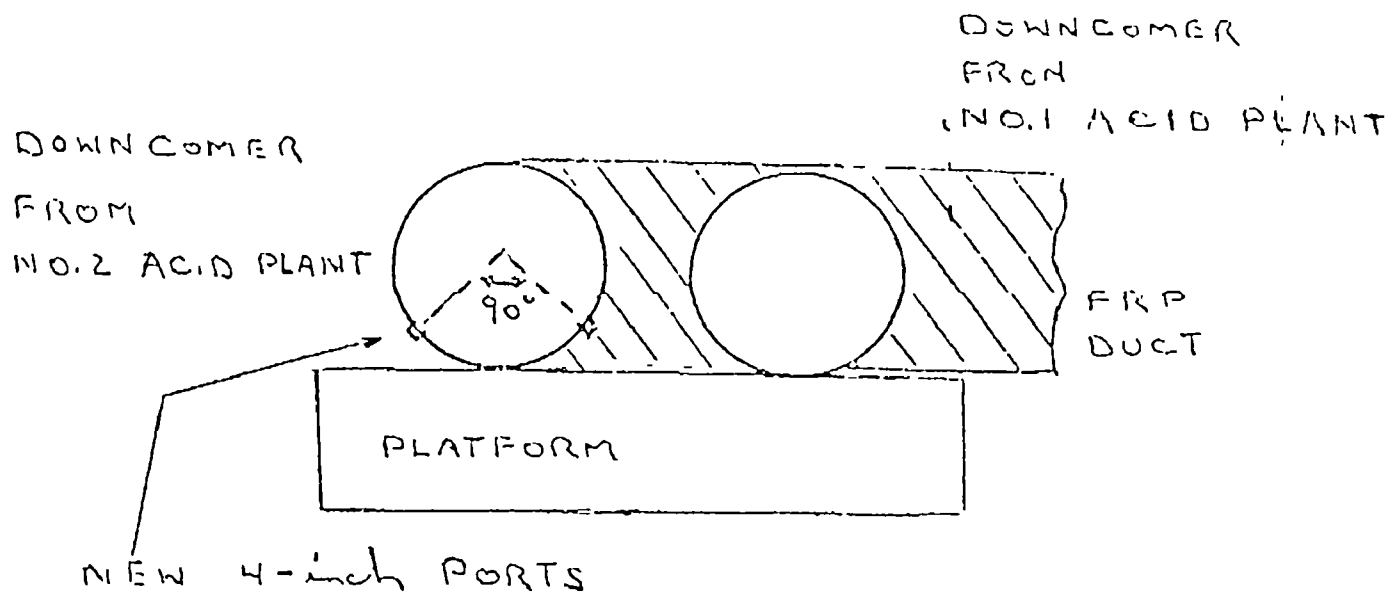


Figure 2. Velocity Measurement Site at No. 2 Acid Plant

45/10



### Smelter Acid Plant

An existing sampling site is available in the 54 inch diameter FRP downcomer which connects the outlet from the acid plant to the downstream fan. Two 4.0 inch diameter ports are now installed on the front side (with respect to the platform) of the duct, one port 2 ft. below the other. This sampling location has clearances of approximately 10 and 30 ft., respectively, from the nearest downstream and upstream flow disturbances. Both SO<sub>2</sub> monitoring and velocity measurements will be conducted at this site.

The upstream port requires a 4.0 inch diameter pipe nipple with standard threads while two additional 4.0 inch diameter ports must be installed at the same level as the existing downstream port. The new ports must be installed at 45° to the existing port, one on either side.

### Sinter Machine Weak Stream

The sinter machine weak stream (flue B) will be sampled approximately 10 ft. upstream of the breeching with the high velocity flue. Two 4.0 inch diameter ports (90° separation) must be installed in the side and top of the 42 inch diameter duct 10 ft. upstream of the connection with the high velocity flue. These ports will be used to measure SO<sub>2</sub> with the Method 8 train and velocity. A third port, equipped with a 4.0 inch diameter pipe nipple with standard threads, must be installed on the side of the duct 2 ft. upstream of the other ports (see figure 3).

### Blast Furnace

The blast furnace will be sampled in the gooseneck duct which connects the furnace with the brick flue leading to the high velocity flue (see figure 4). This location is the only practical sampling location available. Five 4.0 inch diameter ports should be installed in the top of the gooseneck at the location where the platform grating has been removed to expose the ducting. The new ports should be placed in a line perpendicular to the duct's axis with a separation between ports (center to center) of D/5, where D is the inside dimension (width) of the duct, and a separation between the horizontal walls and the nearest port of D/10. Since it is not possible to verify which blast furnace will be operating during the test period, the above modifications should be made for both.

HIGH VELOCITY FLUE

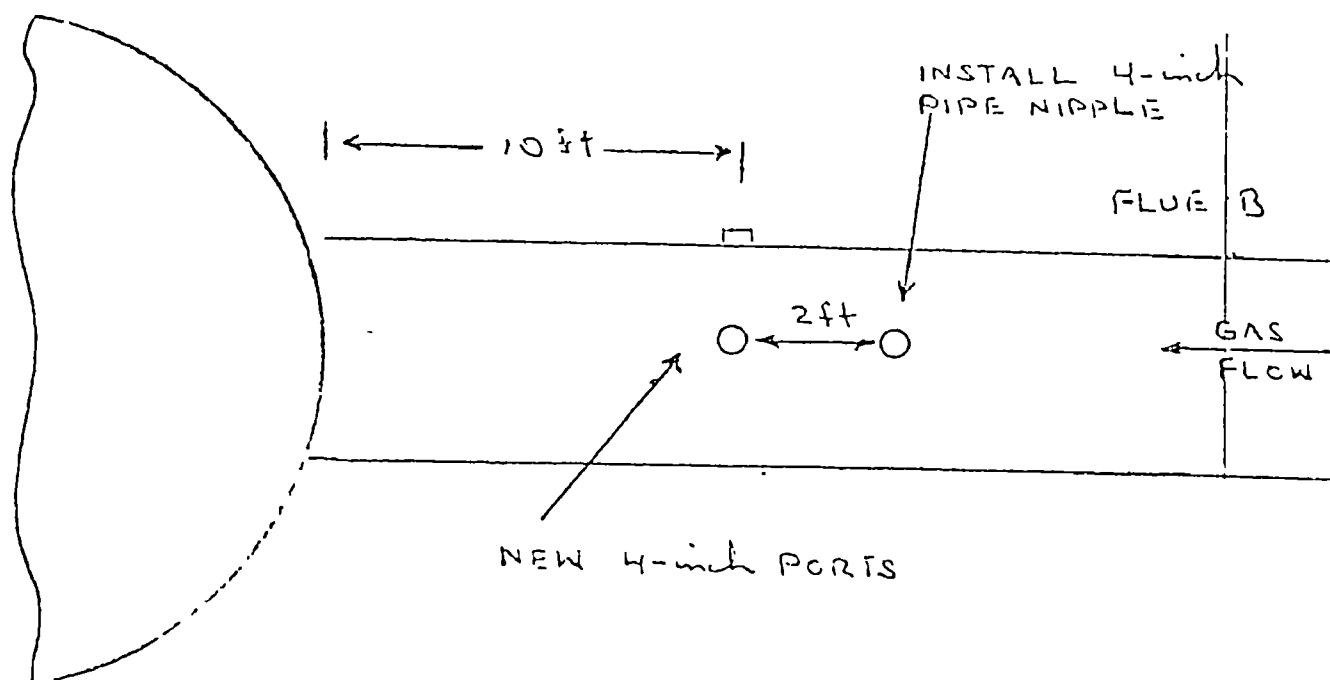


Figure 3. Sampling Site at Sinter Machine Weak Stream

11/5/1 10,

SIDE VIEW

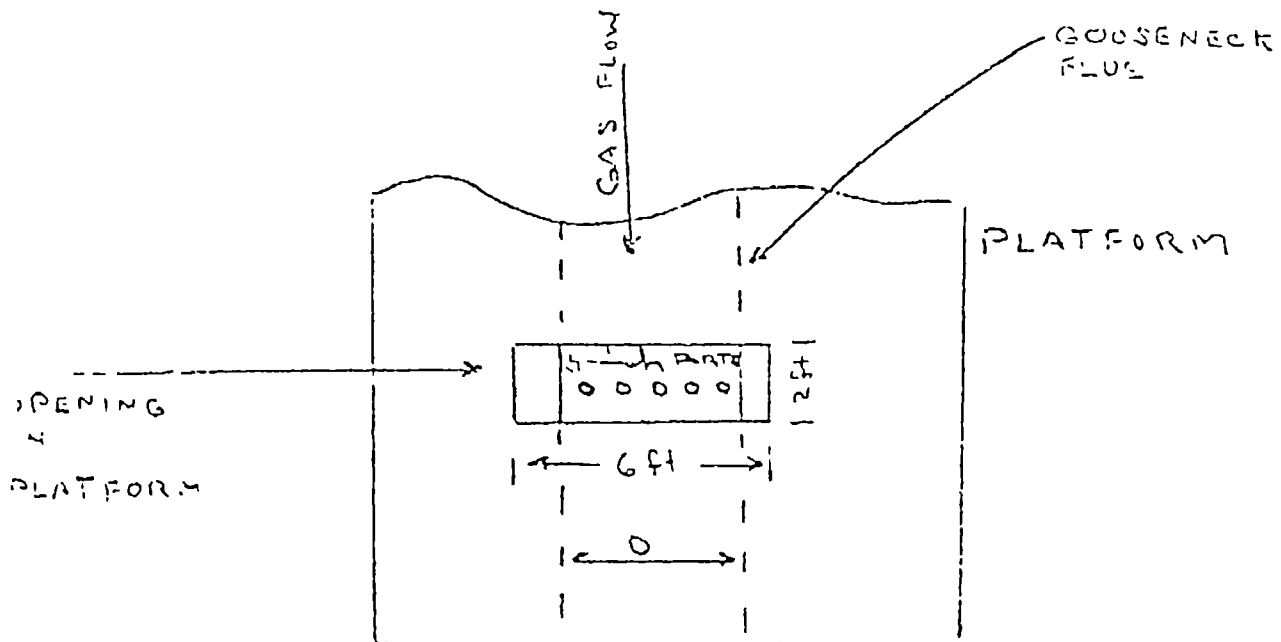
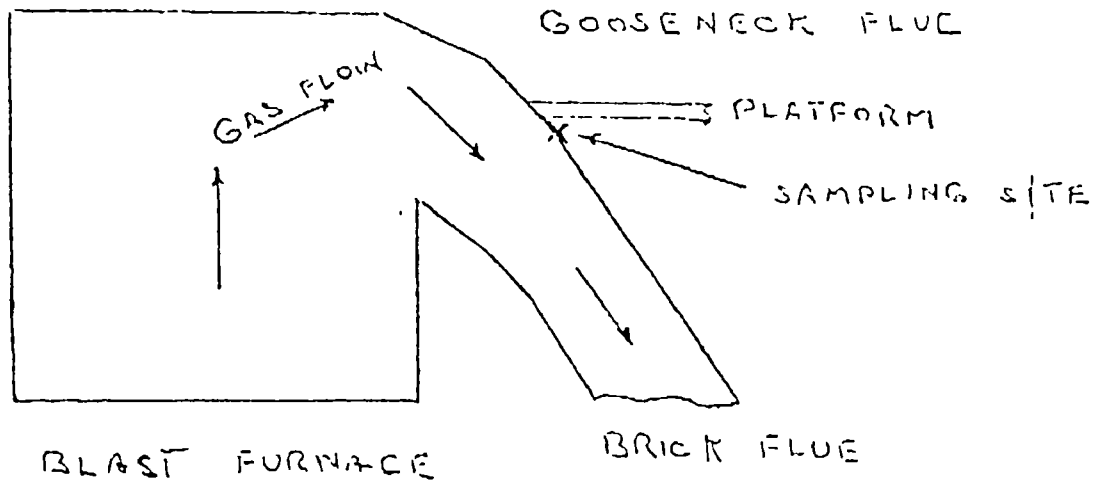


Figure 4. Sampling Site at Blast Furnaces

1/1/77

ENCLOSURE 2

PROCESS OPERATING DATA

During the course of the sampling and measurement program it will be necessary to collect certain process operating data. This data is necessary to assess the validity of the measurements. The following is a list of operating logs that must be supplied to EPA for each day of the sampling program:

<u>Location</u>	<u>Operating Log Title</u>
Zinc Plant	1. Daily Operating Report, No. 5 Roaster
	2. Flash Roasters Daily Report (Roaster 1-4)
	3. Daily Operating Log Sheet (Acid Plants 1 & 2)
Lead Smelter	4. Daily Operating Log Sheet (Smelter Acid Plant)
	5. Sinter Plant Operators Process Control Sheet
	6. Sinter Plant Process Control Sheet
	7. Main Baghouse Operation Log
	8. Lead Refinery Daily Report
	9. Blast Furnace Process Control Sheet
	10. Blast Furnace Schedule Control Sheet
	11. Casting and Loading Schedule Control Sheet
	12. Smelter Bedding Plant Work Sheet
	13. Ore Preparation Schedule Control Sheet

EPA will also require copies of the strip charts which indicate the SO<sub>2</sub> concentration and gas flow at both the zinc plant stack and the smelter stack and copies of the acid plants' charts which indicate gas flows and inlet SO<sub>2</sub> concentrations.

During the testing program, it will be necessary to have unrestricted access to the acid plant control rooms (zinc plant and smelter), the blast furnace control room, the sinter plant control room, and the roaster control rooms.

In addition, EPA will need to discuss with responsible Bunker Hill personnel the following items regarding continuous SO<sub>2</sub> measurements obtained by Bunker Hill. Specifically this is applicable to the smelter main stack, the zinc plant main stack, the inlet and outlets of the three acid plants and the sinter machine outlets:

1. Data Validation

- a. Criteria used
- b. Type of sample and instrument checks

2. Audit Procedures

3. Calibration

- a. Calibration procedure
- b. Schedule of calibration
- c. Type of standards used and type of devices used in flow calibration
- d. Flow checks on dilution system, if any

4. Preventive Maintenance

- a. Copy of procedure, or if done by vendor, copy of his procedure
- b. System checks and replacements done, filters, etc., and cleaning procedures

APPENDIX B  
PROJECT PLAN AND FIELD MODIFICATIONS TO PLAN

*Long*

PROJECT PLAN  
BUNKER HILL LEAD SMELTER/ZINC PLANT SOURCE TESTING PHASE .  
KELLOGG, IDAHO  
MAY 8-28, 1978

I. OBJECTIVES

The objectives of the survey are to determine SO<sub>2</sub> concentrations and flow rates at the following five locations in the Bunker Hill Smelter:

1. Tailgas from the No. 1 acid plant (zinc plant)
2. Tailgas from the No. 2 acid plant (zinc plant)
3. Tailgas from the No. 3 acid plant (lead smelter)
4. Sinter machine weak stream (lead smelter)
5. Blast furnace exhaust (lead smelter)

II. BACKGROUND

The Bunker Hill Company operates a combined lead smelter/zinc plant at Kellogg, Idaho. On November 19, 1975, Region X of the Environmental Protection Agency disapproved Regulation "S" of Idaho's State Implementation Plan (SIP), which limited sulfur dioxide (SO<sub>2</sub>) emissions from the Bunker Hill complex, and substituted a replacement regulation. This action was challenged by the Bunker Hill Company with the result that the U. S. Court of Appeals (Ninth Circuit) remanded the regulation to EPA for "further consideration of the technological feasibility of certain modifications of petitioner's smelter operations which would be required by the substituted regulations."

Region X subsequently requested that the National Enforcement Investigations Center (NEIC) conduct tests at the five locations discussed above to develop data for use in the remand proceeding.

A reconnaissance of the Bunker Hill complex was conducted by NEIC March 27-30, 1978, to evaluate sampling sites and identify process data to be collected in conjunction with the testing. Region X subsequently informed the Company of necessary sampling site modifications and process data requirements. Testing has been scheduled for the period of May 8-28, 1978. The following project plan has been developed for the scheduled testing, but is subject to change in the field.

### III. SAMPLING

#### Data Requirements

This sampling program is designed to provide approximately 40 hours of SO<sub>2</sub> concentration data and intermittent concurrent flow data for each of the three acid plant outlets and the sinter machine weak gas stream. The determination of gas stream flow rates will require the measurement of gas velocity, temperature, molecular weight, and moisture content. Approximately six hours (3 @ 2-hour tests) of SO<sub>2</sub> emission data (average concentration and emission rate) will be collected for the blast furnace. This will include concurrent determination of gas flow rates.

#### Sampling and Measurement Procedures

Sulfur dioxide concentrations at the acid plant outlets and the sinter machine weak stream will be measured with TECO pulsed fluorescent analyzers. During the testing at each site, the TECO analyzers will be checked for compliance with the following performance specifications\*:

Parameter	Specification
1. Accuracy <sup>a</sup> -----	≤20 percent of reference mean value
2. Calibration Error <sup>a</sup> -----	≤5 percent of each (50% and 90% span) calibration gas mixture.
3. Zero Drift (2-hours) <sup>a</sup> -----	≤2 percent of span
4. Zero Drift (24-hours) <sup>a</sup> -----	≤2 percent of span
5. Calibration Drift (2-hours) <sup>a</sup> ----	≤2 percent of span
6. Calibration Drift (24-hours) <sup>a</sup> ---	≤2.5 percent of span
7. Response Time-----	15 minutes maximum

*a Expressed as sum of absolute mean value plus 95 percent confidence interval of a series of tests.*

The calibration error check will be conducted in Denver and all other performance specification checks will be made in the field. The test procedures presented in Appendix D of 40 CFR 52.2850 [Attachment A]

*\* Performance specifications are those indicated in Appendix D of 40 CFR 52.2850 with: 1) "span" substituted for "emission standard" for the zero and calibration drift requirements 2) the 24-hour drift specifications adjusted to agree with 40 CFR 60 Appendix B, Performance Specification 2. Monitoring will be conducted using a span concentration of 5,000 ppm for the sinter machine weak gas stream and a span of 500 ppm for the acid plant outlets.*



will be used to verify the monitoring system's ability to meet the above performance specifications. The operational period specification will not be addressed since the monitors will only be operated while attended. The accuracy performance specification requires that the average  $\text{SO}_2$  concentration measured by the TECO over a one-hour period be compared to the results of a concurrent one-hour Method 8 sample. Nine such comparisons are required and will be performed. The analytical and computational portions of Method 8 as they relate to determination of sulfuric acid mist, as well as the requirement for isokinetic sampling, will be omitted since only  $\text{SO}_2$  data is desired. Sampling with the Method 8 train will be conducted at a constant rate (0.5 scfm).

It is estimated that approximately four days will be required at each sampling site to complete the performance specification check. During the latter three days of this period, the monitor will also be providing a continuous record (~30 hours in 10-hour intervals) of the  $\text{SO}_2$  concentration of the gas stream. An anticipated additional 10 hours of  $\text{SO}_2$  monitoring will be provided at each site following the completion of the performance specification check.

In conjunction with the continuous  $\text{SO}_2$  monitoring, gas flow rates will be determined at one-hour intervals throughout the day. Method 2 (using an isolated S-type pitot tube) with traversing according to Method 1 will be used to measure gas velocities. Temperature measurements will be collected with a thermocouple-potentiometer arrangement at the same time as the velocity measurements.

A gas sample will be collected (Method 3, grab) concurrent with the velocity measurements and analyzed with Fyrite analyzers (every third sample will be analyzed using an Orsat analyzer) to allow calculation of gas molecular weight. Moisture content of the acid plant tailgas streams can be assumed to be zero while the moisture content of the sinter machine weak gas stream will be determined from the Method 8 sampling and from independent Method 4 sampling.

The blast furnace  $\text{SO}_2$  emissions will be measured with a Method 8 sampling train (3 @ 2-hour runs), however, the analytical and computational portions of Method 8 as they relate to determination of sulfuric acid mist, as well as the requirement for isokinetic sampling, will be omitted since only  $\text{SO}_2$  data is desired. Sampling will be conducted at a rate proportional to gas velocity while traversing according to Method 1. Four gas samples will be collected (Method 3, grab) during each two-hour sampling run and analyzed using either Fyrite gas analyzers (three samples) or an Orsat analyzer (one sample). Moisture content of the gas stream will be determined from the weight gain of the Method 8 train impingers.

## Sampling Locations

### No. 1 Acid Plant (Station 01)

A sampling location for the tailgas is available in the 36-inch diameter stainless steel duct which connects the absorption tower with the downcomer to the fiberglass reinforced plastic (FRP) ductwork which leads to the stack. Two 4-inch diameter ports are located on one side of this duct with a 2 foot separation between the two ports. The distance between the sampling location and the nearest upstream and downstream flow disturbances are 40 and 10 feet respectively.

An additional 4-inch diameter sampling port will be installed in the top of the duct at 90° to the existing downstream port. The two downstream ports will be used to measure velocity and for the Method 8 train while the TECO will be installed in the upstream port. Sulfur dioxide concentrations will be measured at the midpoint of the duct. Twelve points will be used for velocity measurements.

### No. 2 Acid Plant (Stations 02 and 03)

An existing sampling site (Station 02) is located in the 42-inch diameter horizontal stainless steel duct which connects the absorption tower with the downcomer to the FRP duct. This site is similar to the one at the No. 1 Acid Plant (i.e., two 4-inch sampling ports are installed on one side of the duct with approximately a 2 foot separation between them). The existing sampling location is less than 4 feet downstream of a constriction in the ducting (duct changes from 52 to 42-inch diameter). Since the separation between this constriction and farthest sampling port is less than 7 feet required by Method 1, no velocity measurements will be taken at this site. However, the existing ports will be used to monitor the SO<sub>2</sub> concentration of the acid plant tailgas. The TECO will be installed in the upstream port while a Method 8 train will be used at the downstream port. Sulfur dioxide concentrations will be measured at the midpoint of the duct.

Two 4-inch ports with a 90° separation will be installed in the downcomer to the FRP duct at a location 7.0 feet above the existing platform. Both ports will be accessible from the platform. This location (Station 03) is approximately 20 feet downstream of the SO<sub>2</sub> sampling site (Station 02) and will be used to measure velocity. Twenty-four sample points will be used.

### Smelter Acid Plant (Station 04)

An existing sampling site is available in the 54-inch diameter FRP downcomer which connects the outlet from the acid plant to the downstream fan. Two 4-inch diameter ports are now installed on the front side

(with respect to the platform) of the duct, one port 2 feet below the other. This sampling location has clearances of approximately 10 and 30 feet, respectively, from the nearest downstream and upstream flow disturbances.

Both SO<sub>2</sub> monitoring and velocity measurements will be conducted at this site. The existing upstream port will be used for the TECO while the downstream port will be used for the Method 8 sampling. Sulfur dioxide concentrations will be measured at the midpoint of the duct.

Two additional 4-inch ports will be installed at the same level as the existing downstream port, each at 45° to the existing port. Velocity will be measured at these ports using 24 sample points.

#### Sinter Machine Weak Stream (Station 05)

The sinter machine weak stream (flue B) will be sampled approximately 10 feet upstream of the breeching with the high velocity flue. Two 4-inch diameter ports (90° separation) will be installed in the side and top of the 42-inch diameter duct 10 feet upstream of the connection with the high velocity flue. These ports will be used for the Method 8 train and velocity measurements (28 points). A third 4-inch port will be installed on the side of the duct 2 feet upstream of the other ports for the TECO.

#### Blast Furnace (Station 06)

The blast furnace will be sampled in the gooseneck duct which connects the furnace with the brick flue which leads to the high velocity flue. This location does not meet the minimum requirements of Method 1 (<1 diameter clearance between upstream flow disturbance and sampling location), however, it is the only practical sampling location available.

Five 4-inch diameter ports will be installed in the top of the gooseneck at the location where the platform grating has been removed to expose the ducting. The new ports will be placed in a line perpendicular to the duct's axis with an equal separation between ports (center to center). Since it is not possible to verify which blast furnace will be operating during the test period, the above modifications will be made for both. Twenty-five sampling points will be used.

#### Sample Handling and Analytical Requirements

Much of the data collected during this survey will be in-situ measurements; i.e., velocity and temperature measurements, Fyrite analysis, SO<sub>2</sub> concentrations. This data will be recorded on the prescribed forms (recorder paper) and handled according to established Chain-of-Custody procedures.

Only the peroxide catch (Impingers 2 and 3 with appropriate wash) of the Method 8 trains used for the performance specification testing and the blast furnace tests (~40 samples @ 1,000-ml/sample) will be retained and returned to Denver in polypropylene [Nalgene] containers under chain-of-custody procedures. Sulfate analyses will be according to Method 8\*. Approximately 14 sample blanks will also be retained for analysis.

In addition to the above analytical requirements, 12 cylinders of span gas ( $\text{SO}_2$  in air) will be analyzed prior to the start of the survey (within two weeks) and again at its completion to verify the  $\text{SO}_2$  concentration. Analyses will be by the barium-thorin titration procedure as required in Appendix D of 40 CFR 52.2850. It will also be necessary to check the isopropanol to be used during the sampling for peroxide impurities per Method 8.

### Sampling Schedule

Sampling will be conducted simultaneously at two locations throughout most of the survey. The No. 3 acid plant (lead smelter) and the sinter machine will be tested first followed by the No. 1 and 2 acid plants (zinc plant). It is anticipated that approximately seven days (including set up and tear down time) will be required at each site. The actual sampling program at each site will cover about a five-day period and will include:

- |                            |   |  |
|----------------------------|---|--|
| First Day                  | - | Performance specification checks of TECO for response time and zero and calibration drifts (2 and 24 hours). Flow measurements.  |
| Second Through Fourth Days | - | Performance specification checks for zero and calibration drifts (2 and 24 hours) and accuracy (3 @ 1-hour Method 8 tests/day). Flow measurements and monitoring of $\text{SO}_2$ concentration. |
| Fifth Day                  | - | Performance specification checks for zero and calibration drifts (24 hours). Flow measurements and monitoring of $\text{SO}_2$ concentrations.   |

The sampling at the blast furnace will either be conducted at the end of the survey or, if possible, simultaneous with other testing. It is anticipated that three days (including set up and tear down) will be required to complete this sampling.

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\* Barium-thorin titration

#### IV. Quality Control (QC) Program

Methods 2, 3, 4 and 8 will be conducted according to the procedures in Appendix A of 40 CFR 60 (unless previously noted). All calibration requirements and QC checks identified in Appendix A will be followed. This will involve calibration of the pitot tubes, dry gas meters, orifices and thermocouples both prior to the start of the survey and at its completion. In addition, blank samples (one blank/day for each site) of the peroxide solution used during the survey will be returned to Denver for analysis. All peroxide samples saved as blanks will have been first used as a second wash for Impingers 2 and 3.

Operation of the TECO continuous SO<sub>2</sub> monitors will be in accordance with the calibration and QC procedures in Appendix D of 52.2850\*. In addition, the following QC procedures will be implemented:

1. An in-house span check will be performed on the analyzers at least two times/day using 50% and 90% span gases. The length of the span check will depend on the time required for the TECO to stabilize.
2. All span gases will be checked for SO<sub>2</sub> concentrations both before the survey begins and at its completion.
3. A span check will be performed at least once per site with a span gas of unknown concentration provided by the Center's Quality Assurance officer.

The flow and pressure measurement devices used in the TECO conditioning/dilution system will be calibrated according to the following procedures:

1. Wet test meter - A calibration check of the meter will be performed before and after the study using the NBS traceable wet test meter at NEIC. The wet test meter has a range between 2 and 8 l/min. It will be used in the calibration of limiting orifices.
2. Dry gas meter - The dry gas meter will be calibrated against a NBS traceable wet test meter before and after the study. The dry gas meter will be used to calibrate the rotameter used in the dilution air systems. It has a range of 5 to 80 l/min.

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\* As previously mentioned, the 24-hour drift specifications have been modified to agree with 40 CFR 60, Appendix B, Performance Specification 2. In addition, "span" has been substituted for "emission standard" in the zero and calibration drift specifications.

3. Bubble flow meter - The bubble flow meter used in this study was calibrated against a NBS standard by the manufacturer. Other bubble flow meters used will be calibrated against this standard bubble flow meter. The bubble flow meter will be used to calibrate limiting orifices and to check the flow of the TECO. It has a range of 0 to approximately 2 l/min. Other flowmeters will be checked as needed during the study.
4. Rotameter - Rotameters used in this study will be calibrated against the above mentioned standards. The rotameter will be used in the air dilution system. It has a range of 500 ml/min to 30 l/min. The rotameter will be calibrated (10 points) before the study and after the study. During the study, 3 points on the calibration curve will be checked at the beginning and end of the testing at each site.
5. Orifices - All orifices used will be calibrated against the calibrated bubble flowmeter, wet test meter, or dry gas meter if necessary. The orifices will be used in the SO<sub>2</sub> dilution flow system. The range can vary depending on the capillary size. They will be calibrated before and after testing period at each site. Also they will be checked physically for clogging each day before startup.
6. Pressure gages and vacuum gages - All gages will be checked against a calibrated (NBS traceable) pressure transducer. The pressure gages will be used in the dilution system. They have a range of 0 to 100 psi. The gages will be checked before the survey. The only critical gage is the one in the SO<sub>2</sub> dilution system. It will be checked at the beginning and end of testing period at each site.

#### V. PROCESS DATA

Copies of the following plant operating logs will be obtained by NEIC for each day of the testing period.

<u>Location</u>	<u>Operating Log Title</u>
Zinc Plant	<ol style="list-style-type: none"> <li>1. Daily Operating Report, No. 5 Roaster</li> <li>2. Flash Roasters Daily Report (Roasters 1-4)</li> <li>3. Daily Operating Log Sheet (Acid Plants 1 &amp; 2) Lead Smelter</li> </ol>

## Lead Smelter

4. Daily Operating Log Sheet (Acid Plant)
5. Sinter Plant Operators Process Control Sheet
6. Sinter Plant Process Control Sheet
7. Main Baghouse Operation Log
8. Lead Refinery Daily Report
9. Blast Furnace Process Control Sheet
10. Blast Furnace Schedule Control Sheet
11. Casting and Loading Schedule Control Sheet
12. Smelter Bedding Plant Work Sheet
13. Ore Preparation Schedule Control

NEIC will obtain copies of the strip chart which indicates the SO<sub>2</sub> concentration and gas flow at both the zinc plant stack and the smelter stack and copies of the acid plants' charts which indicate gas flows and inlet SO<sub>2</sub> concentrations.

In addition, the NEIC will conduct a QC check of the SO<sub>2</sub> measurements obtained by Bunker Hill at the smelter and zinc plant stacks, the inlets and outlets of the three acid plants and the sinter plant. This QC check will include the following:

1. Data Validation
  - a. Criteria used.
  - b. Type of sample and instrument checks.
2. Audit Procedures
3. Calibration
  - a. Calibration procedures.
  - b. Schedule of calibration.
  - c. Type of standards used and type of devices used in flow calibration.
  - d. Flow checks on dilution system, if any.
4. Preventive Maintenance
  - a. Copy of procedure, or if done by vendor, copy of his procedure.
  - b. System checks and replacements done, filters, etc., and cleaning procedures.

## VI. FIELD REQUIREMENTS

### Personnel

- 3 Engineers
- 1 Chemist
- 8 Technicians

### Vehicles

- 2 Mobile Laboratories-NEIC
- 2 Sedans-GSA Motor Pool, Spokane, Washington
- 1 Step Van-NEIC
- 1 Station Wagon-Rental, Spokane, Washington

### Safety

Field personnel will adhere to NEIC and Company safety requirements. The following safety equipment will be worn by all NEIC employees while on site (except when in the offices or mobile lab areas):

- Hard hat
- Safety shoes
- Safety glasses (side shields not required)
- Long sleeve shirts
- Respirators with MSA-dust and fume filters (worn where posted)

### Time Schedule

- May 6-8 - Equipment and laboratories to study area
- May 8-28 - Conduct sampling activities
- June 30 - Draft report to Region
- Final report available two weeks following receipt of comments from Region.



## FIELD MODIFICATIONS TO PROJECT PLAN

During the actual testing, the Bunker Hill Project Plan was modified as indicated below:

1. The TECO analyzer was used to measure  $\text{SO}_2$  concentrations at the blast furnace instead of Method 8 and no flow measurements were conducted at that site.
2. The performance specifications used to evaluate the TECO analyzers were altered by omitting the 24-hour drift specifications and using the proposed acid plant emission limit (2,600 ppm) and the average Method 8 result at the Sinter Machine weak stream (8,700 ppm) for drift calculations.
3. All performance specification checks were conducted in the field.
4. Flow measurements were conducted at 2-hour intervals vs. the proposed 1-hour intervals.
5. Moisture content of the acid plant tailgas streams were not assumed to be zero. Moisture was determined from results of Method 8 sampling.
6. Flow measurements at Acid Plant 1 were conducted in the downcomer to the FRP dust (Station 48907) instead of the originally proposed site (Station 48901).
7. The method 8 train was used at the upstream port at Acid Plant 1 (Station 48901) instead of the downstream port.
8. Span gases originally intended to be used for Quality Control checks were used as calibration gases.
9. The TECO analyzer was used to determine dilution rates, therefore, flow and pressure measurement devices in the TECO dilution system were not calibrated according to the procedures initially proposed.
10. No QC check of the Bunker Hill  $\text{SO}_2$  monitors was conducted because of the Company's objection.

APPENDIX C  
SAMPLING TRAIN DESCRIPTION

## STACK SAMPLING EQUIPMENT

The Scientific Glass Model AP-5000 modular STAC-O-LATUR<sup>tm</sup> sampling train consists of a control unit, a sampling unit and a vacuum unit. The units are connected together with quick disconnect electrical and air lines, and umbilical cords.

The AP-500 control unit contains the following

1. Dual-inclined manometer (range 0-5" H<sub>2</sub>O) for indicating the pitot tube velocity pressure and the orifice pressure drop.
2. Temperature controls for the oven and probe.
3. A flow valve and a bypass valve for adjusting sampling rates.
4. Digital Temperature Indicator (DTI) which give an instant readout from six (6) points; stack, probe, oven, impinger outlet, meter inlet, meter outlet by the use of a selector switch.
5. Umbilical cords (50 and 100 ft lengths) which interconnect the control and sampling units.
6. Communications sets are wired through umbilical cord from control unit to the sampling unit.\*

The sampling unit is made up of three distinct sections; impinger case, oven and probe. All three sections can be converted to form one sampling unit or can be separated for unusual sampling conditions. Below are the individual component descriptions:

1. Probe Sheath - Made of 316 stainless steel. The nozzle end is packed with asbestos string. The ball joint (sampling unit) end has a woven telfon O Ring as packing material.

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\* Separate communication system used during this test program.

2. Probe liner - 5/8" O.D. medium wall glass (pyrex) or stainless steel (316) tubing logarithmically wrapped with nicrome heating element, having a resistance of 2 ohms/ft. The liner is insulated with fiberglass and asbestos with a type K thermocouple imbedded for sensing the probe temperature.
3. Filter Frit - Porous glass frit (coarse) banded to silicone rubber.
4. Oven - Fiberglass insulated capable of maintaining 120°C (248°F) in cold weather (0°C).

The vacuum unit (pump) is capable of drawing a high vacuum (50 cm Hg) and a moderate volume (14 lpm) of air. The pump is rotary fiber vane type which does not require lubrication, but oil bath filters are used for pump protection.

APPENDIX D  
CHAIN-OF-CUSTODY

ENVIRONMENTAL PROTECTION AGENCY  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

CHAIN OF CUSTODY PROCEDURES  
June 1, 1975

GENERAL

The evidence gathering portion of a survey should be characterized by the minimum number of samples required to give a fair representation of the water, air or solid waste sampled. To the extent possible, the quantity of samples and sample locations will be determined prior to the survey.

Chain of Custody procedures must be followed to maintain the documentation necessary to trace sample possession from the time taken until the evidence is introduced into court. A sample is in your "custody" if:

1. It is in your actual physical possession, or
2. It is in your view, after being in your physical possession, or
3. It was in your physical possession and then you locked it up in a manner so that no one could tamper with it.

All survey participants will receive a copy of the survey study plan and will be knowledgeable of its contents prior to the survey. A pre-survey briefing will be held to re-appraise all participants of the survey objectives, sample locations and Chain of Custody procedures. After all Chain of Custody samples are collected, a de-briefing will be held in the field to determine adherence to Chain of Custody procedures and whether additional evidence type samples are required.

SAMPLE COLLECTION

1. To the maximum extent achievable, as few people as possible should handle the sample.
2. Water, air, or solid waste samples shall be obtained, using standard field sampling techniques.
3. Sample tags (Exhibit I) shall be securely attached to the sample container at the time the complete sample is collected and shall contain, at a minimum, the following information: station number, station location, data taken, time taken, type of sample, sequence number (first sample of the day - sequence No. 1, second sample - sequence No. 2, etc.), analyses required and samplers. The tags must be legibly filled out in ballpoint (waterproof ink).
4. Blank samples shall also be taken with preservatives which will be analyzed by the laboratory to exclude the possibility of container or preservative contamination.
5. A pre-printed, bound Field Data Record logbook shall be maintained to record field measurements and other pertinent information necessary to refresh the sampler's memory in the event he later takes the stand to testify regarding his actions during the evidence gathering activity. A separate set of field notebooks shall be maintained for each survey and stored in a safe place where they could be protected and accounted for at all times. Standard formats (Exhibits II and III) have been established to minimize field entries and include the date, time, survey, type of samples taken, volume of each sample, type of analysis, sample numbers, preservatives, sample location and field measurements such as temperature, conductivity,

DO, pH, flow and any other pertinent information or observations. The entries shall be signed by the field sampler. The preparation and conservation of the field logbooks during the survey will be the responsibility of the survey coordinator. Once the survey is complete, field logs will be retained by the survey coordinator, or his designated representative, as a part of the permanent record.

6. The field sampler is responsible for the care and custody of the samples collected until properly dispatched to the receiving laboratory or turned over to an assigned custodian. He must assure that each container is in his physical possession or in his view at all times, or locked in such a place and manner that no one can tamper with it.
7. Colored slides or photographs should be taken which would visually show the outfall sample location and any water pollution to substantiate any conclusions of the investigation. Written documentation on the back of the photo should include the signature of the photographer, time, date and site location. Photographs of this nature, which may be used as evidence, shall be handled recognizing Chain of Custody procedures to prevent alteration.

#### TRANSFER OF CUSTODY AND SHIPMENT

1. Samples will be accompanied by a Chain of Custody Record which includes the name of the survey, samplers' signatures, station number, station location, date, time, type of sample, sequence number, number of containers and analyses required (Fig. IV). When turning over the possession of samples, the transferor and transferee will sign, date and time the sheet. This record sheet allows transfer of custody of a group of samples in the field, to the mobile laboratory or when samples are dispatched to the NEIC - Denver laboratory. When transferring a portion of the samples identified on the sheet to the field mobile laboratory, the individual samples must be noted in the column with the signature of the person relinquishing the samples. The field laboratory person receiving the samples will acknowledge receipt by signing in the appropriate column.
2. The field custodian or field sampler, if a custodian has not been assigned, will have the responsibility of properly packaging and dispatching samples to the proper laboratory for analysis. The "Dispatch" portion of the "Chain of Custody Record" shall be properly filled out, dated, and signed.
3. Samples will be properly packed in shipment containers such as ice chests, to avoid breakage. The shipping containers will be padlocked for shipment to the receiving laboratory.
4. All packages will be accompanied by the Chain of Custody Record showing identification of the contents. The original will accompany the shipment, and a copy will be retained by the survey coordinator.
5. If sent by mail, register the package with return receipt requested. If sent by common carrier, a Government Bill of Lading should be obtained. Receipts from post offices, and bills of lading will be retained as part of the permanent Chain of Custody documentation.
6. If samples are delivered to the laboratory when appropriate personnel are not there to receive them, the samples must be locked in a designated area within the laboratory in a manner so that no one can tamper with them. The same person must then return to the laboratory and unlock the samples and deliver custody to the appropriate custodian.

LABORATORY CUSTODY PROCEDURES

1. The laboratory shall designate a "sample custodian." An alternate will be designated in his absence. In addition, the laboratory shall set aside a "sample storage security area." This should be a clean, dry, isolated room which can be securely locked from the outside.
2. All samples should be handled by the minimum possible number of persons.
3. All incoming samples shall be received only by the custodian, who will indicate receipt by signing the Chain of Custody Sheet accompanying the samples and retaining the sheet as permanent records. Couriers picking up samples at the airport, post office, etc. shall sign jointly with the laboratory custodian.
4. Immediately upon receipt, the custodian will place the sample in the sample room, which will be locked at all times except when samples are removed or replaced by the custodian. To the maximum extent possible, only the custodian should be permitted in the sample room.
5. The custodian shall ensure that heat-sensitive or light-sensitive samples, or other sample materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained.
6. Only the custodian will distribute samples to personnel who are to perform tests.
7. The analyst will record in his laboratory notebook or analytical worksheet, identifying information describing the sample, the procedures performed and the results of the testing. The notes shall be dated and indicate who performed the tests. The notes shall be retained as a permanent record in the laboratory and should note any abnormalities which occurred during the testing procedure. In the event that the person who performed the tests is not available as a witness at time of trial, the government may be able to introduce the notes in evidence under the Federal Business Records Act.
8. Standard methods of laboratory analyses shall be used as described in the "Guidelines Establishing Test Procedures for Analysis of Pollutants," 38 F.R. 28758, October 16, 1973. If laboratory personnel deviate from standard procedures, they should be prepared to justify their decision during cross-examination.
9. Laboratory personnel are responsible for the care and custody of the sample once it is handed over to them and should be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the tests were run.
10. Once the sample testing is completed, the unused portion of the sample together with all identifying tags and laboratory records, should be returned to the custodian. The returned tagged sample will be retained in the sample room until it is required for trial. Strip charts and other documentation of work will also be turned over to the custodian.
11. Samples, tags and laboratory records of tests may be destroyed only upon the order of the laboratory director, who will first confer with the Chief, Enforcement Specialist Office, to make certain that the information is no longer required or the samples have deteriorated.




# EXHIBIT I

EPA, NATIONAL ENFORCEMENT INVESTIGATIONS CENTER			
Station No.	Date	Time	Sequence No.
Station Location			<input type="checkbox"/> Grab <input type="checkbox"/> Comp.
<input type="checkbox"/> BOD <input type="checkbox"/> Solids <input type="checkbox"/> COD <input type="checkbox"/> Nutrients	<input type="checkbox"/> Metals <input type="checkbox"/> Oil and Grease <input type="checkbox"/> D.O. <input type="checkbox"/> Bact. <input type="checkbox"/> Other.	Remarks/Preservative:	
Samplers:			

Front

ENVIRONMENTAL PROTECTION AGENCY  
 OFFICE OF ENFORCEMENT  
 NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
 BUILDING 53, BOX 25227, DENVER FEDERAL CENTER  
 DENVER, COLORADO 80225



Back

## EXHIBIT II

1. SURVEY, PHASE\_\_\_\_\_, DATE \_\_\_\_\_

OF SAMPLE \_\_\_\_\_

## ANALYSES REQUIRED

STATION NUMBER	STATION DESCRIPTION	TOTAL VOLUME	TYPE CONTAINER	PRESERVATIVE	NUTRIENTS	BOD	COD	TOC	TOTAL SOLIDS	SUSPENDED SOLIDS	ALKALINITY	DO	pH*	CONDUCTIVITY*	TEMPERATURE*	TOTAL COLIFORM	FECAL COLIFORM	TURBIDITY	OIL AND GREASE	METALS	BACII	PESTICIDES	HFPB	TRACE ORGANICS	PHENOL	CYANIDE

ARKS

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

[illegible]

ENVIRONMENTAL PROTECTION AGENCY  
Office Of Enforcement  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
Building 53, Box 25227, Denver Federal Center  
Denver, Colorado 80225

SURVEY	SAMPLERS: (Signature)
--------	-----------------------

Relinquished by: (Signature)		Received by: (Signature)		Date/Time
Relinquished by: (Signature)		Received by: (Signature)		Date/Time
Relinquished by: (Signature)		Received by: (Signature)		Date/Time
Relinquished by: (Signature)		Received by Mobile Laboratory for field analysis: (Signature)		Date/Time
Dispatched by: (Signature)	Date/Time	Received for Laboratory by:		Date/Time
Method of Shipment:				

## CHAIN OF CUSTODY RECORD

[illegible]

## CHAIN OF CUSTODY RECORD

489 04

★GPO 679-040

## CHAIN OF CUSTODY RECORD

[illegible]

## CHAIN OF CUSTODY RECORD

[illegible]

489 00

**Distribution**   Orig – Accompany Shipment  
1 Copy – Survey Coordinator Field Files

★GPO 679-040



## CHAIN OF CUSTODY RECORD

SURVEY						SAMPLERS: (Signature)						
STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ NO	NO OF CONTAINERS	ANALYSIS REQUIRED			
				Water		Air						
				Comp	Grab							
-18905	WEAK STREAMSINGER	5/16/78	1245	(TAG#) 1747	✓	06	1	H <sub>2</sub> O <sub>2</sub> /SC <sub>2</sub>				
48905	" "	"	1645	(TAG#) 1748	✓	07	1	"				
48905	" "	"	2000	(TAG#) 1749	✓	08	1	"				
48905	" "	"	2000	(TAG#) 1750	✓	09	1	"				
Relinquished by: (Signature)			Received by (Signature)					Date/Time				
[Signature]			Brian R. [Signature]					5/1-1/09-50				
Relinquished by: (Signature)			Received by: (Signature)					Date/Time				
Brian L. Ragge			Timothy R. Osoy					5/22/78   11:15				
Relinquished by: (Signature)			Received by: (Signature)					Date/Time				
Timothy R. Osoy			Steven H. [Signature]					5/28/78   10:55				
Relinquished by: (Signature)			Received by Mobile Laboratory for field analysis: (Signature)					Date/Time				
[Signature]			[Signature]					[ ]				
Dispatched by: (Signature)		Date/Time	Received for Laboratory by:		Date/Time							
Steven M. Ragge		5/30   10:25	Richard A. [Signature]		5/30   10:15							
Method of Shipment.												
G.O.V.												

## CHAIN OF CUSTODY RECORD

489 07

## CHAIN OF CUSTODY RECORD

-483 - 3

## CHAIN OF CUSTODY RECORD

489. 15.

☆GPO 679-040

## CHAIN OF CUSTODY RECORD

[illegible]

## CHAIN OF CUSTODY RECORD

489 23

\*GPO 679-040

## CHAIN OF CUSTODY RECORD

\*GPO 679-040

## CHAIN OF CUSTODY RECORD

[illegible]



## CHAIN OF CUSTODY RECORD

[illegible]

## CHAIN OF CUSTODY RECORD

489 22

☆GPO 679-040

## CHAIN OF CUSTODY RECORD

Company  
Splits  
account out of  
1,000 ml

[illegible]

ENVIRONMENTAL PROTECTION AGENCY  
Office Of Enforcement  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
Building 53, Box 25227, Denver Federal Center  
Denver, Colorado 80225

Company splits  
As per 100 ml  
out of 1000 ml

CHAIN OF CUSTODY RECORD

SURVEY				SAMPLERS: (Signature)					
BUNKER HILL				Brian Rogers					
STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ NO	NO OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp	Grab				
48904	ACID PLANT	5-16-78	1245	(SAMPLER FILLED)	1770		04		H <sub>2</sub> O <sub>2</sub> /S <sub>02</sub> 100ml
48904	ACID PLANT	5-16-78	1650	1771	1650 RLP		05		H <sub>2</sub> O <sub>2</sub> /S <sub>02</sub> 98ml
48904	ACID PLANT	5-16-78	1933	1772			06		H <sub>2</sub> O <sub>2</sub> /S <sub>02</sub> 99ml
48904	ACID PLANT	5-16-78	1943	1773			07		H <sub>2</sub> O <sub>2</sub> BLANK 100ml
48905	WEAK SINTER PLANT	5-16-78	1245	1774			06		H <sub>2</sub> O <sub>2</sub> /S <sub>02</sub> 98ml
48905	WEAK SINTER PLANT	5-16-78	1645	1775	1645 RLP		07		H <sub>2</sub> O <sub>2</sub> /S <sub>02</sub> 99ml
48905	WEAK SINTER PLANT	5-16-78	2000	1776			08		H <sub>2</sub> O <sub>2</sub> /S <sub>02</sub> 109ml
48905	WEAK SINTER PLANT	5-16-78	2000	1777			09		H <sub>2</sub> O <sub>2</sub> BLANK 98ml
Relinquished by: (Signature)				Received by: (Signature)					Date/Time
Brian L. Rogers				Don E. Halberg for ALSID, SHOUDE					5/18/78
Relinquished by: (Signature)				Received by: (Signature)					Date/Time
				REPRESENTING BUNKER HILL					
Relinquished by: (Signature)				Received by: (Signature)					Date/Time
Relinquished by: (Signature)				Received by Mobile Laboratory for field analysis. (Signature)					Date/Time
Dispatched by: (Signature)		Date/Time		Received for Laboratory by:			Date/Time		
Method of Shipment.									

ENVIRONMENTAL PROTECTION AGENCY  
Office Of Enforcement  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
Building 53, Box 25227, Denver Federal Center  
Denver, Colorado 80225

*Company Split  
Approx. 100 ml  
out of 1000 ml.*

CHAIN OF CUSTODY RECORD

SURVEY					SAMPLERS. (Signature)				
<i>BUNKER HILL</i>					<i>Bunker Hill - making company split</i>				
STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ NO	NO OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp	Grab				
48904	ACID PLANT	5/17/78	1155	(1778)			08	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 112 ml
48904	ACID PLANT	5/17/78	1400	(1779)			09	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 100 ml
48904	ACID PLANT	5/17/78	1146	(1780)			10	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 97 ml
48904	ACID PLANT	5/17/78	1931	(1781)			11	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 103 ml
48904	ACID PLANT	5/17/78	1942	(1782)			12	1	H <sub>2</sub> O <sub>2</sub> BLANK 70
48905	WEAK STREAM SINTER	5/17/78	1230	(1783)			10	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 123 ml
48905	WEAK STREAM SINTER	5/17/78	1905	(1784)			11	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 99 ml
48905	WEAK STREAM SINTER	5/17/78	1900	(1785)			12	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 95 ml
48905	WEAK STREAM SINTER	5/17/78	1935	(1786)			13	1	H <sub>2</sub> O <sub>2</sub> BLANK 103 ml

Relinquished by: (Signature) <i>Brian L. Regard</i>	Received by: (Signature) <i>LA. Stollery FOR ASA</i>	Date/Time 1745 5/21/78
Relinquished by: (Signature)	Received by: (Signature) <i>REP. ENFORCEMENT BUNKER HILL</i>	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by Mobile Laboratory for field analysis (Signature)	Date/Time
Dispatched by: (Signature)	Date/Time	Received for Laboratory by:
Method of Shipment:		Date/Time

489 09

Distribution Orig. - Accompany Shipment  
1 Copy - Survey Coordinator Field Files

ENVIRONMENTAL PROTECTION AGENCY  
Office Of Enforcement  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
Building 53, Box 25227, Denver Federal Center  
Denver, Colorado 80225

**CHAIN OF CUSTODY RECORD**

*Company Split*

SURVEY				SAMPLERS: (Signature)					
<i>Bunker Hill Co</i>				<i>J. O. Aug - Company Split</i>					
STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ NO	NO OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp	Grab				
48902	Acid plant NO.2	5/21/78	1600	(Sample Tag) 1892		✓	01	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 75ml
48902	Acid plant NO.2	5/21/78	1845	1891		✓	02	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 98ml
48902	Acid plant NO.2	5/22/78	1255	1893		✓	03	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 99ml
48902	Acid plant NO.2	5/22/78	1525	1894		✓	04	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 77ml
48902	Acid plant NO.2	5/22/78	1835	1895		✓	05	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> 490ml
48902	Acid plant NO.2	5/22/78	1845	1898		✓	06	1	H <sub>2</sub> O <sub>2</sub> Blank 480ml

Relinquished by: (Signature) <i>Timothy R. O'Aug</i>		Received by: (Signature) <i>Jim Estalling FOR ASA</i>		Date/Time <i>5/23/78 1839</i>
Relinquished by: (Signature)		Received by: (Signature) <i>REP. BUNKER HILL</i>		Date/Time
Relinquished by: (Signature)		Received by: (Signature)		Date/Time
Relinquished by: (Signature)		Received by Mobile Laboratory for field analysis. (Signature)		Date/Time
Dispatched by (Signature)		Date/Time	Received for Laboratory by:	Date/Time
Method of Shipment.				

## CHAIN OF CUSTODY RECORD

Company split

## Cum gratia Split

SURVEY Bunker Hill Co.				SAMPLERS: (Signature) <i>J. O'Leary - Company Spint</i>					
STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ NO	NO OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp	Grab				
8902	Acid Plant NO. 2	5/23/78	1330	(Sample Type) NO. 1	✓	07	<sup>no</sup> 1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> <sup>490 ml</sup>	
8902	Acid Plant NO. 2	5/23/78	1708	1812	✓	08	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> <sup>summl</sup>	
8902	Acid Plant NO. 2	5/23/78	1915	1814	✓	09	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> <sup>490 ml</sup>	
8902	Acid Plant NO. 2	5/23/78	1725	1814	✓	10	1	H <sub>2</sub> O <sub>2</sub> Blanks <sup>490 ml</sup>	
8902	Acid Plant NO. 2	5/24/78	1250	1824	✓	11	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> <sup>490 ml</sup>	
8902	Acid Plant NO. 2	5/24/78	1258	1825	✓	12	1	H <sub>2</sub> O <sub>2</sub> Blanks <sup>490 ml</sup>	
18902	Acid Plant	5/24/78	1712	1830	✓	13	1	H <sub>2</sub> O <sub>2</sub> /SO <sub>2</sub> <sup>490 ml</sup>	

Relinquished by (Signature) <i>Timothy J. O'Leary</i>	Received by (Signature) <i>Lynn Estabrook FOR ASA</i>	Date/Time <i>5/25/78 0952</i>
Relinquished by: (Signature)	Received by: (Signature) REP BUNKER HILL	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by (Signature)	Received by Mobile Laboratory for field analysis. (Signature)	Date/Time
Dispatched by (Signature)	Date/Time	Received for Laboratory by:
		Date/Time

Method of Shipment:



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Office Of Enforcement  
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Building 53, Box 25227, Denver Federal Center  
Denver, Colorado 80225

*Company Split*

**CHAIN OF CUSTODY RECORD**

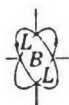
SURVEY <i>Bunker Hill Co</i>	SAMPLERS (Signature) <i>J. O'Quinn - Company Split</i>
------------------------------	--

STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ NO	NO OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp	Grab				
48901	Acid Plant NO.1	5/23/78	1218	18	18	✓	09	1	H <sub>2</sub> O <sub>2</sub> / SO <sub>2</sub> <i>480 ml</i>
48901	Acid Plant NO.1	5/23/78	1525	18	20	✓	<del>10</del> 10	1	H <sub>2</sub> O <sub>2</sub> / SO <sub>2</sub> <i>480 ml</i>
48901	Acid Plant NO.1	5/23/78	1540	18	22	✓	11	1	H <sub>2</sub> O <sub>2</sub> Blank <i>480 ml</i>
48901	Acid Plant NO.1	5/23/78	1900	18	24	✓	12	1	H <sub>2</sub> O <sub>2</sub> / SO <sub>2</sub> <i>510 ml</i>
48901	Acid Plant NO.1	5/24/78	1225	18	32	✓	13	1	H <sub>2</sub> O <sub>2</sub> / SO <sub>2</sub> <i>480 ml</i>
48901	Acid Plant NO.1	5/24/78	1235	18	34	✓	14	1	H <sub>2</sub> O <sub>2</sub> Blank <i>525 ml</i>
48901	Acid Plant NO.1	5/24/78	1605	18	34	✓	15	1	H <sub>2</sub> O <sub>2</sub> / SO <sub>2</sub> <i>480 ml</i>
48901	Acid Plant NO.1	5/24/78	1810	18	38	✓	16	1	H <sub>2</sub> O <sub>2</sub> / SO <sub>2</sub> <i>480 ml</i>

Relinquished by (Signature) <i>Timothy R O'Quinn</i>		Received by: (Signature) <i>Wm E. ... for ASA</i>		Date/Time <i>5/25/78 0950</i>
Relinquished by (Signature)		Received by: (Signature) <i>PER BUNKER HILL</i>		Date/Time
Relinquished by: (Signature)		Received by: (Signature)		Date/Time
Relinquished by: (Signature)		Received by Mobile Laboratory for field analysis (Signature)		Date/Time
Dispatched by (Signature)	Date/Time	Received for Laboratory by:		Date/Time
Method of Shipment.				

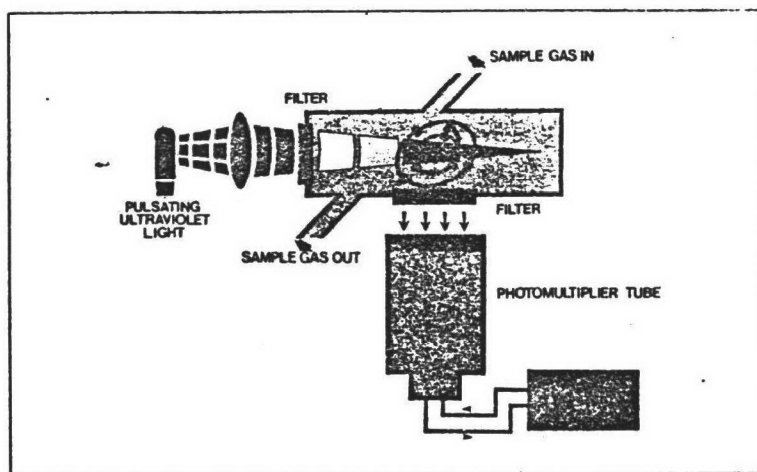
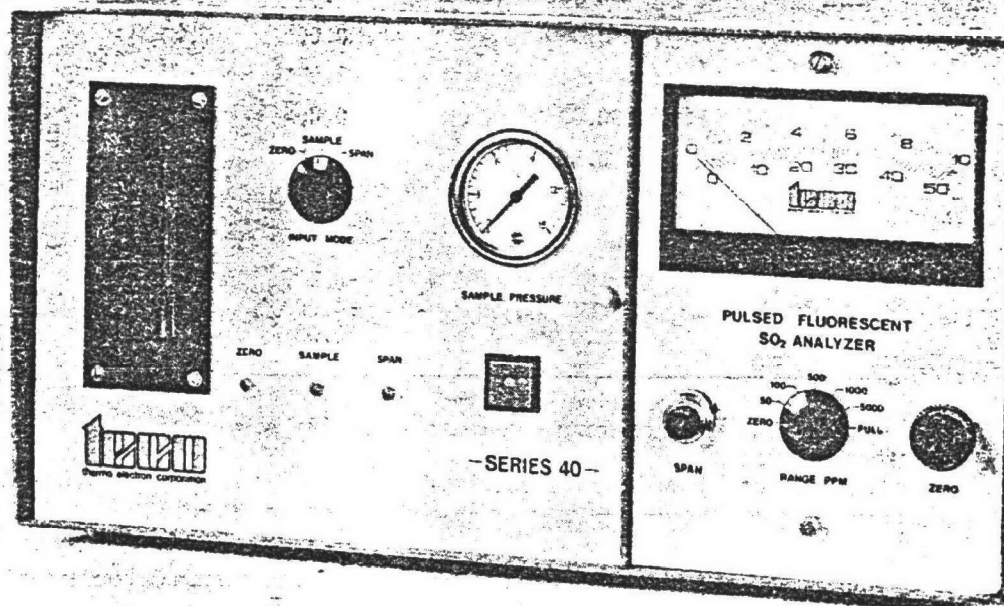
APPENDIX E  
TECO ANALYZER DESCRIPTION\*

\* Obtained from Instrumentation for Environmental Monitoring published by the Lawrence Berkeley Laboratory, University of California, Berkeley, California.



Stationary Source Monitor

Model 40 SO<sub>2</sub> Analyzer



Class

Stationary

Principle  
of Operation

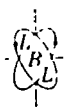
Pulsed Ultraviolet Fluorescence: Gas sample is illuminated by a pulsed xenon lamp attenuated with a monochromatic UV filter. The excited SO<sub>2</sub> molecules subsequently emit longer wavelength UV radiation which is transmitted through a guard filter to a photomultiplier. The output signal from the detector is linear with SO<sub>2</sub> concentration. Optional sulfur converter oxidizes H<sub>2</sub>S and other reduced sulfur compounds to SO<sub>2</sub> by means of a low-temperature catalytic process.

Lower Detectable  
Limit

1 ppm

Range

5000 ppm



## Interferences

INTERFERENCE DATA SUMMARY		
Interferent	Concentration ppm	Interference Equiv., ppm
H <sub>2</sub> S	0.100	0.00 <sup>g</sup>
NO <sub>2</sub>	0.500	0.00 <sup>g</sup>
NO	1,000.0	2.0 <sup>a,g</sup>
CO <sub>2</sub>	750.00	0.00 <sup>g</sup>
O <sub>3</sub>	0.500	0.00 <sup>g</sup>
M-Xylene	0.194	0.005 <sup>g</sup>
CO	50.0	0.005 <sup>g</sup>
		-0.005 <sup>*</sup>
		-0.003 <sup>†</sup>
		0.00 <sup>‡</sup>

<sup>\*</sup>Observed at 95% relative humidity.<sup>a,g</sup>  
<sup>†</sup>Calculated from quenching constants,<sup>f</sup> for 80% relative humidity.<sup>g</sup>  
<sup>‡</sup>Observed at 80% relative humidity.<sup>g</sup>

## Multiparameter Capability

SO<sub>2</sub> (H<sub>2</sub>S and other reduced sulfur compounds, with optional converter)

## Sampling

Method: Extractive. Gas sample is dehumidified with a permeation dryer  
Volume: 0.4-1 liter/min (1-2 scfh)  
Maximum Temperature Input: 50°C  
Collection Efficiency: 10%

## Performance

Accuracy: ± 0.5% relative to calibration gas<sup>a,c</sup>  
Precision: ± 0.5% full scale  
Repeatability: ± 0.5%  
Linearity: ± 1% full scale  
Noise: 0.5%  
Lag Time: 10 sec<sup>f</sup>  
Rise Time: 4 sec (90%)  
Retention Time:  
Fall Time: 4 sec (90%)  
Zero Drift: <1% in 7 days,<sup>a</sup> 1% in 24 hrs<sup>e</sup>  
Span Drift: <1% in 7 days,<sup>a</sup> 2% in 24 hrs<sup>e</sup>

## Operation

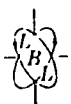
Ambient Temperature Range: 0°C to 40°C  
Temperature Compensation: None required  
Relative Humidity Range: 0-100%  
Calibration: External  
Procedure:  
Warm-up Time: 20 min.  
Unattended Period: 7 days to 1 month, depending on frequency of calibration  
Maintenance: Replacement of particulate filter. Xe lamp life is over 1 year during continuous operation.

## Requirements

Power: 150 watts at 115V AC (60Hz)  
Weight: 40 pounds  
Dimensions: 17 "W x 9"H x 23"D

## Features

Output: Analog display, also 0.1 V output  
Training: No special training required. Instruction manual included  
Options: sulfur converter (converts H<sub>2</sub>S) and other reduced sulfur compounds to SO<sub>2</sub>. External timer for automating sequence of zero, span, and measurement.



References

- (a) Manufacturer's Brochure BA-1-20M-675.
- (b) Schwartz, F. P., Okabe, M., and Whittaker, J. K., "Fluorescence Detection of Sulfur Dioxide in the Air at the Parts per Billion Level", *Anal. Chem.* 46, 1024-1028 (1974).
- (c) W. J. Mager, D. J., and Helm, D. A. "Source Level SO<sub>2</sub> Analysis via Pulsed Fluorescence," Report *ISA Aid 74402*, pp. 9-18, presented at APCA Meeting, Pittsburgh, Pennsylvania (August 1974).
- (d) Zollner, W. J., personal communication, (July 9, 1975).
- (e) Shen, T. T., *Chem. Eng.* 82, 109 (May 26, 1975).
- (f) Okabe, H., Splittsone, P. L., and Ball, J. J., *J. APCA* 23, 514 (1973).
- (g) Warner Carlson, private communication, June 25, 1976.

Cost<sup>f</sup>

Model 40: \$5950; catalytic sulfur converter: \$2000; Timer: \$100-800.00.

Remarks

The pulse length of the Xe source is approx. 10  $\mu$ sec. Advantages of the Xe arc lamp include its long working life compared to other UV sources. The range limitation for this SO<sub>2</sub> monitor (Ref. C) is the result of absorption "self" quenching by SO<sub>2</sub>, which begins to become important at levels above 5000 ppm. Other workers (Ref. b) observed self quenching, at concentrations on the order of 1600 ppm SO<sub>2</sub>, using a different excitation source. Dehumidification of gas stream minimizes SO<sub>2</sub> fluorescence quenching by water (b,c).

"In situations where more accurate measurements are desired, emission spectrographic techniques (Model 40) should be employed."<sup>e</sup>

Address

Thermo Electron Corporation (TECO)  
Environmental Instruments Division  
85 First Avenue  
Waltham, Massachusetts 02154  
(617) 890-8700

APPENDIX F  
CALIBRATION DATA

## NEIC Procedure for Pitot Tube Calibration

### Introduction

The Type-S pitot tube is used by NEIC to measure stack gas velocity during source sampling. The pitot tube coefficient ( $C_p$ ) of this instrument is determined by calibration against a traceable National Bureau of Standards (NBS) standard pitot tube. The Type-S pitot tube is calibrated on a probe sheath with a  $\frac{1}{2}$  inch diameter nozzle attached. All pitot tubes are calibrated from 305 m/min (1000 ft/min) to 1524 m/min (5000 ft/min). Pitot tubes used during tests will subsequently be recalibrated at a minimum of 3 points within the velocity range observed during testing. Tubes which have been damaged or suspected of being damaged during field use will be recalibrated over the entire range (i.e. 305 to 1524 m/min).

### I. Equipment Required

A. Flow System - Calibration is performed in a flow system meeting the following minimum requirements:

- (1) The air stream is confined in a well-defined cross sectional area, either circular or rectangular. The minimum size is 30.5 cm (12 inches) diameter for circular ducts and at least 25 cm (10 inches), as the shortest dimension for rectangular ducts.
- (2) Entry ports provided in the test section, shall be a minimum of 8 duct diameters downstream and 2 diameters upstream of any flow disturbance, e.g. bend, expansion, contraction, opening, etc.

- (3) The flow system must have the capacity to generate over the range of 305 m to 1524 m (1000 ft. - 5000 ft.)/min. Velocities in this range must be constant with time to guarantee steady flow during calibration.

B. Calibration Standard

A standard type pitot tube either calibrated directly by N.B.S. or traceable to an N.B.S. standard shall be the calibration standard.

C. Differential Pressure Gauge

An inclined or expanded scale manometer shall be used to measure velocity head ( $\Delta P$ ). Such gauges shall be capable of measuring  $\Delta P$  to within  $\pm 0.13$  mm (0.005 inches)  $H_2O$ . A micro-manometer capable of measuring with 0.013 mm (0.0005 in)  $H_2O$  will be used to measure  $\Delta P$  of less than 13 (0.5")  $H_2O$ .

D. Pitot Tube Lines

Flexible lines made of tygon or similar tubing shall be used.

E. Thermometer

A mercury in glass or other type thermometer checked against a mercury in glass thermometer is considered suitable.

F. Barometer

A mercury column barometer shall be available to determine atmospheric pressure.

II. Physical Check

1. The openings are sharp and do not have a rolled edge.
2. The impact planes of sides A & B are perpendicular to the Traverse Tube axis [Figure 2].



3. The impact planes are parallel to the longitudinal tube axis [Figure 3].

### III. Calibration Procedure

The Type-S pitot tube shall be assigned an identification number. The first digit of the number is the effective length of the tube, followed by a dash and consecutive numbers for the number of tubes of the same effective length. i.e. 5-1 signifies a five foot pitot tube and is the number one tube. Calibration proceeds as follows.

- A. Fill manometer with clean oil of the proper specific gravity. Attach and leak check all pitot tube lines.
- B. Level and zero monometer.
- C. Position the standard pitot tube in the test section at the calibration point. If the flow system is large enough and does not interfere with the Type-S tube the standard tube may be left in the system.
- D. Insert the Type-S tube into the flow system.
- E. Checks for the effect of turbulence are made as follows:
  1. Read  $\Delta P$  on both Type-S and standard pitot tubes with the standard pitot tube in place and compare with readings when the standard tube is withdrawn from system.
  2. Read  $\Delta P$  on the Type-S tube at centerline of flow system, then take readings while moving the tube to the side of the system. This will define the boundary turbulence layer.
  3. Position the Type-S tube so that their impact openings are perpendicular to the duct cross sectional area and

check for null (zero) reading. Absence of a null reading at this position indicates non-laminar flow conditions.

- F. Read  $\Delta P_{std}$  and record on data table.
- G. With the Type-S "A" leg orientated into the flow read  $\Delta P_s$  and record on data table.
- H. Repeat steps F and G until three sets of velocity data have been obtained.
- I. Remove Type-S pitot tube and rotate probe nozzle until it aligns with side "B" impact openings.
- J. Insert the Type-S pitot tube and proceed as in steps F through H.
- K. Adjust flow system to new velocity and repeat F-J.
- L. Record air temperature in the test system and barometric pressure during testing.

#### IV. Calculations

1. At each "A"-side and "B"-side velocity setting, calculate the three values of  $C_p$  (s) as follows:

$$C_{ps} = C_{p \text{ std}} \sqrt{\frac{\Delta P_{\text{std}}}{\Delta P_s}}$$

Where:

$C_{ps}$  - Type-S pitot tube coefficient

$C_{p \text{ std}}$  - Standard pitot tube coefficient (NBS)

$\Delta P_{\text{std}}$  - Velocity head, measured by Standard pitot tubing inches  $H_2O$

$\Delta P_s$  - Velocity head, measured by the Type-S pitot tube, inches  $H_2O$

2. Calculate  $\bar{C}_p$ , the average (mean of the three  $C_p$ (s) values.

3. For each  $\bar{Cp}$  calculated in step 2, calculate  $\sigma$ , the average deviation from the mean as follows:

$$\sigma(\text{Side "A" or "B"}) = \frac{1}{3} \frac{Cp (s) - \bar{Cp} (A \text{ or } B)}{3}$$

4. The pitot is acceptable if:

- (a) The "A" and "B" side average deviations calculated by equation 2 are  $\leq 0.01$ .
- (b) The difference of the "A" and "B" sides  $\bar{Cp}$  calculated by equation 1 is  $\leq 0.01$  for each individual velocity.

5. Calculate the test section velocity as follows:

$$\bar{V} = KCp \sqrt{\frac{T \bar{\Delta P} \text{ std}}{PM}}$$

Where:

$\bar{V}$  = Average test-section velocity, ft/min

K = 5130 (constant)

Cp = Coefficient of standard pitot tube

T = Temperature of gas stream  $^{\circ}\text{R}$

P = Barometric pressure, inches Hg

M = Molecular weight of air = 29.0

$\bar{\Delta P} \text{ std}$  = Average of the three standard pitot tube readings, inches  $\text{H}_2\text{O}$

#### V. Record Keeping

Flow system data and information on each pitot tube shall be recorded in a bound book.

The flow system data shall include:

1. The tunnel cross-sectional area and length up-stream and down-stream of the test site (ft.) from disturbances.

2. Time tunnel used (hrs)
3. Air temperature (<sup>0</sup>F) in flow system and barometric pressure (inches Hg).
4. All checks for turbulence and flow distribution.
5. Velocity range (ft/min).

The pitot tube information shall include:

1. I.D. number
2. Checks for physical damages, errors noted and modifications.
3. Dates and surveys pitot tubes were used.
4. Date of calibrations, coefficient and dates of re-calibration.

The calibration records will be kept on file at NEIC. Copies of the appropriate calibration dates will be furnished for each source test project.

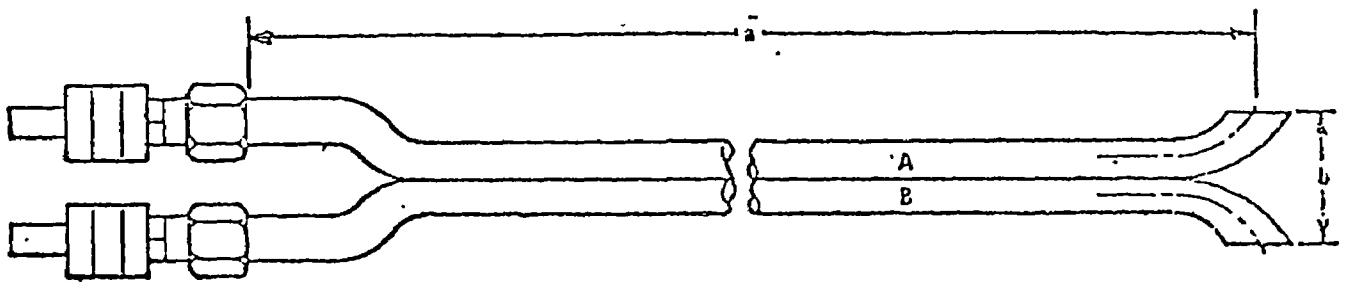


Figure 1. Measurement of Type-S pitot tube length (dimension "a") and impact-plane separation distance (dimension "b").

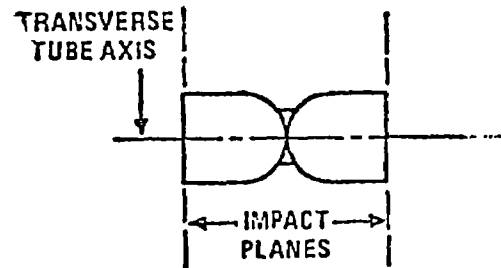


Figure 2. Type-S pitot tube, end view; impact-opening planes perpendicular to transverse tube axis.

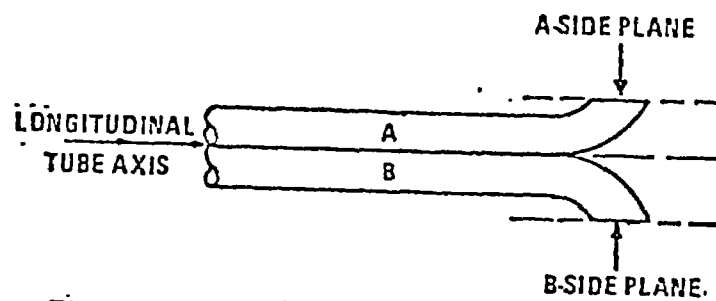


Figure 3. Type-S tube, top view; impact-opening planes parallel to longitudinal tube axis.

From "A TYPE-S PITOT TUBE CALIBRATION STUDY" by  
Robert F. Vollaro, October 15, 1975

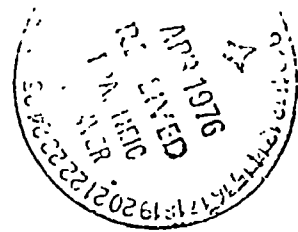
U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

WASHINGTON, D.C. 20234

March 24, 1976

NEM:pg  
TN G-42979  
213.08  
2130608



## REPORT OF CALIBRATION

on

Airflow Pitot-Static Tube  
12" x 4 mm

submitted by

Environmental Protection Agency  
National Enforcement Investigations Center  
Denver Federal Center  
Denver, Colorado 30225

Reference: Order No. WV-6-99-0516-H dated February 23, 1976.

The calibration was performed in the five-foot by seven-foot rectangular test section of the NBS closed-circuit dual test section wind tunnel. The tunnel provides an essentially uniform air stream with a very low turbulence intensity. The tube under test was inserted into the air stream through a hole in the tunnel wall and held in place by a clamping arrangement with all fittings outside the tunnel. The tube was aligned with the flow and positioned so that the static holes were approximately 8-1/2 inches from the tunnel sidewall. The boundary layer on the tunnel wall was approximately 1.6 inches thick.

Calibration of the tube consisted of determining the calibration factor  $K$  where  $K$  is defined as the ratio of the differential pressure indicated by the tube under test to the differential pressure indicated by the NBS laboratory standard. This was done by a direct comparison in which the tube under test and the NBS tube were mounted in the tunnel, 16 inches apart, and at the same distance from the tunnel sidewall.

The calibration factor  $K$  for a tube of this type may be dependent on the Reynolds number per unit length,  $V/\nu$ , where  $V$  is the air speed and  $\nu$  is the kinematic viscosity. This parameter is therefore given in the attached table, along with the corresponding values of  $V$  in the units requested, as the properties of the gas in which this instrument is used may be an important consideration. The values of  $K$  tabulated are the average of four independent determinations at each of the corresponding values of  $V/\nu$ .

For the Director,

P. S. Klebanoff, Chief  
Aerodynamics Section  
Mechanics Division, IBS

Table 1

Airflow Pitot-Static Tube  
12" x 4 mm

Reynolds Number Per Meter $V/ \times 10^{-3}, m^{-1}$	True Air Speed m/s	K
0.046	0.7	1.09
0.072	1.1	1.0
0.098	1.5	0.97
0.124	1.9	0.98
0.182	2.8	0.99
0.243	3.8	0.98
0.395	6.2	0.990
0.553	8.6	0.992
0.868	13.6	0.997
1.181	18.5	0.998
1.489	23.4	0.999
1.798	28.3	0.998
2.111	33.4	0.998
2.423	38.5	0.999
3.027	48.6	0.998

US Environmental Protection Agency  
National Enforcement Investigations Center-Denver

Calibration Pitot Tube: ID Number N1351 Cp 99  
Type-S Pitot Tube ID Number: 5-2 - 72.5

$T = 72^\circ\text{F}$   
 $P_n = 629 \text{ mm of Hg}$   
 $(24.78 \text{ in})$

[illegible]

During Pitot Calibration:

probe sheath attached NO  
nozzle attached NO  
sampling isokinetically

Performed By: J. Kellert  
A. Pausik

Calibration Date: 4/19/78



# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-2

Calibration Date 6/8/78

By A. Prink

Checked By K. L. Strauss

[illegible]

Jul 15, 1976

## National Enforcement Investigations Center-Denver

5-2

$$P_A = 631 \text{ mm of Hg}$$

$$(24.86 \text{ In})$$
$$T_A = 71.5^\circ F$$
[illegible]

sampling isokinetically —

A. Pringle  
J. Wilson

Calibration Date: 6/8/78

Calibration Pitot Tube: ID Number NBS Cp 0.99  
Type-S Pitot Tube ID Number: 5-2

Calibration Pitot Tube: ID Number NBS Cp 0.99  
Type-S Pitot Tube ID Number: 5-2

Type-S Pitot Tube ID Number: 5-2

During Pitot Calibration:  
probe sheath attached Yes  
nozzle attached Yes  
sampling isokinetically No

Calibration Date: 2-23-77

Leg Average Cp

Environmental Protection Agency  
National Enforcement Investigations Center-Denver

Libration Pitot Tube: ID Number 135-1 Cp .99  
-S Pitot Tube ID Number: 5-3

$T = 69^\circ\text{F}$   
 $P = 628 \text{ mm of Hg}$   
 (2474 in) |

$\Delta P$ Standard Pitot	$\Delta P$ S-Type Pitot		$C_p$		Comments
	A leg	B leg	A	B	
2.00	310	305	.795	.802	
	310	310	.795	.795	
2.00	315	305	.789	.802	
1.50	235	235	.791	.791	
	235	240	.791	.783	
1.50	235	235	.791	.791	
1.00	160	165	.783	.771	
	165	165	.771	.771	
1.00	160	165	.783	.771	
0.760	1.25	1.20	.772	.788	
	1.25	1.25	.772	.772	
0.760	1.25	1.20	.772	.788	
0.500	0.760	0.750	.803	.808	
	0.760	0.750	.803	.808	
0.500	0.760	0.750	.803	.808	
0.250	0.400	0.400	.783	.783	Micro Manometer
	0.400	0.400	.783	.783	
0.250	0.400	0.400	.783	.783	
0.060	0.100	0.100	.767	.767	
	0.100	0.100	.767	.767	
0.060	0.100	0.100	.767	.767	
			.784	.786	Leg Average $C_p$

ing Pitot Calibration:  
probe sheath attached YES  
nozzle attached YES  
sampling isokinetically —

Calibrated By: J. Smith Calibration Date: 3/8/18  
10' High

# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-3.

Calibration Date 3/8/78

By A. Quirk

Checked By R. J. [Signature]

$\Delta P$ Std Tube	$V$ (Test Section)	$C_P$		$\sigma$		diff
		A	B	A	B	
200	6155.65 ✓	.793	.800	.003	.003	.007
1.50	5330.95 ✓	.791	.788	.000	.004	.003
1.00	4352.70 ✓	.779	.771	.005	.000	.008
0.760	3794.60 ✓	.772	.783	.000	.007	.011
0.500	3077.83 ✓	.803	.808	.000	.000	.005
0.250	2176.35 ✓	.783	.783	.000	.000	.000
0.060	1066.19 ✓	.767	.767	.000	.000	.000

Calibration Pitot Tube: ID Number 135-1 Cp .99  
Type-S Pitot Tube ID Number: 5-3

[illegible]

During Pitot Calibration:  
probe sheath attached No  
nozzle attached No  
sampling isokinetically Yes

Performed By: A. J. Smith  
 Of 6110: 7

Calibration Date: 4/19/78

# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-3

Calibration Date 4/19/78

By H. H. H. H.

Checked By [Signature]

[illegible]

April 15, 1976

US'Environmental Protection Agency  
National Enforcement Investigations Center-Denver

Calibration Pitot Tube: ID Number NBS-1 Cp .99  
Type-S Pitot Tube ID Number: 5-3 0.9999

$$P_A = 630 \text{ mm of Hg}$$
$$(24.82 \text{ in})$$
$$T_A = 71.0^\circ \text{F}$$
[illegible]

During Pitot Calibration:  
probe sheath attached YES  
nozzle attached YES  
sampling isokinetically -

Performed By: J. Kusik  
T. Wilson

Calibration Date: 6/8/78



# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-3

Calibration Date 6/8/78

By J. P. Smith

Checked By J. H. Kesteven

Std Tube	V (Test Section)	CP		$\sigma$		diff
		A	B	A	B	
1.50	5342.51	.762	.767	.004	.000	.005
1.00	4362.14	.771	.771	.000	.000	.000
0.760	3802.83	.772	.772	.000	.000	.000
0.500	3084.50	.769	.769	.001	.001	.000
0.240	2137.00	.759	.760	.002	.002	.000
0.110	1446.76	.753	.753	.000	.000	.000

11 15, 1976

US Environmental Protection Agency  
National Enforcement Investigations Center-Denver

Calibration Pitot Tube: ID Number NBS-1

Cp .99

Type-S Pitot Tube ID Number: S-4

$P_a = 620 \text{ mm Hg}$   
 $T_a = 66^\circ \text{F}$

$\Delta P$ Standard Pitot	$\Delta P$ S-Type Pitot		Cp		Comments
	A leg	B leg	A	B	
2.00	3.15	3.20	.789	.783	
	3.15	3.15	.789	.789	
2.00	3.15	3.15	.789	.789	
1.50	2.35	2.30	.791	.799	
	2.35	2.25	.791	.808	*
1.50	2.30	2.25	.799	.808	
0.99	1.60	1.65	.779	.767	
	1.65	1.65	.767	.767	
0.99	1.65	1.65	.767	.767	
0.77	1.25	1.25	.777	.777	
	1.25	1.25	.777	.777	
0.77	1.25	1.25	.777	.777	
0.50	.810	.795	.778	.785	
	.805	.795	.780	.785	
0.50	.810	.795	.778	.785	
0.20	.340	.340	.759	.759	Micro-Manometer
	.335	.340	.765	.759	↓
0.20	.340	.350	.759	.748	
0.050	.090	.090	.738	.738	
	.090	.090	.738	.738	
0.050	.090	.090	.738	.738	
			.773	.773	Leg Average Cp

During Pitot Calibration:

probe sheath attached Yes  
nozzle attached Yes  
sampling isokinetically no

Performed By: Janeth R. O'Quinn  
A. Quinn

Calibration Date: 1/17/78

# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-4

Calibration Date 1/17/78

By A. Davis

Checked By Freeman Wilson

$\Delta P$ Std Tube	$V$ (Test Section)	$\overline{CP}$		$\sigma$		diff
		A	B	A	B	
2.00	6191.24	.789	.787	.000	.003	.003
1.50	5361.77	.794	.805	.004	.004	.000
0.99	4355.92	.771	.767	.005	.000	.005
0.77	3841.56	.777	.777	.000	.000	.000
0.50	3095.62	.779	.785	.001	.000	.001
0.200	1957.84	.761	.755	.003	.005	.002
0.050	978.92	.738	.738	.000	.000	.000

April 15, 1976

Calibration Pitot Tube: ID Number 135-1 Cp .99  
Type-S Pitot Tube ID Number: 5-4

$$P_A = 628 \text{ mm of Hg}$$
$$(24.74 \text{ in})$$

During Pitot Calibration:  
probe sheath attached     No      
nozzle attached     No      
sampling isokinetically     

Calibration Date: 4/19/78

# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-4

Calibration Date 4/19/78

By A. Pausitz

Checked By Jim Kellert

$\Delta P$ Std Tube	$V$ (Test Section)	$CP$		$\sigma$		diff
		A	B	A	B	
2.00	6184.78	.795	.793	.000	.003	.002
1.50	5356.18	.799	.799	.000	.000	.000
1.00	4373.30	.783	.783	.000	.000	.000
0.75	3787.39	.783	.783	.000	.000	.000
0.50	3092.39	.783	.783	.000	.000	.000
0.25	2186.65	.783	.783	.000	.000	.000

April 15, 1976

Calibration Pitot Tube: ID Number NBS-1 Cp .99  
Type-S Pitot Tube ID Number: 5-4

$P_A = 631 \text{ mm of Hg}$   
(24.86 in.)  
 $T_A = 71.5^\circ \text{F}$

During Pitot Calibration:  
 probe sheath attached NO  
 nozzle attached NO  
 sampling isokinetically -

Calibration Date: 6/8/78

# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-4

Calibration Date 6/8/78

By J. Grubbs

Checked By [Signature]

[illegible]

April 15, 1976

US Environmental Protection Agency  
National Enforcement Investigations Center-Denver

Calibration Pitot Tube: ID Number IVBS-1 Cp .99  
Type-S Pitot Tube ID Number: 5-6  $P_s = 620 \text{ mmHg}$

$P_g = 620 \text{ mmHg.}$

$$T_u = 66^{\circ}\text{F}$$

$\Delta P$ Standard Pitot	$\Delta P$ S-Type Pitot		$C_p$		Comments
	A leg	B leg	A	B	
2.00	3.20	3.20	.783	.783	
	3.20	3.20	.783	.783	
2.00	3.20	3.20	.783	.783	
1.50	2.35	2.35	.791	.791	
	2.35	2.35	.791	.791	
1.50	2.35	2.40	.791	.783	
1.00	1.65	1.65	.771	.771	
	1.65	1.65	.771	.771	
1.00	1.65	1.65	.771	.771	
0.74	1.25	1.25	.762	.762	
	1.25	1.25	.762	.762	
0.74	1.25	1.25	.762	.762	
0.49	0.80	0.81	.775	.770	
	0.80	0.80	.775	.775	
0.49	0.80	0.81	.775	.770	
0.260	0.425	0.420	.774	.779	Micro-MANOMETER ↓
	0.425	0.425	.774	.774	
0.260	0.425	0.425	.774	.774	
0.050	0.080	0.080	.783	.783	
	0.080	0.080	.783	.783	
0.050	0.080	0.080	.783	.783	
			0.777	0.776	Leg Average $C_p$

During Pitot Calibration:

probe sheath attached Yes

nozzle attached yes

sampling isokinetically —

Performed By: L. H. Smith

Calibration Date: 1/17/78

James E. Keller



# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-6.

Calibration Date 1/17/78

By James E. Kellustas

Checked By A. Duvik

$\Delta P$ d Tube	W (Test Section)	$\overline{CP}$		$\sigma$		diff
		A	B	A	B	
0.00	6191.24	0.783	0.783	0.00	0.00	0.00
50	5361.77	0.791	0.788	0.00	0.004	0.004
1.00	4377.87	0.771	0.771	0.00	0.00	0.00
2.74	3765.98	0.762	0.762	0.00	0.00	0.00
10.49	3064.51	0.775	0.772	0.00	0.002	0.002
22.60	2232.28	0.774	0.776	0.00	0.002	0.002
2.050	978.92	0.783	0.783	0.00	0.00	0.00

U.S. Environmental Protection Agency  
National Enforcement Investigations Center-Denver

Calibration Pitot Tube: ID Number NBS-1 Cp .99  
Type-S Pitot Tube ID Number: 5-6

$$P_A = 631 \text{ mm of Hg}$$
$$(2486 \text{ IN})$$
$$T_A = 71.0^\circ \text{F.}$$
[illegible]

During Pitot Calibration:

probe sheath attached YES  
nozzle attached YES  
sampling isokinetically —

Performed By: A. Lewis

Calibration Date: 6/8/78

# PITOT TUBE CALCULATION SHEET

Tube ID Number 5-6

Calibration Date 6/8/78

By J. J. [Signature]

Checked By J. K. [Signature]

[illegible]

Jul 15, 1976

NEIC PROCEDURE FOR  
CALIBRATION OF DRY GAS METER  
AND ORFICE METER

Dry gas meters are used in source testing units to accurately measure sample volumes drawn during testing. A critical orifice is also installed to provide a known sampling rate so that isokinetic sampling can be maintained. These units will be calibrated before and after each sampling trip.

Calibration is accomplished by making simultaneous total volume measurements with a calibrated wet test meter and the dry gas meter. The wet test meter must be previously calibrated from a primary standard. Calibration is performed follows:

1. Level wet test meter and adjust the water level to the proper point.
2. Level and zero the manometer on sampling control unit.
3. Leak check unit and air hoses at 15 inch Hg (leakage rate must be zero). Assemble vacuum line to the wet test meter.  
(Caution: NO NOT Leak Check System by Plugging the Inlet to the Wet Test Meter, this will cause internal damage to the meter.)
4. Warm up control unit by operating vacuum pump for 30 minutes with wet test meter connected in series.
5. Close the course valve and open the fine adjust (by-pass) valve.
6. Turn on vacuum pump, open course adjust valve and turn the fine adjust valve until manometer reads 0.5" H<sub>2</sub>O (ΔH).

7. Simultaneously record the dry gas meter reading, wet test meter reading and time. Record temperature of wet test meter, inlet and outlet temperature of dry gas meter and atmospheric pressure during the test run.
8. Allow pump to run until the wet test meter indicates exactly 5 cubic feet of air have passed through the system (10 cubic feet when a  $\Delta H$  of 2, 3 and 4 inches  $H_2O$  are used) and record time.
9. Repeat steps 5-9 for  $\Delta H$  of 1", 2" 3" and 4"  $H_2O$ .
10. Calibration record will be kept in a permanent file at NEIC.  
Copies will be made for field use.

#### Calculations

Calculate the accuracy of the dry gas meter ( $\gamma$ ) as follows:

$$\gamma = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6}) (t_w + 460)}$$

Where:

$V_w$  = Volume of gas metered, wet test meter, ft.<sup>3</sup>

$V_d$  = Volume of gas metered, dry gas meter, ft.<sup>3</sup>

$P_b$  = Atmospheric pressure, inches Hg

$t_d$  = Dry gas meter temperature, °F ( $\frac{t_{d \text{ in}} + t_{d \text{ out}}}{2}$ )

$t_w$  = Wet test meter temperature, °F

If  $\gamma \neq 1.00$  ( $\pm 0.02$ ) then gas meter will be taken to Public Service Company of Colorado gas meter shop for adjustment and/or repair.

$$\text{Orifice meter coefficient } (\Delta H @ = \frac{0.317 \Delta H}{P_b (t_d + 460)} \left[ \frac{(t_w + 460) \theta}{V_w} \right])$$

Where:

$V_w$  = Volume of gas metered, wet test meter,  $\text{ft}^3$

$P_b$  = Atmospheric pressure

$t_d$  = Dry gas meter temperature,  $^{\circ}\text{F}$

$t_w$  = Wet test meter temperature,  $^{\circ}\text{F}$

$\theta$  = Time elapsed, minutes

# Orifice Meter Calibration

Date 12/7/77

Box No. SGI - 1

Barometric pressure,  $P_b =$  24.19 in. Hg Dry gas meter No. 1  
At QUIL

Orifice Manometer setting, $\Delta H$ in. $H_2O$	Gas volume wet test meter $V_w$ , $ft^3$	Gas volume dry gas meter $V_d$ , $ft^3$	Temperature				Time $\theta$ , min	y	$\Delta H @$
			Wet Test	Dry gas meter					
			Meter $t_w$ , $^{\circ}F$	Inlet $t_{di}$ , $^{\circ}F$	Outlet $t_{do}$ , $^{\circ}F$	Average $t_d$ , $^{\circ}F$			
0.5	5	5.05	59	64	65	64.5	11.75	1.00	<del>1.92</del> 1.865 2.12W
1.0	5	5.07	59	69	69	69	8.43	1.00	<del>1.88</del> 1.897 2.12W
2.0	10	10.11	59	73	71	72	11.91	1.01	1.882
3.0	10	10.09	59	78	74	76	9.77	1.01	1.886
4.0	10	10.08	59	82	76	79	8.52	1.02	1.90
Average								1.01	1.8904

Calculations		y	$\Delta H @$
$\Delta H$	$\frac{\Delta H}{13.6}$	$\frac{V_w P_b (t_d + 460)}{V_d (P_b + \Delta H) (t_w + 460)}$	$\frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[ \frac{(t_w + 460) \theta}{V_w} \right]^2$
0.5	0.0368	$\frac{5 \times 24.19 (64.5 + 460)}{5.05 (24.19 + 0.0368) (59 + 460)}$	$\frac{0.0317 \times 5}{24.19 (64.5 + 460)} \left[ \frac{(59 + 460) 11.75}{5} \right]^2$
1.0	0.0737	$\frac{5 \times 24.19 (69 + 460)}{5.07 (24.19 + 0.0737) (59 + 460)}$	$\frac{0.0317 \times 1.0}{24.19 (69 + 460)} \left[ \frac{(59 + 460) 8.43}{5} \right]^2$
2.0	0.147	$\frac{10 \times 24.19 (72 + 460)}{(24.19 + 0.147) (59 + 460)}$	$\frac{0.0317 \times 2.0}{24.19 (72 + 460)} \left[ \frac{(59 + 460) 11.91}{10} \right]^2$
3.0	0.219	$\frac{10 \times 24.19 (76 + 460)}{10.09 (24.19 + 0.219) (59 + 460)}$	$\frac{0.0317 \times 3.0}{24.19 (76 + 460)} \left[ \frac{(59 + 460) 9.77}{10} \right]^2$
4.0	0.294	$\frac{10 \times 24.19 (79 + 460)}{10.08 (24.19 + 0.294) (59 + 460)}$	$\frac{0.0317 \times 4.0}{24.19 (79 + 460)} \left[ \frac{(59 + 460) 8.52}{10} \right]^2$

Where:  $V_w$  = Volume, wet test meter  
 $V_d$  = Volume Dry gas meter  
 $T_w$  = Temperature, Wet Test Meter  
 $T_d$  = Temperature, Dry Gas Meter  
 $P_b$  = Atmospheric Pressure, Inches Hg  
 $\theta$  = Time, minutes

Calibration by: A. P. Smith  
 Checked by: J. R. Wolf

Remarks: LEAK CHECK @ 15" Hg = 0.0 CFM

4/24/77 Calibration after repair by Scientific Glass  
 New Orifice installed J.R.W.

# Orifice Meter Calibration

Date 6/5/78

Box No. SCI #1

Barometric pressure,  $P_b =$  24.63 (Bld #53) in. Hg

Dry gas meter No. #1

Orifice Manometer setting, $\Delta H$ in. H <sub>2</sub> O	Gas volume wet test meter $V_w$ , ft <sup>3</sup>	Gas volume dry gas meter $V_d$ , ft <sup>3</sup>	Temperature				Time $\theta$ , min	$\gamma$	$\Delta H @$
			Wet Test Meter	Dry gas meter					
				Inlet	Outlet	Average			
0.5	5	5.005	62.0	75	77	76.0	11.98	1.02	1.88
1.0	5	5.000	62.0	78	79	78.5	8.54	1.03	1.90
2.0	10	10.080	62.0	79	82	80.5	12.03	1.02	1.88
3.0	10	10.075	62.0	84	80	82.0	9.87	1.02	1.89
4.0	10	10.040	62.0	86	81	83.5	8.61	1.02	1.91
Average								1.02	1.89

Calculations		$\gamma$	$\Delta H @$
$\Delta H$	$\frac{\Delta H}{13.6}$	$\frac{V_w P_b (t_d + 460)}{V_d (P_b + \Delta H) (t_w + 460)}$	$\frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[ \frac{(t_w + 460) \theta}{V_w} \right]^2$
0.5	0.0368	$\frac{5 \times 24.63 (76 + 460)}{5.005 (24.63 + 0.0368) (62 + 460)}$	$\frac{0.0317 \times 0.5}{24.63 (76 + 460)} \left[ \frac{(62 + 460) 11.98}{5} \right]^2$
1.0	0.0737	$\frac{5 \times 24.63 (78.5 + 460)}{5.000 (24.63 + 0.0737) (62 + 460)}$	$\frac{0.0317 \times 1.0}{24.63 (78.5 + 460)} \left[ \frac{(62 + 460) 8.54}{5} \right]^2$
2.0	0.147	$\frac{10 \times 24.63 (80.5 + 460)}{10.080 (24.63 + 0.147) (62 + 460)}$	$\frac{0.0317 \times 2.0}{24.63 (80.5 + 460)} \left[ \frac{(62 + 460) 12.03}{10} \right]^2$
3.0	0.219	$\frac{10 \times 24.63 (82 + 460)}{10.075 (24.63 + 0.219) (62 + 460)}$	$\frac{0.0317 \times 3.0}{24.63 (82 + 460)} \left[ \frac{(62 + 460) 9.87}{10} \right]^2$
4.0	0.294	$\frac{10 \times 24.63 (83.5 + 460)}{10.040 (24.63 + 0.294) (62 + 460)}$	$\frac{0.0317 \times 4.0}{24.63 (83.5 + 460)} \left[ \frac{(62 + 460) 8.61}{10} \right]^2$

Where:  $V_w$  = Volume, wet test meter

$V_d$  = Volume Dry gas meter

$T_w$  = Temperature, Wet Test Meter

$T_d$  = Temperature, Dry Gas Meter

$P_b$  = Atmospheric Pressure, Inches Hg

$\theta$  = Time, minutes

Calibration by: D. Smith

Checked by: T. Wilson

Remarks: LEAK CHECK @ 15" Hg = 0.00 CFM

4/24/77



# Orifice Meter Calibration

Date 12/6/77

Box No. SGI 2

Barometric pressure,  $P_b$  = in. Hg Dry gas meter No. 2  
2432 At Quail

Orifice Manometer setting, $\Delta H$ in. H <sub>2</sub> O	Gas volume wet test meter $V_w$ , ft <sup>3</sup>	Gas volume dry gas meter $V_d$ , ft <sup>3</sup>	Temperature				Time $\theta$ , min	$\gamma$	$\Delta H @$
			Wet Test Meter $t_w$ , °F	Dry gas meter					
				Inlet $t_{di}$ , °F	Outlet $t_{do}$ , °F	Average $t_d$ , °F			
0.5	5	5.05	62	74	77	75.5	12.05	1.01	1.91*
1.0	5	5.06	62	75	78	76.5	8.68	1.01	1.995
2.0	10	10.10	62	79	79	79	12.32	1.02	2.000
3.0	10	10.10	62	84	80	82	10.09	1.02	2.001
4.0	10	10.08	62	86	81	83.5	8.77	1.02	2.010
Average								1.017	1.984

Calculations		$\gamma$	$\Delta H @$
$\Delta H$	$\frac{\Delta H}{13.6}$	$\frac{V_w P_b (t_d + 460)}{V_d (P_b + \Delta H) (t_w + 460)}$	$\frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[ \frac{(t_w + 460) \theta}{V_w} \right]^2$
0.5	0.0368	$\frac{5 \times 24.32 (75.5 + 460)}{5.05 (24.32 + 0.0368) (62 + 460)}$	$\frac{0.0317 \times 0.5}{24.32 (75.5 + 460)} \left[ \frac{(62 + 460) 12.05}{5} \right]^2$
1.0	0.0737	$\frac{5 \times 24.32 (76.5 + 460)}{5.06 (24.32 + 0.0737) (62 + 460)}$	$\frac{0.0317 \times 1.0}{24.32 (76.5 + 460)} \left[ \frac{(62 + 460) 8.68}{5} \right]^2$
2.0	0.147	$\frac{10 \times 24.32 (79 + 460)}{10.10 (24.32 + 0.147) (62 + 460)}$	$\frac{0.0317 \times 2.0}{24.32 (79 + 460)} \left[ \frac{(62 + 460) 12.32}{10} \right]^2$
3.0	0.219	$\frac{10 \times 24.32 (82 + 460)}{10.10 (24.32 + 0.219) (62 + 460)}$	$\frac{0.0317 \times 3.0}{24.32 (82 + 460)} \left[ \frac{(62 + 460) 10.09}{10} \right]^2$
4.0	0.294	$\frac{10 \times 24.32 (83.5 + 460)}{10.08 (24.32 + 0.294) (62 + 460)}$	$\frac{0.0317 \times 4.0}{24.32 (83.5 + 460)} \left[ \frac{(62 + 460) 8.77}{10} \right]^2$

Where:  $V_w$  = Volume, wet test meter

$V_d$  = Volume Dry gas meter

$T_w$  = Temperature, Wet Test Meter

$T_d$  = Temperature, Dry Gas Meter

$P_b$  = Atmospheric Pressure, Inches Hg

$\theta$  = Time, minutes

Calibration by: J. P. Smith

Checked by: J. P. Smith

Remarks: LEAK CHECK @ 15" Hg = 0.0 CFM

\* Ran Twice

4/24/77

$V_d = 5.11$

$T_w = 62$

$T_d = 82$

$\theta = 12.08$

$$\gamma = \frac{5 \times 24.32 (82 + 460)}{5.11 (24.32 + 0.0368) (62 + 460)} = 1.01$$

$$\Delta H @ = \frac{0.0317 \times 0.5}{24.32 (82 + 460)} \left[ \frac{(62 + 460) 12.08}{5} \right]^2 = 1.91$$

calibration after refitting  
by Scientific glass,  
new orifice

# Orifice Meter Calibration

Date 6/5/78 Box No. SGI #2  
 Barometric pressure,  $P_b = \frac{24.61 (P_b \neq 53)}{\text{in. Hg}}$  Dry gas meter No. #2

Orifice Manometer setting, $\Delta H$ in. $H_2O$	Gas volume wet test meter $V_w$ , $ft^3$	Gas volume dry gas meter $V_d$ , $ft^3$	Temperature				Time $\theta$ , min	y	$\Delta H @$
			Wet Test Meter	Dry gas meter					
				Inlet	Outlet	Average			
0.5	5	5.05	64.0	69	73	71.0	12.15	1.00	1.97
1.0	5	5.05	64.5	71	73	72.0	8.72	1.00	2.03
2.0	10	10.05	65.0	77	75	76.0	12.30	1.01	2.00
3.0	10	10.03	65.0	80	77	78.5	10.11	1.01	2.02
4.0	10	10.02	65.5	83	79	81.0	8.81	1.02	2.04
Average								1.01	2.01

Calculations		y	$\Delta H @$
$\Delta H$	$\frac{\Delta H}{13.6}$	$\frac{V_w P_b (t_d + 460)}{V_d (P_b + \Delta H) (t_w + 460)}$	$\frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[ \frac{(t_w + 460) \theta}{V_w} \right]^2$
0.5	0.0368	$\frac{5 \times 24.61 (71 + 460)}{5.05 (24.61 + 0.0368) (64 + 460)}$	$\frac{0.0317 \times 0.5}{24.61 (71 + 460)} \left[ \frac{(64 + 460) 12.15}{5} \right]^2$
1.0	0.0737	$\frac{5 \times 24.61 (72 + 460)}{5.05 (24.61 + 0.0737) (64.5 + 460)}$	$\frac{0.0317 \times 1.0}{24.61 (72 + 460)} \left[ \frac{(64.5 + 460) 8.72}{5} \right]^2$
2.0	0.147	$\frac{10 \times 24.61 (76 + 460)}{10.05 (24.61 + 0.147) (65 + 460)}$	$\frac{0.0317 \times 2.0}{24.61 (76 + 460)} \left[ \frac{(65 + 460) 12.30}{10} \right]^2$
3.0	0.219	$\frac{10 \times 24.61 (78.5 + 460)}{10.03 (24.61 + 0.219) (65 + 460)}$	$\frac{0.0317 \times 3.0}{24.61 (78.5 + 460)} \left[ \frac{(65 + 460) 10.11}{10} \right]^2$
4.0	0.294	$\frac{10 \times 24.61 (81 + 460)}{10.02 (24.61 + 0.294) (65.5 + 460)}$	$\frac{0.0317 \times 4.0}{24.61 (81 + 460)} \left[ \frac{(65.5 + 460) 8.81}{10} \right]^2$

Where:  $V_w$  = Volume, wet test meter  
 $V_d$  = Volume Dry gas meter  
 $T_w$  = Temperature, Wet Test Meter  
 $T_d$  = Temperature, Dry Gas Meter  
 $P_b$  = Atmospheric Pressure, Inches Hg  
 $\theta$  = Time, minutes

Calibration by: J. Smith  
 Checked by: T. Wilson

Remarks: LEAK CHECK @ 15" Hg = 0.00 CFM

4/24/77

PROJECT NO. \_\_\_\_\_

THERMOCOUPLE CALIBRATION

Calibration Standard OVEN # 2

Pitot Tube I.D. No.	Calibration Temperature °F	Thermocouple Reading °F
2-1	261	261
3-1	258	258
* 5-1	258	243
5-2	266	266
5-3	258	258
5-4	263	263
5-5	259	256
8-1	259	257
8-2	261	259
10-1	248	248
* 10-2	269	261
10-3	250	250
10-4	268	265

Date 3/23/78

Calibrated By A. P. Smith

Check By SMC. noc 3-23-78

PROJECT NO. 489

THERMOCOUPLE CALIBRATION

Calibration Standard ASTM #4

Pitot Tube I.D. No.	Calibration Temperature °F	Thermocouple Reading °F
5-2	unable to calibrate - thermocouple was removed at Bunker Hill	
5-3	149	147
5-4	148	142
5-4	316	304
5-L	162	150
5-6	305	283

Date 6/14/78

Calibrated By M. Pruitt

Check By K. Johnston

ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF ENFORCEMENT  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER  
DENVER, COLORADO 80225

o Mr. Tim Osag  
Field Operations Branch

DATE May 18, 1978

THROUGH: Chief, Chemistry Branch



ROM R. C. Ross

UBJECT Results from Pre-Survey Analyses of SO<sub>2</sub> Calibration Gases - Bunker Hill Study

Background

SO<sub>2</sub> will be monitored by a continuous method during the Bunker Hill Study which will entail the use of SO<sub>2</sub> calibration gases in an air matrix. 40 CFR, No. 194, October 6, 1975, requires that these calibration gases be analyzed in triplicate two weeks prior to start-up of the continuous monitoring to demonstrate stability and accuracy of the gases. The method of analysis of these gases is directed by Method 6, 40 CFR 60.

Results

The results are presented on Table 1. The first column gives the cylinder designation. The second column gives the concentration stated by the manufacturer on the tank. The third column tabulates individual triplicate determinations at NEIC; the fourth column averages these triplicate determinations. The fifth column is the percent difference between the manufacturer's value and the average value determined at NEIC. The last column is the standard deviation among triplicate determinations relative to average concentration and reported in percent. These determinations were conducted between May 8 and May 10. A bias of minus 1% in these results may be expected because the titration determinations on QC standards were consistently about 1% low. Using the average % deviation from Table 1 as an estimate of the overall standard deviation, the overall precision of the measurements can be estimated to be + 4% at the 95% confidence level.

Methodology

Figure 1 shows the sampling train used to collect the gases. During sampling, gas was fed into the manifold at approximately 2 liters per minute. The excess sample not sampled passed through a soap bubble flow meter so that excess could be demonstrated at any time during a sampling run. Vacuum on the other end pulled the sample at a constant flow rate of 1.079  $\pm$  3.5% through the impingers. A rate meter (calibrated rotameter) was placed in line to demonstrate that the flow was constant. A drying tube with indicating Dryerite was placed before the rotameter so that the rotameter would be protected from moisture carried over from the impingers and also so that its calibration would apply to SO<sub>2</sub> in air on a dry gas basis.

Impinger 1 contained 15 ml of 80% isopropanol (IPA). Impingers 2, 3, and 4 contained 15 ml of 3%  $\text{H}_2\text{O}_2$ . After a run, the contents of impingers 2 and 3 were combined, brought to 100 ml in a volumetric flask, an aliquot withdrawn and titrated with standardized  $\text{BaCl}_2$  using 2-3 drops of thorin indicator and enough IPA added to the sample so that it was 80% IPA before titration.

The titrant,  $\text{BaCl}_2$ , was standardized against .02 N  $\text{H}_2\text{SO}_4$ . The  $\text{H}_2\text{SO}_4$  was standardized potentiometrically against 0.02 N  $\text{NaOH}$ . The  $\text{NaOH}$ , in turn, was standardized against 0.02 N potassium acid phthalate (primary standard grade).

Air flow rates through the train were regulated by means of a calibrated critical orifice. The vacuum on the end of the system was sufficient to establish a critical pressure ratio<sup>1</sup> of 0.25 on the orifice. The flow in the system was measured by means of a calibrated rotameter with round stainless steel float. Leak checks were made by attaching a vacuum gauge to the head of the impinger train, monitoring its stability at 10 inches mercury (vacuum) for 30 seconds. Flows were timed for 20.0 minutes by means of a stop watch.

Calibrations of the rotameter and orifice were done against a soap bubble flow meter using ambient air at 620 mm and 23°C.

### Discussion

Method 6 specifies a dry gas meter with an accuracy of  $\pm 2\%$  be used in the sampling train for gas volume measurements. With a dry gas meter, after all runs have been completed, the calibration of the meter must be shown to be within 5% of the initial calibration or all runs are voided.

From my own knowledge of small dry gas meters, they are inaccurate at flow rates much below several liters per minute. I talked to the area representative for Rockwell dry gas meters and he said that for Model T-110, which we have, the lowest flow rate where accuracy of  $\pm 2\%$  could be expected is 10 scfm per hour--this corresponds to 4.67 liters per minute. It was for this reason that a rotameter and orifice were used to measure flow rate--from which total sampling volume was calculated. A meter does exist with one full dial turn equal to 0.1 scfm but it is basically the same meter inside as the T-110. The discrepancy between the stated requirements in Method 6 for the dry gas meter vs what is possible at a flow rate of 1 liter/minute is what led to the choice of rotameter and critical orifice set-up for flow measurements.

During the course of sampling it was noted that between runs the height of the rotameter ball changed slightly. This seemed strange because the critical orifice should have maintained a constant flow rate. Late on the

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<sup>1</sup>Upstream pressure divided by downstream pressure

second day of analytical runs by hooking up a mercury manometer in-line and just before the rotameter it could be observed that by varying the volume of excess calibration gas fed into the manifold over the range used in the analytical determinations, pressure changes at the rotameter as high as 34 mm could be produced.

The observed readings on the rotameter ranged from 81.5 to 84 mm. An average pressure drop of 20 mm was observed over the later determinations. Therefore, corresponding to 82.75 mm on the rotameter and 597 mm pressure, the average flow rate of 1079 ml/min at 597 mm Hg was used in all of the calculations. This, after calculation, would account in an overall variability in the measurements of about  $\pm 3.5\%$ .

#### Quality Control

All standardizations were performed at least in duplicate. The  $\text{BaCl}_2$  in 80% IPA titrant was restandardized daily with no change. Five audit samples from EPA, RTP were analyzed during the course of the work. These concentrations were originally unknown to myself; results were as follows:

<u>Sample Number</u>	<u>Result, mg/dscm</u>	<u>True, mg/dscm</u>	<u>% Deviation</u>
5299	133	137	-2.9
4348	5080	5148	-1.3
2141	4320	4347	-0.6
7399	1392	1411	-1.3
8253	2570	2593	-0.9

Sample number 5299 required only 1.7 ml of titrant and should not be included in an assessment of accuracy since the limits of error on this titrant volume are an order of magnitude larger than on the other determinations including the other gas cylinder determinations reported here. On this basis, I had a consistent bias of approximately minus 1%.

Replicate titrations were performed during the sample analyses with results agreeing to better than 1%. Blanks of 3%  $\text{H}_2\text{O}_2$  were titrated with  $\text{BaCl}_2$  and results subtracted from sample values (blanks were almost negligible). IPA was checked for peroxide by the procedure given in Method 6 and found to be quite acceptable. 3%  $\text{H}_2\text{O}_2$  was made fresh each day of analysis.

To examine collection efficiency, during three of the runs involving the higher concentration calibration gases, the fourth impinger (Figure 1), filled with 3%  $\text{H}_2\text{O}_2$ , was analyzed separately after the test. When the contents were analyzed, no  $\text{SO}_2$  (as  $\text{SO}_4^{2-}$ ) was detected in the fourth impinger during any of these three determinations.

Leak checks were performed both before and after each sample run.

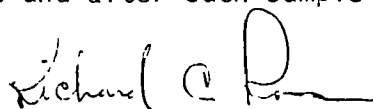
  
Richard C. Ross

Table 1

<u>Sample No.</u>	<u>Stated Conc. ppm</u>	<u>SO<sub>2</sub> Found ppm</u>	<u>Ave Found ppm</u>	<u>Percent Change from Stated Conc.</u>	<u>Relative SD*-%</u>
B-1 Scott	2490 $\pm$ 2%	2333			
B-1 Scott		2353	2365	- 5	1.4
B-1 Scott		2410			
B-2 Scott	2607 $\pm$ 2%	2396			
B-2 Scott		2481	2464	- 5.5	2.0
B-2 Scott		2516			
QC-1 Scott	997 $\pm$ 1%	919			
QC-1 Scott		947	927	- 7	1.5
QC-1 Scott		916			
B-3 Scott	2534 $\pm$ 2%	2602			
B-3 Scott		2326	2485	- 1.9	4.7
B-3 Scott		2528			
QC-3 Scott	4500 $\pm$ 1%	4560			
QC-3 Scott		4496	4510	+ 0.2	0.8
QC-3 Scott		4473			
A-2 Linde	957	1095			
A-2 Linde		1142	1130	+18	2.3
A-2 Linde		1155			
A-1 Linde	971	1118			
A-1 Linde		1118	1100	+13.3	2.2
A-1 Linde		1065			
A-3 Linde	950	955			
A-3 Linde		986	946	- 0.4	3.8
A-3 Linde		899			
C-2 Linde	4329	4599			
C-2 Linde		4625	4656	+ 7.6	1.4
C-2 Linde		4745			



Table 1  
-continued

<u>Sample No.</u>	<u>Stated Conc. ppm</u>	<u>SO<sub>2</sub> Found ppm</u>	<u>Ave Found ppm</u>	<u>Percent Change from Stated Conc.</u>	<u>Relative SD*-%</u>
C-1 Linde	4408	4578			
C-1 Linde		4618	4580	+ 4	0.6
C-1 Linde		4555			
C-3 Linde		4601			
C-3 Linde	4395	4734	4640	+ 5.7	1.3
C-3 Linde		4602			

\*SD = Standard Deviation

Ave = 2.0

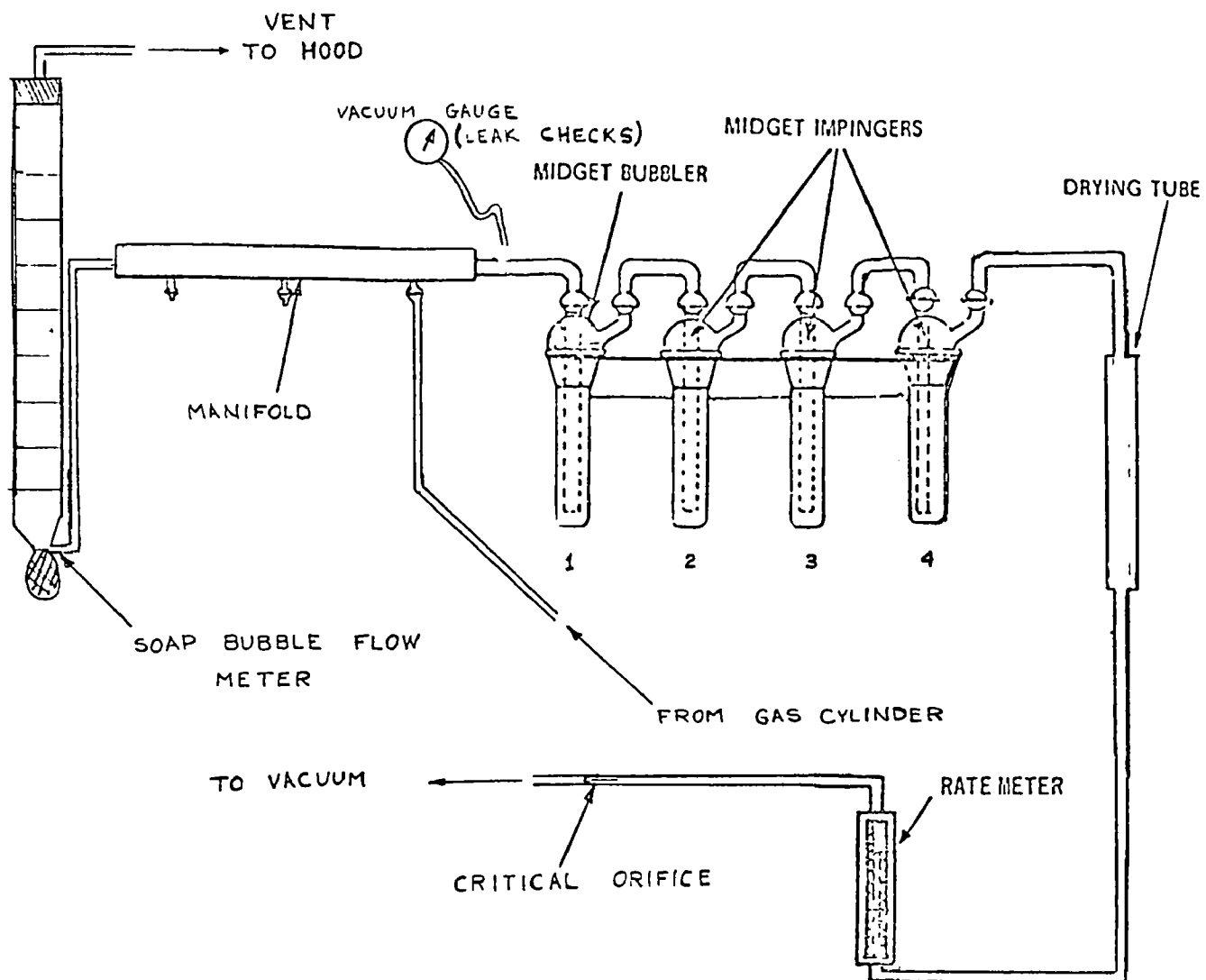


FIG 1 -  $\text{SO}_2$  sampling train.

ENVIRONMENTAL PROTECTION AGENCY  
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BUILDING 53, BOX 25227, DENVER FEDERAL CENTER  
DENVER, COLORADO 80225

TO Tim Osag  
Field Operations Branch

DATE June 27, 1978

FROM R. C. Ross

SUBJECT Post-Survey Analysis of SO<sub>2</sub> Calibration Gases - Bunker Hill

In accordance with the Bunker Hill study plan, the SO<sub>2</sub> calibration gases were reanalyzed after return to Denver. The method of analysis was directed by Method 6, 40 CFR 60. Table 1, attached, lists the gases analyzed, the suppliers stated concentration, individual and average concentrations as determined here, and finally the percent change of the concentrations found versus the supplies stated concentrations. This data was previously transmitted on June 20th; there are no changes shown in the results. Six replicate determinations of a gas standardized against NBS SRMS demonstrated a precision of analysis of  $\pm 2\%$  over a four day period.

#### Methodology

Figure 1 illustrates the sampling train used to collect the samples. A number 22 gauge needle was used to control the flowrate. The mercury manometer just before the critical orifice served to indicate any pressure changes. Rotameter 2 was used to indicate a constant flow throughout the sample runs. The drying tube was inserted to protect the rotameter and critical orifice from condensed water.

The fritted bubbler normally used in Method 6 in the first impinger was exchanged for an open type of bubble because of uncontrollable and excessive frothing at this impinger. Rotameter number 1 was placed in the system to show an excess of SO<sub>2</sub> at the sampling manifold.

Before and after each sample run, a soap bubble flow meter (SBFM) was connected to the head of the impingers to measure the flowrate through the system. Readings at rotameter 2 and at the Hg manometer were recorded before, during and after each run. Barometric pressure and room temperature were recorded for each run. Except for the RTP audit gas (which was made up in nitrogen) the system was calibrated against ambient air entering the system through the SBFM. For those runs involving the audit gas, the system was calibrated with nitrogen. Leak checks were performed for each sample run. The critical pressure ratio\* at the limiting orifice was checked and found to be  $< 0.4$ .

\*Downstream pressure divided by the upstream pressure.

Impinger 1 contained 15 ml 80% isopropanol (IPA) - water. Impingers 2, 3, and 4 contained 15 ml 3%  $\text{H}_2\text{O}_2$  solution freshly prepared each day. The combined solutions from impingers 2 and 3 were titrated against standardized  $\text{BaCl}$  after each run. Typically 10 minute runs with 15 minutes purge times were used.

### Discussion

The flowrates used were typically 1.05 liter per minute which for a 10 minute sample run would yield a 10 liter sample. Pressure at the mercury manometer stayed at  $25 \pm$  mm Hg below ambient pressure.

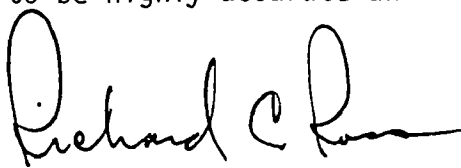
The results in table 1 reflect corrections made for pressure, temperature, water vapor and the amount of  $\text{SO}_2$  removed from the volumetric flowrate before passing through the metering critical orifice.

Reference solutions obtained from EPA, RTP indicate that the titer value used to obtain the results in table 1 is about 0.5% low. This is substantiated by the fact that although 0.01 equivalents of  $\text{BaCl}_2$  were added per liter of titrating solution the titer value used, determined by potentiometric standardization, was .00994 equivalents per liter.

Values obtained from post-survey determinations in some cases are slightly different from those obtained on the pre-survey determinations. Also the agreement between the manufacturers values and the post-survey value on the average is slightly better than before. The difference between the pre- and post values obtained is attributable to better control of the numerous variable effecting the measurements during the post-survey determinations.

### Quality Control

Reference solutions obtained from RTP demonstrate the titration procedure to be accurate to better than 1%. The results of the audit gas analyzed six times during these determinations had a standard deviation of less than 1%; using two standard deviations as a criteria the precision was better than 2%. The agreement between the average value and the stated value shows that the analytical results for this cylinder are consistent with the SRM's against which the audit gas was calibrated - thus the average result appears to be highly accurate and indicative of the other results in table 1.



R. C. Ross

cc: Meiggs  
Young

TABLE I

Gas Cylinder	Stated Conc. ppm	NEIC Measured Conc. ppm	Ave. Cone	% Deviation from stated
QC-1	997	974 1005	980	-1.2
QC-3	4500	975 4470 4539 4507	4500	+0.1
A-1	971	1063 1061 1062 1066	1060	+9.5
A-2	957	1048 1041 1039	1040	+9.0
A-3	950	950 958 970	960	+1
B-1	2490	2466 2442 2396 2441	2440	-2.2
B-2	2607	2544 2582 2505	2540	-2.4
B-3	2534	2430 2442 2469	2450	-3.4
C-1	4408	4614 4639 4610	4620	+4.8
C-2	4329	4539 4543 4610	4530	+4.6
C-3	4395	4609 4590 4579	4590	+4.5
RTP Audit Gas III 6/12 6/12 6/13 6/14 6/14 6/15	2268	2306 2269 2288 2251 2262 2242	2370	+0.1 s = 24 ppm 2s = 48 ppm (2%)

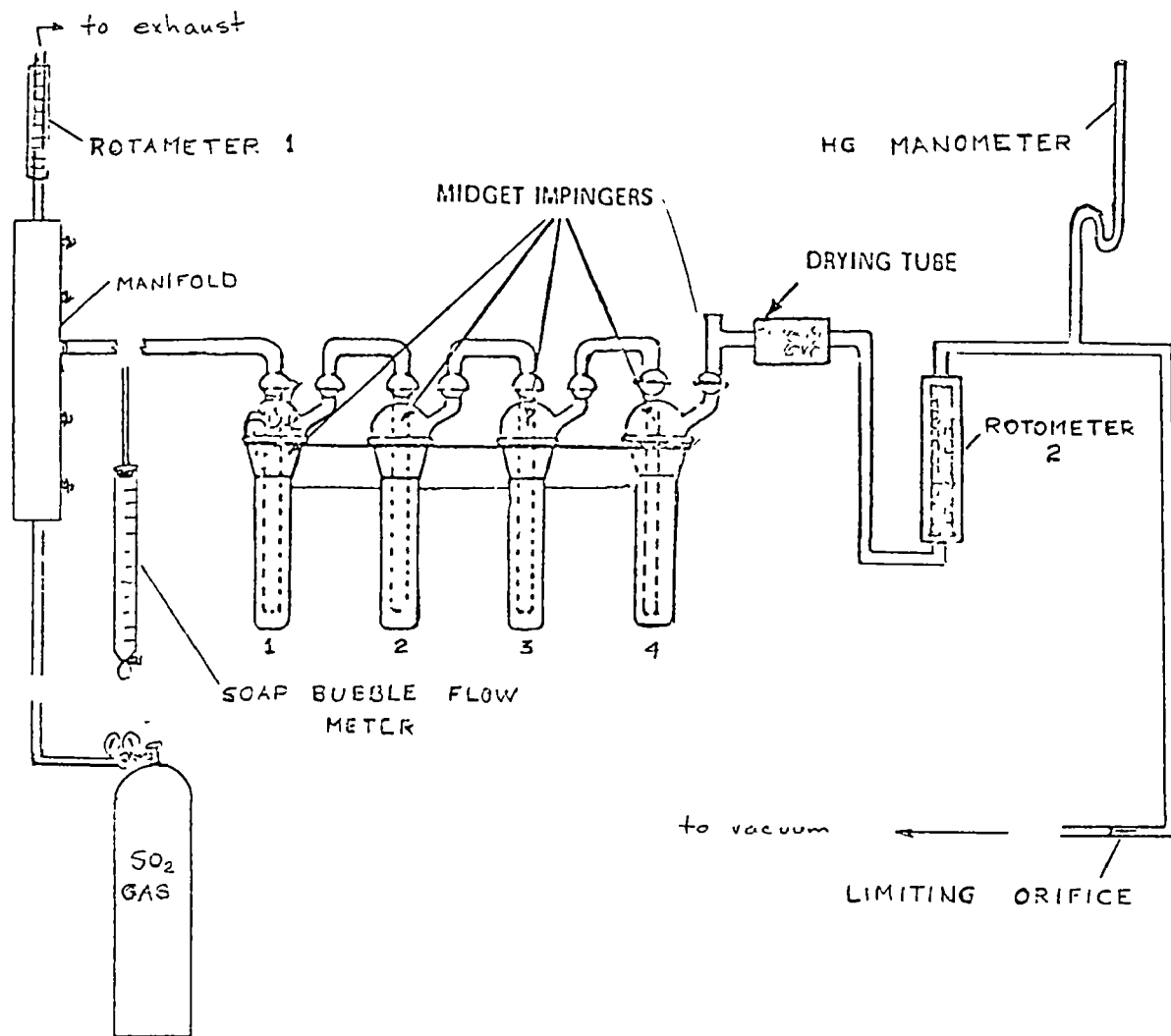


FIG 1 - SO<sub>2</sub> sampling train.