

Note: This is a reference cited in AP 42,
Compilation of Air Pollutant Emission Factors,
Volume I Stationary Point and Area Sources.
AP42 is located on the EPA web site at www.
epa.gov/ttn/chief/ap42/

The file name refers to the reference number,
the AP42 chapter and section. The file name
"ref02_c01s02.pdf" would mean the reference
is from AP42 chapter 1 section 2. The
reference may be from a previous version of
the section and no longer cited. The primary
source should always be checked.

EMB 71-PC-08/FEA
71 FERROALLOY
PRODUCTION
AP-42 Section 7.4
Reference Number
12.4
30

TEST NUMBER FA-1
REVISION

**Foote Mineral Company
Vancoram Operations
Steubenville, Ohio**

By
R. N. ALLEN

Contract Number CPA 70-81

RESOURCES RESEARCH, INC.
A SUBSIDIARY OF TRW INC.
WESTGATE PARK • 7600 COLSHIRE DRIVE • MCLEAN, VIRGINIA 22101

71 PC08
71-PC-08
FEA

TEST NUMBER FA-1

11470

REVISION

**Foot Mineral Company
Vancoram Operations
Steubenville, Ohio**

By
R. N. ALLEN

Contract Number CPA 70-81

RESOURCES RESEARCH, INC.
A SUBSIDIARY OF TRW INC.
WESTGATE PARK • 7600 COLSHIRE DRIVE • MCLEAN, VIRGINIA 22101

TEST NUMBER FA-1

FOOTE MINERAL COMPANY
VANCORAM OPERATIONS
STEUBENVILLE, OHIO

R. N. Allen

August 1971

Resources Research, Inc.
A Subsidiary of TRW Inc.
7600 Colshire Drive
McLean, Virginia 22101

Contract Number CPA 70-81

I. TABLE OF CONTENTS

	<u>Page</u>
II. INTRODUCTION	2
III. SUMMARY OF RESULTS	4
IV. PROCESS DESCRIPTION	9
V. LOCATION OF SAMPLING POINTS	14
VI. PROCESS OPERATION	16
VII. SAMPLING PROCEDURES	18
VIII. CLEANUP AND ANALYTICAL PROCEDURES	19
IX. DISCUSSION	20
A. Results	20
B. Operating Conditions	22
C. Test Conditions	23
X. APPENDIX	25
A. Complete Particulate Results with Example Calculations	
B. Complete Gaseous Results with Example Calculations	
C. Complete Operation Results (not applicable)	
D. Field Data	
E.1 Sampling Procedures	
E.2 Cleanup and Analytical Procedures	
F. Laboratory Report	
G. Test Logs	
H. Related Reports (not applicable)	
I. Project Participants and Titles	
J. Calculation of Escaping Fumes and Dust	
K. Particle Sizing Procedures	
L. Description of Chemical Analyses of Particulate on Filters	

LIST OF TABLES

<u>Table No.</u>	<u>Title</u>	<u>Page</u>
1	Comparison of OAP and ASME Methods	4
2	Summary of Results	6
3	Plant Operating Conditions	8

LIST OF FIGURES

<u>Figure No.</u>	<u>Title</u>	<u>Page</u>
1	Sampling Locations	2
2	Process Flow Diagram	10
3	Typical Furnace	11
4	Schematic Cross Section of Furnace Under Test	12
5	Sample Point Locations	15

II. INTRODUCTION

Source emission tests are being performed on a series of electric furnace installations, known as reactive metals or ferroalloys, for the Office of Air Programs, Environmental Protection Agency. The tests include grain loading measurements, particle size analyses, and chemical analyses for a variety of furnace formulations and control devices. The initial series of tests, contained in this report, were performed at the Foote Mineral Company, Vancoram Operations, P. O. Box 217, Steubenville, Ohio, 43952, during the week of May 17, 1971.

Emissions for this particular plant were determined for a ferrochrome silicon furnace (No. 25) and a chrome ore-lime melt furnace (No. 6). Both of these units were hooded furnaces without control devices. Each hood was provided with induced draft exhaust fans so that most of the dust and fumes were removed by exhaust stacks rather than random escape, as shown in the diagram below. Further detailed diagrams and descriptions are included in Sections IV and V (Process Description and Location of Sampling Points).

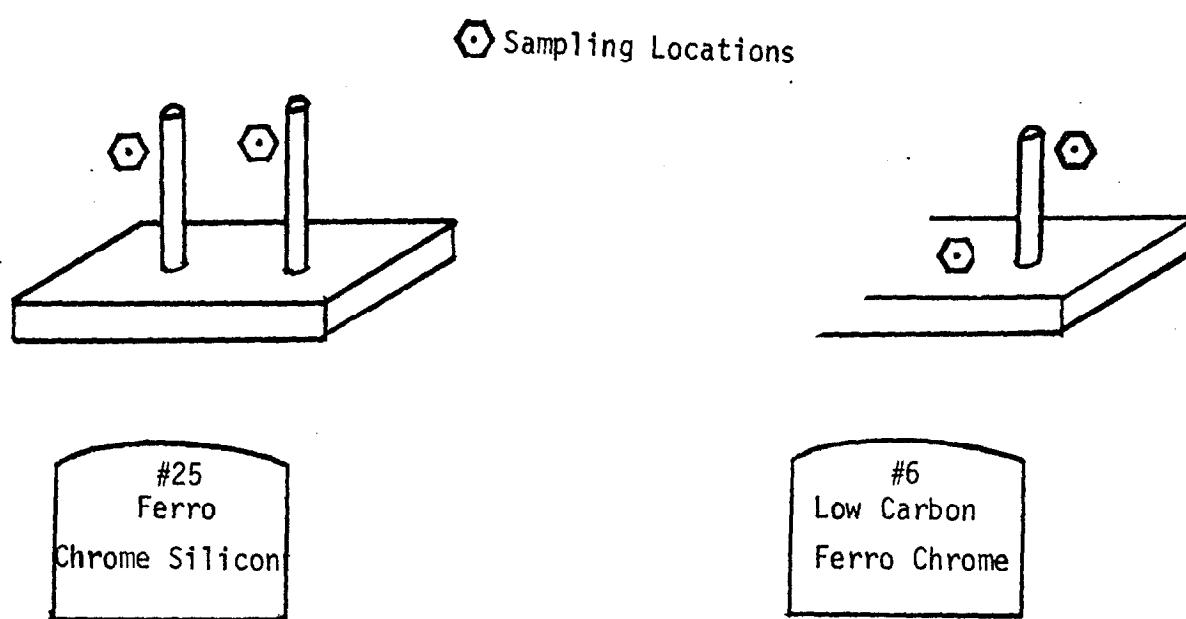


Figure 1

During this particular survey particulate matter was sampled using the special OAP train and a so-called ASME train using an alundum thimble filter within the stack. Sulfur oxides were sampled using the Shell Development method and integrated combustion gases were sampled in a gas bag with analysis by standard Orsat. Particle size was measured *in situ* with an Andersen Sampler and a Brink Sampler. Most of the above samples were collected in duplicate runs.

III. SUMMARY OF RESULTS

Total "catches" obtained on the various samples taken by the two methods (OAP and ASME) are shown in Table I below.

<u>Sampling Method</u>	<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>grains/SCF</u>	<u>Lbs/Hour</u>	<u>OAP vs. ASTM</u>
OAP	25 E-1	May 18	1438-1732	0.225	128.8	-28%
ASME	25 E-1	May 18	1438-1727	0.301	179.4	
OAP	25 E-2	May 19	0950-1125	0.388	239.7	
OAP	25 W-1	May 18	1430-1727	0.087	68.1	
ASME	25 W-2	May 19	0953-1130	0.208	202.2	- 2%
OAP	25 W-2	May 19	0953-1128	0.263	198.1	
OAP	6 E-1	May 20	1129-1436	0.154	54.8	-10%
ASME	6 E-1	May 20	1129-1343	0.183	60.4	
OAP	6 E-2	May 20	1533-1740	0.178	62.2	-16%
ASME	6 E-2	May 20	1534-1745	0.219	72.3	

Emissions from the east and west exhaust stacks of Furnace 25 varied widely in the two tests, both between the two stacks and between the two test runs. One of two stoking machines failed during the second test and emissions were visibly different.

The comparison between the "catch" by the OAP and ASME methods was reasonably close in some trials. It was somewhat unexpected that results from the OAP method were less than those obtained with the ASME method. There are wide fluctuations in the amount of dust or fume being produced from one moment to the next, and the traverses of OAP and ASME probes were in opposite directions to avoid interference. No errors or losses occurred to the best of our knowledge other than those inherent with each sampling method.

A summary of the various plant emissions measured is shown in Table 2.

Nearly 100 percent of the total emissions from Furnace 25 were being removed by the hood and draft fans. An additional duct was built into the same system to collect fumes generated during tapping operations. This collection efficiency does not include any dust or fumes produced by the pouring and hauling of the molten metal and slag during the subsequent operations that follow tapping of the furnace.

A large amount of dust was escaping from Furnace 6, apparently due to the greater difficulty of hooding a tilting furnace. Measurements with a high volume sampler indicated that just slightly over half of the total emissions were being emitted from the exhaust stack.

Analyses by photograph, atomic absorption, X-ray diffraction, electron beam X-ray microanalysis and optical spark emission indicated the compositional variations and striations within each sample. Atomic absorption indicated that emissions from Furnace 25 were approximately 70% SiO_2 and chromium ranged from 0.7 to 2.1%. The X-ray diffractions indicated that all samples were largely amorphous (non-crystalline). However, the two samples from Furnace 6 indicated recognizable patterns in the class of compounds called spinels. The electron beam X-ray microanalysis indicated that there were concentration gradients in which chromium, magnesium, and iron were concentrated near the filter, while zinc and calcium were near the surface of the collected fume. Optical (spark) emissions served as a check with the atomic absorption and electron beam results. In addition, numerous trace impurities were found which were not identified in the other approaches.

Particle size analysis was conducted dynamically within the stacks by means of the Andersen impactor sampler. This unit was inadequate for the very fine fumes encountered because over half of the particles passed

TABLE 2
SUMMARY OF RESULTS

Run Number	25 E-1	25 E-2	25 W-1	25 W-2	6E-1	6E-2
Date	5/18/71	5/19/71	5/18/71	5/19/71	5/20/71	5/20/71
✓ Stack Flow Rate - SCFM * dry	66,800	72,100	91,500	87,900	41,492	40,901
✓ % Water Vapor - % Vol.	1.28	2.22	1.01	0.76	0.88	0.24
✓ % CO ₂ - Vol % dry	0.8	0.8	0.5	0.5	0.0	0.1
✓ % O ₂ - Vol % dry	20.3	20.3	20.1	20.1	20.8	20.8
% Excess air @ sampling point	3383	3383	2010	2010	7,704	8,667
✓ SO ₂ Emissions - ppm dry	13.5	10.7	17.0	-	0	0
NO _x Emissions - ppm dry	N/A	N/A	N/A	N/A	N/A	N/A
<u>Particulates</u>						
Probe, Cyclone, & Filter Catch gr/SCF* dry	0.220	0.376	0.0468	0.249	0.141	0.175
gr/CF @ Stack Conditions	0.153	0.264	0.316	0.193	0.124	0.148
lbs./hr.	126.0	232.3	36.7	187.6	50.2	61.4
<u>Total Catch</u> gr /SCF * dry	0.225	0.388	0.0869	0.263	0.154	0.178
gr /CF @ Stack Conditions	0.156	0.272	0.0655	0.204	0.136	0.150
✓ lbs./hr.	128.8	239.7	68.1	193.1	54.8	62.2

70°F, 29.92" Hg

completely through all stages and were deposited on the final filter. A second series of tests was conducted with the Brink cascade impactor. In this case, some three-fourths of the particles were on measurable stages, and the mass median diameter was found to be between 0.6 and 0.7 microns.

Table 3 shows some of the more important operating parameters of each furnace during the testing period.

TABLE 3
PLANT OPERATING CONDITIONS

Date	Time Tested	Location/ Test No.	FURNACE					Remarks
			Mix	Product	Tapped	Delay		
6/18/71	1438-1558	25 E-1	Quartzites, Chrome ores, Carbon reduc- ing agents, and Flux	L. C. FeCrSi (36-40)	1503 hrs	None	Normal	
	1625-1732				1518 1702 1722			
6/19/71	0950-1125	25 E-2	Same as above	Same as above	1109 hrs	None	Abnormal: No stoking due to machine failure. Higher temp. than usual.	
	1430-1555				1125			
6/18/71	1625-1722	25 W-1	Same as above	Same as above	1503 hrs	None	Normal	
					1518 1702 1722			
6/19/71	0953-1128	25 W-2	Same as above	Same as above	1109 hrs	None	Normal stoking but affected by lack of stoking on east side	
					1127			
6/20/71	1129-1223	6 E-1	Chrome ores and lime	Ore lime melt	See delay	1222-1238 (5&6 down) 1247-1255 (5&6 down Tap 5) 1332-1340 (6 down only)	Normal	
6/20/71	1533-1627	6 E-2	Chrome ores and lime	Ore lime melt	See Delay	1700-1707 (6 down only) Tapped	Normal	

IV. PROCESS DESCRIPTION

The reactive metals are generally ferroalloys which are produced in submerged arc electric furnaces. The facilities under consideration in this report are open furnaces, with hooding, but without collection systems to reduce the emission of fumes and dust following collection. Figure 2 is a process flow diagram indicating the inlet and outlet materials. A diagram (Figure 3) is included to show more details on a typical furnace. Figure 4 indicates the cross-sectional view of the actual furnaces under test in this survey.

The electric arc is employed as a concentrated source of heat. Chrome, manganese and other ores are added to the surface of the furnace through mechanized equipment and chutes. Additional carbon in the form of coke, wood chips, etc., is an integral part of the furnace mix, along with specialized fluxes, etc. The mix is added directly to the surface of the furnace through chutes and is then spread over the surface with stoking machines.

The very high temperatures produced initiate a reaction in the bottom of the furnaces and form a layer of metal which is tapped at appropriate times. As the ore and carbonaceous materials settle to the bottom of the furnace, the heat, in conjunction with a lack of oxygen, react with the oxide ores to produce carbon monoxide which reacts further chemically, as a reducing agent, in order to remove oxygen from the original ores and thus produce the elemental metal. Escaping gases are burned at the surface of the furnace in the so-called open units. In closed furnaces, these gases may be burned in such manner to salvage their heat value.

Furnace 25 produced a ferrochrome silicon product. Soderberg type electrodes are formed in place from a "paste" rather than using prebaked carbon electrodes. Furnace 6 was an open arc unit using the prebaked carbon electrodes. Induced draft fans are employed to pull fumes from

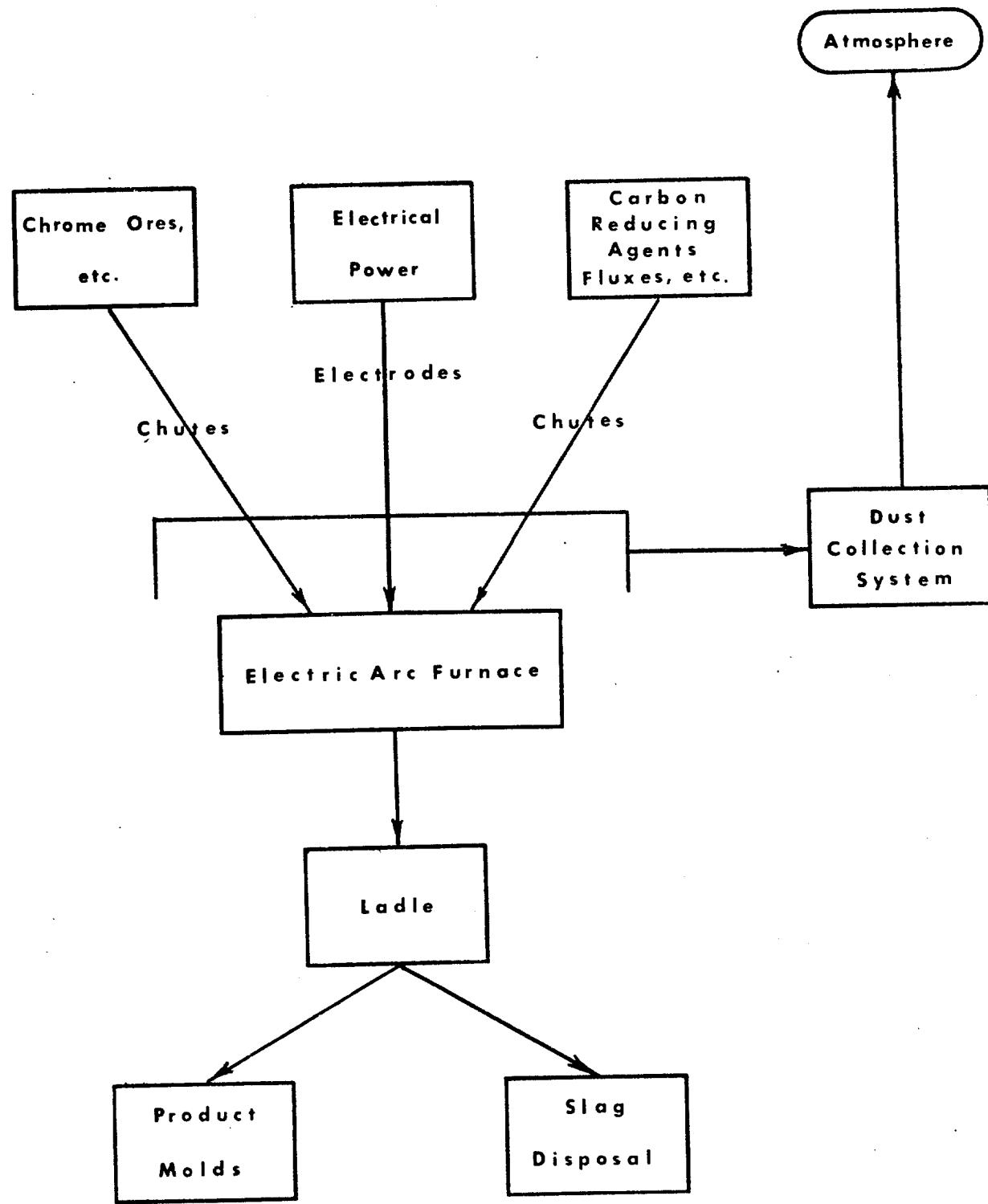


FIGURE 2. PROCESS FLOW DIAGRAM

TYPICAL 26' DIA. ROUND-3 PHASE FURNACE

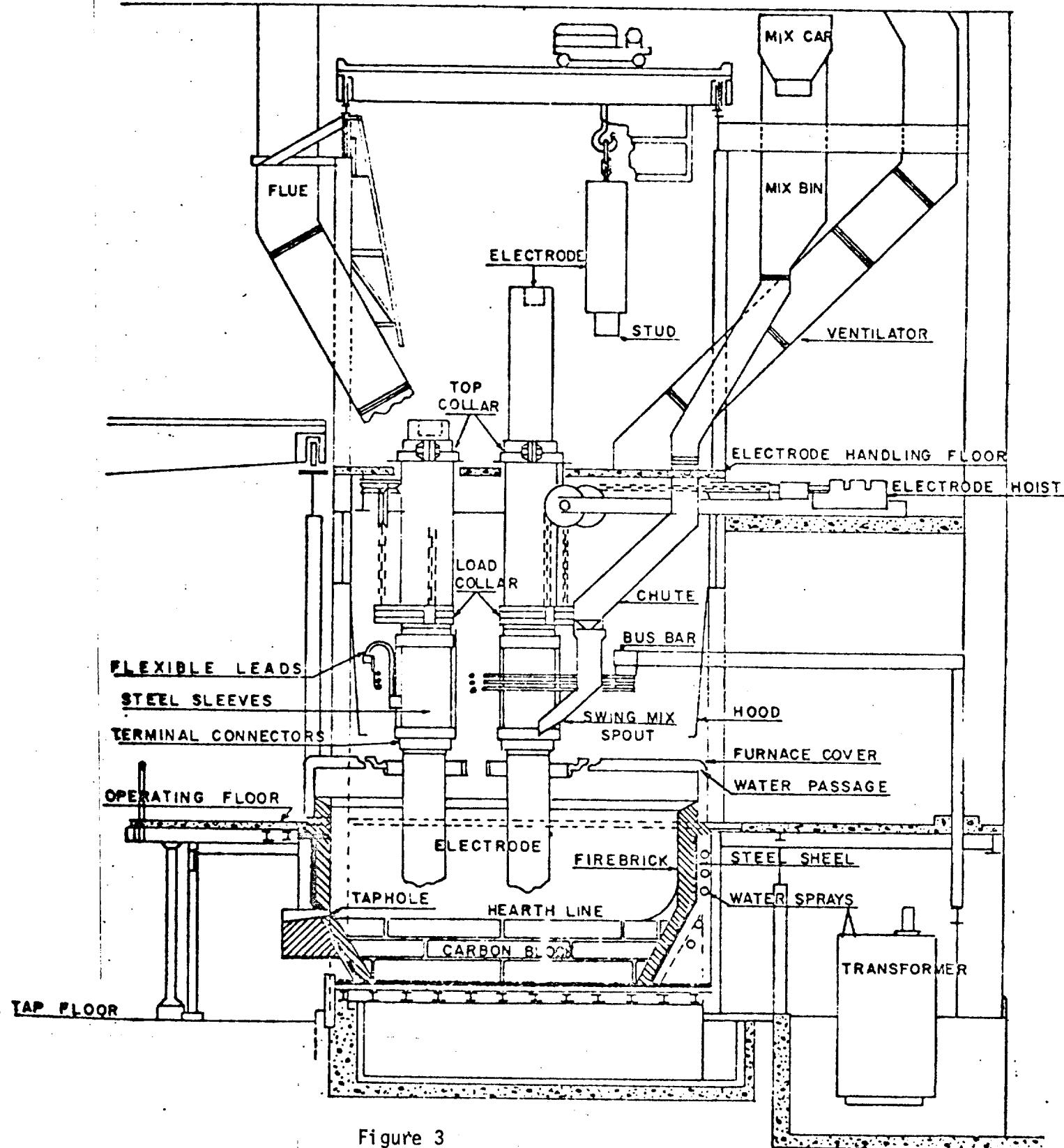


Figure 3

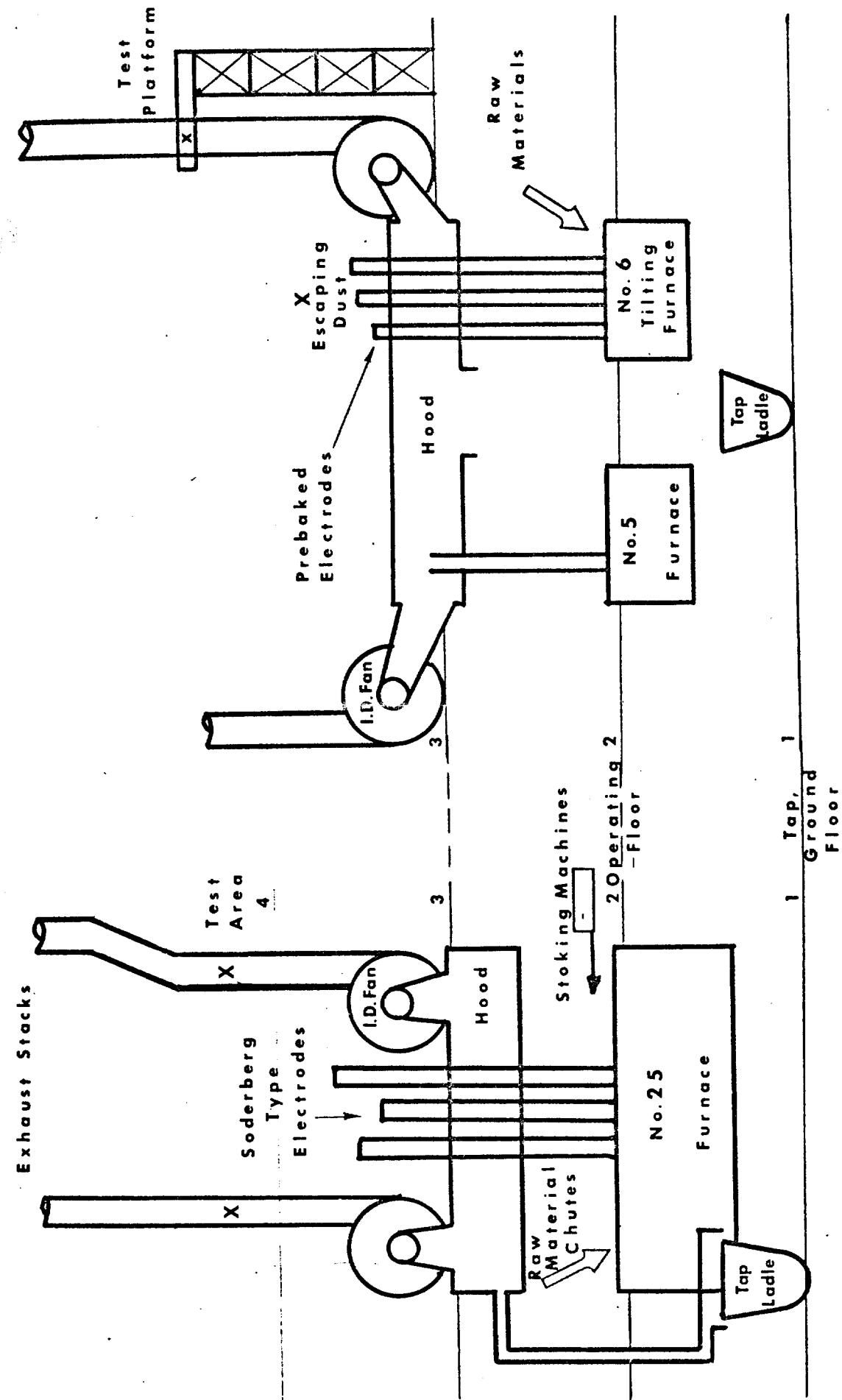


FIGURE 4. SCHEMATIC CROSS SECTION OF FURNACE UNDER TEST

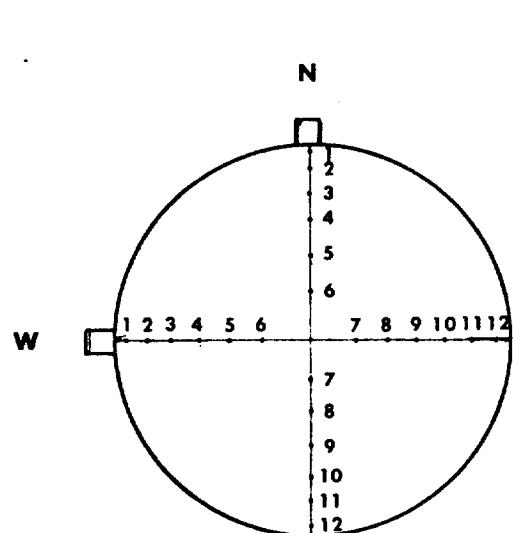
the hooding into exhaust stacks such that emissions are discharged above the roof level. Any escaping fumes rise to louvers or monitors in the roof where they are discharged.

The furnaces are tapped at intervals somewhat less than two hours into ladles. The slag is removed from this ladle and disposed of by various means. Molten product is poured into molds, after which it is broken into usable sizes. Product from the No. 6 furnace is somewhat different in that it is a simple ore lime melt rather than a metallic product.

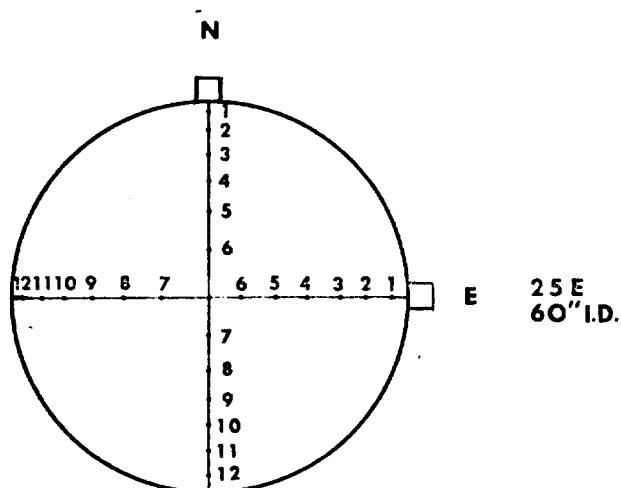
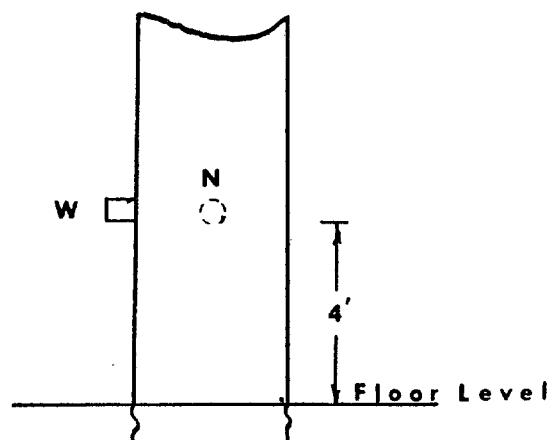
V. LOCATION OF SAMPLING POINTS

Sample port locations were selected where most satisfactory, during a presurvey inspection trip, and approved by the OAP Project Officer. Two ports, 90° apart, were provided by the plant personnel at each stack tested. Furnace 25, east and west stacks, had ports approximately four feet above the fourth floor level, or three stack diameters above the fan. A more satisfactory location was not accessible. Furnace 6 exhaust stack had the same diameter as the exhaust stacks from Furnace 25, but the sampling port locations were after approximately 25 feet of straight flow above the fan. These were not conveniently accessible from the standard floor but were reached by special scaffolding, which was erected by the plant for this series of tests. Figure 4 (page 12) is a simplified cross-section of the furnaces under test and indicates the relative location of sampling ports.

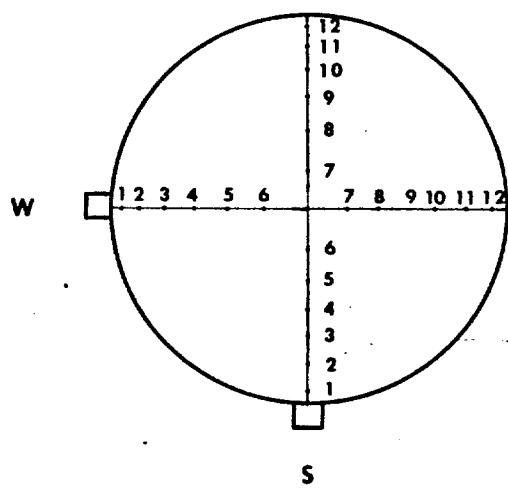
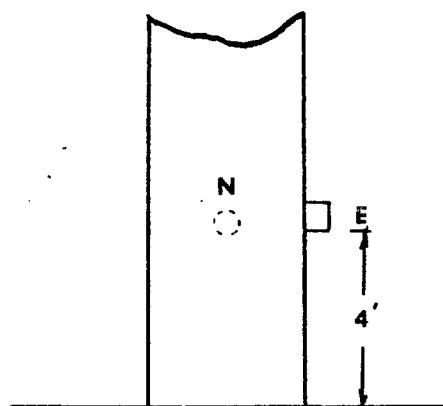
Each of the test cross-sections was divided for 12-position sampling from each of two different ports. The centroid of six equal areas was used for determining velocity, temperature, and samples. All three of the stacks tested had identical diameters and cross-sections. The sample port locations were slightly different with a long run of straight uniform flow, but the sample points within each stack were identical. Figure 5 shows a sketch of typical port locations and sample points. The test points were selected using the proposed source test method 6 by EPA/OAP in conjunction with previous experience and discussion with the Project Officer.



N
1
25 W
60" I.D.



E 25 E
60" I.D.



6 E
60" I.D.

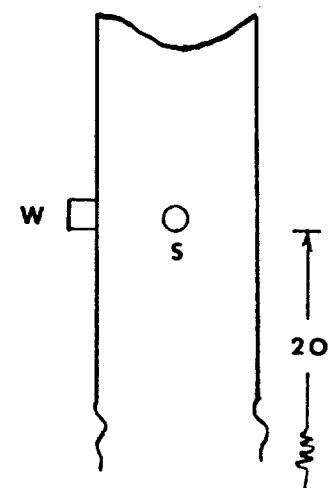


FIGURE 5. SAMPLE POINT LOCATIONS

VI. PROCESS OPERATION

The operation of the two furnaces being tested is somewhat different. Furnace 25 is more typical of the usual ferroalloys furnace, while Furnace 6 differs in that the electric arc is exposed and the furnace is tapped by tilting the entire crucible.

The operation of Furnace 25 is essentially continuous, but it is considered to be an approximately 2-hour cycle as measured by the times at which it is tapped. The tapping cycle basically depends upon the total power produced within the furnace, therefore producing a specific amount of metal product. The chrome ore and reducing agent mix is added at appropriate intervals and spread over the surface of the furnace by stokers. In this large sized furnace, these are electrically operated mechanical stokers. They are small cars with a large pushing ram mounted on the front so that the ore mix, which is dropped into piles on the surface, may be spread around the electrodes.

Furnace 6 operates with an open arc and without stoking. Two furnaces, 5 and 6, are operated jointly. There is, therefore, a problem of two units tilting alternately into a single tapping ladle. These two units also share a single hood over the tapping area. Largely because of the tilting arrangement and the shared tapping area, there is considerably more dust and fumes which are not collected by the hooding arrangement. This fugitive dust emission was estimated with the aid of high volume samplers.

Operation of the furnaces was considered normal for most testing. However, the tests conducted on May 19, 1971, (No. 25) coincided with a period in which one of the stoking machines was removed from service. As a result of this variant, only the west side of the furnace was stoked. The east side developed heat "blows" and tended to run at a much higher temperature than normal. This condition produced an excessive amount of fume and resulted in an unusually large number of individual filters

being required due to clogging. The more important plant operating conditions are shown in Table 3, page 8, under Summary of Results.

VII. SAMPLING PROCEDURES

All test procedures were discussed with the Project Officer in advance. All procedures were essentially the same as those being issued by the Environmental Protection Agency for source sampling.

Preliminary velocity and temperature readings were obtained in order to select nozzle sizes for isokinetic sampling. Particulate sampling was conducted using the OAP train as described in Appendix E-1. A so-called ASME train was used simultaneously with the OAP train. This was done alternately with Furnace 25 where there were two exhaust ducts but for each sample in Furnace 6 where there was a single exhaust stack.

Gas sampling was also conducted in accordance with the proposed EPA Standard Source Testing Methods. Sulfur dioxide was sampled with midget impingers using isopropyl alcohol and hydrogen peroxide solutions. Combustion gases were sampled in plastic bags for immediate analysis with an Orsat analyzer.

VIII. CLEANUP AND ANALYTICAL PROCEDURES

The methods employed for cleanup of the OAP particulate train have become relatively standardized through testing incinerators for government approval. Various sections of the sampling train are washed with acetone and water. The filter is removed carefully and each portion of the collected particulate matter is placed in separate containers. All portions are then dried at ambient conditions and the water is extracted for organic material, as well as being evaporated to dryness. These procedures are outlined in detail in Appendix E-2.

IX. DISCUSSION

A. Results

The OAP sampling train, being a more complex and exacting device for measuring total particulate concentrations, was expected to collect more particulate material than the ASME train. However, this did not occur in any of the four comparisons. The relatively good agreement between OAP and ASME "catches" in two or three of the four tests would indicate that very little condensable material was passing through either the fiberglass filter or the alundum thimble. Although the particle size of this fume being emitted was largely less than one micron, a rapid buildup of filtering surface was apparently able to collect all material within the alundum thimble. In the ASME test 25 W-2, the probe was washed out with acetone (following the alundum thimble holder), but this extra material recovery changed the grain loading from 0.208 to only 0.213 grains per scf.

Initial testing has indicated a severe problem with the reactive metal emissions plugging the filters in the OAP sampling train. This problem is apparently related to both the nature of fumes being generated as well as their concentration. There was a distinct difference in the tendency to clog the filters between Furnace 25 east and west exhaust stacks. The east stack contained a greater concentration of fumes in every test, and the filter train employed at this location clogged very frequently. This tendency to clog the filters, thereby increasing vacuum in the sample train such that isokinetic conditions could no longer be maintained, was especially noticeable during the period that one stoker became inoperable and emission concentrations increased.

All tests were performed when furnaces were considered to be operating under normal conditions, except on May 19 when the stoking machine failure occurred. This lack of stoking allowed the surface of the furnace to develop hot spots and gas blows, which appreciably increased the emission

of fumes. The fumes were also increased on the opposite side of the furnace due to the mixing of air and gases over the furnace and under the hoods.

The hood configuration allowed relatively greater emissions and temperatures to occur on the east side of the furnace under any circumstances. Gas flow and concentrations were therefore dissimilar from side to side during normal operations as well as during abnormal operations.

The large amount of dilution air resulted in very low concentrations of carbon dioxide in the exhaust gases and, therefore, made it completely impossible to detect carbon monoxide with an Orsat analyzer. Excess air was correspondingly great.

One sulfur dioxide sample was mistakenly run using hydrogen peroxide in the first impinger rather than 80% isopropyl alcohol. This sample, therefore, measured total sulfur oxides as opposed to SO_2 . If the one sample can be considered indicative, it would appear that some oxides of sulfur may be emitted in the form of sulfuric acid mist (or SO_3).

There was relatively little difference between the particulates caught in the probe, cyclone, and filter catch versus the total catch, including the condensables, in all samples except 25 W-1. The very low increase due to condensable particulates is also true of Furnace 6. This furnace had even greater air dilution than Furnace 25. Sulfur dioxide emissions were negligible from this unit, and even the carbon dioxide measurements bordered upon being undetectable.

Chemical analysis emphasized the uneven operation of the normal ferro-alloy furnace. Photographs clearly indicated striations due to changes in material being emitted.

Analysis of particle sizes with the Andersen impactor went without particular trouble. However, the average particle size was so small that three-quarters of the total material passed completely through all stages and was deposited upon the final filter. These tests indicated that the unit would not satisfactorily determine particle size without considerable extrapolation.

A Brink cascade impactor was employed for the same conditions and this unit presented satisfactory data for determining mass median diameters. There were several problems with both units in attempting to obtain representative samples over any length of time. The concentration of fumes was so great that sampling had to be cut very short in order to avoid overloading the various stages. In addition, the physical configuration of the Brink sampler made it difficult to place completely in the exhaust stacks. This appeared to have had no detrimental effect upon the samples obtained.

B. Operating Conditions

The operating conditions of a ferroalloy furnace are nonuniform, because of the normal feeding and tapping procedures. The mix material is added from chutes and would tend to produce large particle size dust. This mix is then spread over the furnace with stokers, stirring up dust of other varieties. The reaction forms gases and fumes, therefore producing a third variety of emission. The tapping cycle often produces collapsing areas in the surface, thus exposing hotter materials. There are occasionally gas blows from within the surface. All of these factors produce continually changing emissions. Because of the difficulty in completely enclosing any furnace with hoods, it is not unexpected that emissions would be different from supposedly parallel exhaust systems.

The location of sampling ports only three pipe diameters above the induced draft fan on Furnace 25 is not considered detrimental due to the very fine particle size of emissions. Each cross-section was provided

with two sample ports at right angles. This cross-section was divided into equal areas for 12-position sampling in each port.

The glass-lined probes had been supplied by the Environmental Protection Agency. These were somewhat short, therefore could not reach the last two points of the traverse. Where this occurred, the sample was correspondingly shortened or the very last point obtainable was used for an increased sampling period.

Furnace 25 had two parallel exhaust stacks; however, only one ASME train had been requested. Therefore, these samples (in alundum thimbles) were obtained alternately from the east and then the west stacks.

C. Sampling and Analytical Procedures

ASME and OAP trains were operated in comparison during the same period of time. In order to avoid interference within the stack, the OAP train was operated from points 1, 2, 3, etc., across one diameter. The ASME train was operated on the other diameter from point 12, point 11, point 10, etc. At the conclusion of a traverse in one direction, the two trains were reversed and then the same procedure was employed so that the two probes did not come in contact within the stack.

All procedures were essentially the same as those methods being issued by the Environmental Protection Agency for source sampling. However, some of these new methods employ equipment which was not obtainable on relatively short notice. Particulate sampling with the OAP train was conducted without exception.*

* The ASME train is a standard method which has been used by Resources Research, Inc., continuously at previous locations for industry. Sampling for sulfur dioxide calls for a heated glass probe which was not available. A stainless steel probe was inserted completely within the stack such that its temperature was maintained well above the dew point, and the impingers were connected very close to its exhaust. The impingers were interconnected with polyethylene connections. Since this particular survey, complete glass fittings and heated probes have been received.

Cleanup of the particulate train was done by standard procedure. Recent decisions by OAP now indicate that all acetone samples should be transported to the laboratory in glass containers. This survey employed polyethylene containers, but it is believed to be perfectly satisfactory for total particulate analyses, unless an analysis is required for mercury or some other exotic material which may be leached from the plasticisers or plastic. Results have indicated that the condensable material is unaffected by any such leaching.

APPENDIX A
COMPLETE PARTICULATE RESULTS
WITH EXAMPLE CALCULATIONS

SUMMARY OF RESULTS

Run Number	25 E-1	25 E-2	25 W-1	25 W-2	
Date	5/18/71	5/19/71	5/18/71	5/19/71	
Stack Flow Rate - SCFM * dry	66,800	72,100	91,500	87,900	
% Water Vapor - % Vol.	1.28	2.22	1.01	0.76	
% CO ₂ - Vol % dry	0.8	0.8	0.5	0.5	
% O ₂ - Vol % dry	20.3	20.3	20.1	20.1	
% Excess air @ sampling point	3383	3383	2010	2010	
SO ₂ Emissions - ppm dry	13.5	10.7	17.0	-	
NO _x Emissions - ppm dry	N/A	N/A	N/A	N/A	
<u>Particulates</u>					
Probe, Cyclone, & Filter Catch gr/SCF * dry	0.220	0.376	0.0468	0.249	
gr/CF @ Stack Conditions	0.153	0.264	0.0316	0.193	
lbs./hr.	126.0	232.3	36.7	187.6	
<u>Total Catch</u>					
gr /SCF * dry	0.225	0.388	0.0869	0.263	
gr /CF @ Stack Conditions	0.156	0.272	0.0655	0.204	
lbs./hr.	128.8	239.7	68.1	198.1	

* 70°F, 29.92" Hg

REPORT NO.

PAGE OF PAGES

SOURCE TESTING CALCULATION FORMS

Test. No. 25E/25W

No. Runs Two Each

Name of Firm Foote Mineral Company

Location of Plant Steubenville, Ohio

Type of Plant Reactive Metals

Control Equipment None

Sampling Point Locations Stack Exhaust

Pollutants Sampled Total Particulate, SO₂, CO₂, O₂, CO.

Time of Particulate Test:

Run No. <u>25E-1</u>	Date <u>5/18/71</u>	Begin <u>1438</u>	End <u>1732</u>
Run No. <u>25E-2</u>	Date <u>5/19/71</u>	Begin <u>0950</u>	End <u>1125</u>
Run No. <u>25W-1</u>	Date <u>5/18/71</u>	Begin <u>1430</u>	End <u>1727</u>
Run No. <u>25W-2</u>	Date <u>5/19/71</u>	Begin <u>0953</u>	End <u>1128</u>

PARTICULATE EMISSION DATA

Run No.	25E-1	25E-2	25W-1	25W-2		
P_b barometric pressure, "Hg Absolute	29.2	29.2	29.2	29.2		
P_m orifice pressure drop, "H ₂ O	1.35	1.72	2.8	2.10		
V_m volume of dry gas sampled @ meter conditions, ft. ³	76.12	44.34	105.25	38.70		
T_m Average Gas Meter Temperature, °F	124	108	97	88		
$V_{m\text{std.}}$ Volume of Dry Gas Sampled @ Standard Conditions, ft. ³	67.23	40.02	97.10	36.40		
V_w Total H ₂ O collected, ml., Impingers & Silical Gel.	18.3	19.2	20.4	6.0		
$V_{w\text{gas}}$ Volume of Water Vapor Collected ft. ³ @ Standard Conditions*	0.87	0.91	0.97	0.28		

* 70°F, 29.92" Hg

PARTICULATE EMISSION DATA (cont'd)

Run No.	25E-1	25E-2	25W-1	25W-2	
%M - % Moisture in the stack gas by volume	1.28	2.22	1.01	0.76	
M_d - Mole fraction of dry gas	0.99	0.98	0.99	0.99	
% CO ₂	0.8	0.8	0.5	0.5	
% O ₂	20.3	20.3	20.1	20.1	
% N ₂	78.7	78.7	79.4	79.4	
M W _d - Molecular weight of dry stack gas	28.9	28.9	28.7	28.7	
M W - Molecular weight of stack gas	28.8	28.7	28.6	28.6	
ΔP_s - Velocity Head of stack gas, In.H ₂ O	1.41	1.70	2.47	2.20	
T _s - Stack Temperature, °F	280	261	218	204	
$\Delta P_s \times (T_s + 460)$	32.8	35.0	40.9	38.3	
P _s - Stack Pressure, "Hg. Absolute	29.2	29.2	29.2	29.2	
V _s - Stack Velocity @ stack conditions, fpm	4920	5220	6180	5790	
A _s - Stack Area, in. ²	2826	2826	2826	2826	
Q _s - Stack Gas Volume @ Standard Conditions. *SCFM	66,800	72,100	91,500	87,900	
T _t - Net Time of Test, min.	120	66	114	54	
D _n - Sampling Nozzle Diameter, in.	0.1875	0.1875	0.1875	0.1875	
%I - Percent isokinetic	97	94	103	83	
m _f - Particulate - probe, cyclone and filter, mg.	958.9	976.1	295.1	588.5	
m _t - Particulate - total, mg.	982.6	1009.1	547.5	623.6	
C _{an} - Particulate - probe, cyclone, and filter, gr/SCF	0.220	0.376	0.0468	0.249	
C _{ao} - Particulate - total, gr/SCF	0.225	0.388	0.0869	0.263	
C _{at} - Particulate - probe, cyclone, & filter gr/cf @ stack conditions	0.153	0.264	0.0316	0.193	

PARTICULATE EMISSION DATA (cont'd)

Run No.		25E-1	25E-2	25W-1	25W-2	
C_{au} - Particulate, total, gr/cf @ stack cond.		0.156	0.272	0.0655	0.204	
C_{aw} - Particulate, probe, cyclone, and filter, lb/hr.		126.0	232.3	36.7	187.6	
C_{ax} - Particulate - total, lb/hr.		128.8	239.7	68.1	198.1	
% EA - % Excess air @ sampling point		3383.	3333.	2010.	2010.	

*70°F. 29.92" Hg.

SUMMARY OF RESULTS

Run Number	6E-1	6E-2				
Date	5/20/71	5/20/71				
Stack Flow Rate - SCFM * dry	41,492	40,901				
% Water Vapor - % Vol.	0.88	0.24				
% CO ₂ - Vol % dry	0.0	0.1				
% O ₂ - Vol % dry	20.8	20.8				
% Excess air @ sampling point	7,704	8,667				
SO ₂ Emissions - ppm dry	0	0				
NO _x Emissions - ppm dry	N/A	N/A				
<u>Particulates</u>						
Probe, Cyclone, & Filter Catch	0.141	0.175				
gr/SCF * dry						
gr/CF @ Stack Conditions	0.124	0.148				
lbs./hr.	50.2	61.4				
<u>Total Catch</u>						
gr /SCF * dry	0.154	0.178				
gr /CF @ Stack Conditions	0.136	0.150				
lbs./hr.	54.8	62.2				

* 70°F, 29.92" Hg

REPORT NO.

PAGE OF PAGES

SOURCE TESTING CALCULATION FORMS

Test. No. 6E

No. Runs 2

Name of Firm Foote Minerals Company

Location of Plant Steubenville, Ohio

Type of Plant Reactive Metals

Control Equipment None

Sampling Point Locations Exhaust Stack

Pollutants Sampled Total Particulate, SO₂, CO₂, O₂, CO

Time of Particulate Test:

Run No. 1 Date 5/20/71 Begin 1129 End 1336

Run No. 2 Date 5/20/71 Begin 1533 End 1740

Run No. Date Begin End

PARTICULATE EMISSION DATA

Run No.	6E-1	6E-2					
P _b barometric pressure, "Hg Absolute	29.30	29.30					
P _m orifice pressure drop, "H ₂ O	1.96	1.68					
V _m volume of dry gas sampled @ meter conditions, ft. ³	85.20	85.58					
T _m Average Gas Meter Temperature, °F	125	132					
V _m _{std.} Volume of Dry Gas Sampled @ Standard Conditions, ft. ³	75.23	75.00					
V _w Total H ₂ O collected, ml., Impingers & Silical Gel.	14.4	3.7					
V _w _{gas} Volume of Water Vapor Collected ft. ³ @ Standard Conditions*	0.68	0.18					

* 70°F, 29.92" Hg.

PARTICULATE EMISSION DATA (cont'd)

Run No.	6E-1	6E-2				
%M - % Moisture in the stack gas by volume	0.88	0.24				
M _d - Mole fraction of dry gas	0.991	0.998				
% CO ₂	0.0	0.1				
% O ₂	20.8	20.8				
% N ₂	79.2	79.1				
M W _d - Molecular weight of dry stack gas	28.84	28.85				
M W - Molecular weight of stack gas	28.74	28.83				
ΔP _s - Velocity Head of stack gas, In.H ₂ O	0.47	0.42				
T _s - Stack Temperature, °F	123	154				
ΔP _s X (T _s + 460)	16.57	16.06				
P _s - Stack Pressure, "Hg. Absolute	29.28	29.28				
V _s - Stack Velocity @ stack conditions, fpm	2396.2	2472.3				
A _s - Stack Area, in. ²	2826	2826				
Q _s - Stack Gas Volume @ Standard Conditions. * SCFM	41,492	40,- 901				
T _t - Net Time of Test, min.	108	108				
D _n - Sampling Nozzle Diameter, in.	0.25	0.25				
%I - Percent isokinetic	109.	111.0				
m _f - Particulate - probe, cyclone and filter, mg.	688.3	849.9				
m _t - Particulate - total, mg.	752.6	865.3				
c _{an} - Particulate - probe, cyclone, and filter, gr/SCF	0.141	0.175				
c _{ao} - Particulate - total, gr/SCF	0.154	0.178				
c _{at} - Particulate - probe, cyclone, & filter gr/cf @ stack conditions	0.124	0.148				

PARTICULATE EMISSION DATA (cont'd)

Run No.	6E-1	6E-2					
C_{au} - Particulate, total, gr/cf @ stack cond.	0.136	0.150	-				
C_{aw} - Particulate, probe, cyclone, and filter, 1b/hr.	50.2	61.4					
C_{ax} - Particulate - total, 1b/hr.	54.8	62.2					
% EA - % Excess air @ sampling point	7,704	8,667					

*70°F. 29.92" Hg.

SAMPLE PARTICULATE CALCULATIONS
(Sample 6E-1)

1. Volume of dry gas sampled at standard conditions - 70°F, 29.92" Hg, ft³.

$$V_{m_{std}} = \left(\frac{17.7 \times V_m \frac{P_B + P_m}{13.6}}{(T_m + 460)} \right) = \text{Ft.}^3 =$$

$$\frac{17.7 \times 85.20 (29.3 + \frac{1.95}{13.6})}{585}$$

$$= 75.23$$

2. Volume of water vapor at 70°F and 29.92" Hg, Ft.³

$$V_{w_{gas}} = 0.0474 \times V_w = \text{ft.}^3$$

$$= V_{w_{gas}} = .0474 \times 14.4$$

$$= 0.68$$

3. % moisture in stack gas

$$\%M = \frac{100 \times V_{w_{gas}}}{V_{m_{std}} + V_{w_{gas}}} = \%$$

$$= \%M = \frac{100 \times 0.68}{75.23 + 0.68} = \frac{68}{76.91} =$$

$$= 0.88$$

4. Mole fraction of dry gas

$$M_d = \frac{100 - \%M}{100}$$

$$= M_d = \frac{100 - 0.88}{100} = \frac{99.1}{100}$$

$$= 0.99$$

5. Average molecular weight of dry stack gas

$$M\ W_d = (\%CO_2 \times \frac{44}{100}) + (\%O_2 \times \frac{32}{100}) + (\%N_2 \times \frac{28}{100})$$

$$= (0.0 \times .44) + (20.8 \times .32) + 79.2 \times .28)$$

$$= 28.84$$

6. Molecular weight of stack gas

$$M\ W = M\ W_d \times M_d + 18 (1 - M_d)$$

$$= MW = 28.84 \times .991 + 18(1 - .991)$$

$$= 28.74$$

7. Stack velocity @ stack conditions, fpm

$$V_s = 4350 \times \sqrt{\Delta P_s \times (T_s + 460)} \left[\frac{1}{P_s \times M\ W} \right]^{1/2} = \text{fpm}$$

$$= 4350 \sqrt{0.47 \times (583)} \left[\frac{1}{29.28 \times 28.74} \right]^{1/2}$$

$$= 2396$$

8. Stack gas volume @ standard conditions, SCFM

$$Q_s = \frac{0.123 \times V_s \times A_s \times M_d \times P_s}{(T_s + 460)} = \text{SCFM}$$

$$= \frac{0.123 \times 2396 \times 2826 \times .991 \times 29.28}{(583)}$$

$$= 41,492$$

9. Percent isokinetic

$$\%I = \frac{1032 \times (T + 460) \times V_m}{V_s \times T_t \times P_s \times M_d \times (D_n)^2} = \%$$

$$= \frac{1032 \times (583) \times 85.20}{2396 \times 108 \times 29.28 \times .991 \times .0625}$$

$$= 109$$

10. Particulate - probe, cyclone, and filter, gr/SCF

$$C_{an} = 0.0154 \times \frac{M_f}{V_{m_{std}}} = \text{gr/scf}$$

$$= 0.0154 \times \frac{688.3}{75.23} = (0154)$$

$$= 0.141$$

11. Particulate total, gr/SCF

$$C_{ao} = 0.0154 \times \frac{M_t}{V_{m_{std}}} = \text{gr/SCF}$$

$$= .0154 \times \frac{754.5}{75.23}$$

$$= 0.154$$

12. Particulate - probe, cyclone and filter,
gr/CF at stack conditions

$$C_{at} = \frac{17.7 \times C_{an} \times P_s \times M_d}{(T_s + 460)} = \text{gr/CF}$$
$$= \frac{17.7 \times .141 \times 29.28 \times .991}{583}$$
$$= 0.124$$

13. Particulate - total, gr/CF @ stack conditions

$$C_{au} = \frac{17.7 \times C_{ao} \times P_s \times M_d}{(T_x + 460)} = \text{gr/CF}$$
$$= \frac{17.7 \times 0.154 \times 29.28 \times .991}{583}$$
$$= 0.136$$

14. Particulate - probe, cyclone filter filter, 1b/hr.

$$C_{aw} = 0.00857 \times C_{an} \times Q_s = 1\text{b/hr.}$$
$$= 0.00857 \times .141 \times 41,492$$
$$= 50.2$$

15. Particulate - total, 1b/hr.

$$C_{ax} = 0.00857 \times C_{ao} \times Q_s = 1\text{b/hr.}$$
$$= .00857 \times .154 \times 41,492$$
$$= 54.8$$

16. % excess air at sampling point

$$\% EA = \frac{100 \times \% O_2}{(0.266 \times \% N_2) \% O_2} = \%$$

$$= \frac{100 \times 20.8}{(0.266 \times 79.2) - 20.8}$$

$$= 7700$$

DETERMINATION OF MATERIAL COLLECTED IN ALUNDUM FILTER (ASME TRAIN)

<u>Sample Location</u>	<u>Date Sampled</u>	<u>Time Sampled</u>	<u>Sample Number</u>	<u>grams</u>	<u>grains</u>	<u>Vstd-Metered Gas Vol.</u> <u>(dry, STD)</u>	<u>grains/cu ft</u>	<u>lbs/hr</u>
25 Stack East	5-18-71	1438-1727	25E-1	1.2072	18.63	61.80	0.301	179.4
25 Stack West	5-19-71	0953-1130	25W-2	0.3481	5.37	25.80	0.208	202.2
	5-19-71	0953-1130	25W-2	0.3575*	5.37*	25.80	0.213*	207.*
6 Stack Exhaust	5-20-71	1129-1343	6E- 1	0.8176	12.62	69.00	0.183	60.4
	5-20-71	1534-1745	6E- 2	0.9522	14.69	67.20	0.219	72.3

* These figures include acetone wash from the probe and nozzle.

SAMPLE CALCULATIONS: 6FE-1

1. Isokinetic Sampling (Dry Gas) (cfm)

$$R_m = .33 \times \frac{T_m \times V_s \times d^2}{T_s} \times \frac{P_s}{P_b - P_m} : R_m (\text{corrected}) = R_m \times M_c$$

$$\text{Estimated Value: Average } .33 \times \frac{550}{600} \times 38.0 \times 0.0625 \times \frac{29.3}{22.0} = .83 \times .98 = .82$$

Calculated Value:

2. Water Vapor Volume (cu. ft.)

$$V_v = .00267 \times \frac{V_w \times T_m}{P_b - P_m} = 0.00267 \times \frac{-50 \times 566}{22.6} = -3.36$$

3. Condensate Correction for Meter Rate

$$M_c = \frac{V_m}{V_m + V_v} \times \frac{103.3}{103.3 - 3.4} = 1.03$$

4. Moisture in Metered Gas (cu. ft.)

$$M_m = \frac{V.P. \times V_m}{P_b - P_m} = \frac{1.21 \times 103.32}{22.6} = 5.53$$

5. Percent Moisture (%) Stack Gas

$$\% \text{ Moisture} = \frac{V_v + M_m}{V_v + V_m} \times 100 = \frac{-3.36 + 5.53}{-3.36 + 103.32} = 2.17$$

Saturated at 19.6 %

6. Flue Gas Volume (cfm)

$$\text{as is} = V_o = V_s, \text{ avg.} \times A \times 60 = 19.60 \times 2280 = 44,700$$

$$\text{dry, std cond} = V_o \times \frac{528^{\circ}\text{R}}{T_s} \times \frac{100 - \% \text{ Moisture}}{100} = 44,700 \times \frac{528}{600} \times 978 = 38,500$$

7. Sampled Volumes converted to "standard" conditions (cu. ft.)

$$V_{dg} = V_m - M_m \quad V_{std} = V_{dg} \times \frac{528^{\circ}\text{R}}{T_m} \times \frac{P_b - P_m}{29.92} = \\ 97.79 \times \frac{528}{566} \times \frac{22.6}{29.9} = 69.0$$

APPENDIX B
COMPLETE GASEOUS RESULTS WITH EXAMPLE CALCULATIONS

DETERMINATION OF SO₂ EMISSIONS *

<u>Sample Location</u>	<u>Date Sampled</u>	<u>Time Sampled</u>	<u>Sample Number</u>	<u>milligrams</u>	<u>Vstd-Metered Gas Vol.</u> <u>(dry, STD)</u>	<u>milligrams/cu ft</u>	<u>factor</u>	<u>ppm</u>
25 Stack East	5-19-71	0950-1050	25E-1	1.8	1.74	1.03	13.1	13.5
		1200-1310	2	1.3	1.59	0.818		10.7
25 Stack West	5-18-71	1430-1650	25W-1	9.6**	7.40	1.30**		17.0**
6 Stack Exhaust	5-20-71	1130-1340	6E- 1	0.0	2.98	0.0	0.0	0.0
		1530-1740	2	0.0	2.78	0.0		0.0

* This special format was used instead of the OAP forms because the meter was kept under vacuum, that is before the pump.

** These figures represent total sulfur oxide (SO_x) since the isopropyl impinger was mistakenly filled with 3% hydrogen peroxide.

*** From page 173, Source Testing Manual, County of Los Angeles, California.

CALCULATION OF DRY, STANDARD METER VOLUMES FOR THE SO₂ TRAIN

25W-1) Metered volume - 11.82 CF
 Avg. meter vac. - 7.2 in. of Hg.
 Avg. meter temp. - 103 °F
 Avg. Impinger temp. - 103 °F
 Barometric press. - 29.3 in. of Hg.

Moisture in Metered Gas (CF).

$$\frac{2.11^* \times 11.82}{22.1} = 1.13 \quad * \text{Vapor press. of H}_2\text{O (in. of Hg)} \\ @ 103^{\circ}\text{F.}$$

Dry Standard Meter Volume (CF)

$$\frac{11.82}{1.13} \quad 10.69 \times \frac{530}{563} \times \frac{22.1}{29.92} = 7.40$$

$$\frac{10.69}{10.69}$$

25E-1) Metered volume - 3.40 CF
 Avg. meter vac. - 10.6 in. of Hg.
 Avg. meter temp. - 106 °F
 Avg. Impinger temp. - 106 °F
 Barometric press. - 29.3 in. of Hg.

Moisture in Metered Gas (CF).

$$\frac{2.31^* \times 3.40}{18.7} = 0.42 \quad * \text{Vapor press. of H}_2\text{O (in. of Hg)} \\ @ 106^{\circ}\text{F.}$$

Dry Standard Meter Volume (CF)

$$\frac{3.40}{0.42} \quad 2.98 \times \frac{530}{566} \times \frac{18.7}{29.9} = 1.74$$

$$\frac{2.98}{2.98}$$

25E-2) Metered volume - 3.30 CF
 Avg. meter vac. - 11.0 in. of Hg.
 Avg. meter temp. - 112 °F
 Avg. Impinger temp. - 112 °F
 Barometric press. - 29.3

Moisture in Metered Gas (CF).

$$\frac{2.75^* \times 3.30}{18.3} = 0.49 \quad * \text{Vapor press. of H}_2\text{O (in. of Hg.)} \\ @ 112^{\circ}\text{F}$$

Dry Standard Meter Volume (CF).

$$\frac{3.30}{0.49} \quad 2.81 \times \frac{530}{572} \times \frac{18.3}{29.9} = 1.59$$

$$\frac{2.81}{2.81}$$

ORSAT FIELD DATALocation 25E

Comments:

Date May 18, 1971Time During first hour particulate testOperator N.A.Blessing, C.C. Gonzalez

Test Run	(CO ₂) Reading 1	(O ₂) Reading 2	(CO) Reading 3
1	0.8	20.4	0.0
2	0.8	20.2	0.0
Avg.	0.8	20.3	0.0

ORSAT FIELD DATA

Location 25W Comments:
 Date May 19, 1971
 Time 25 W-1, 25 W-2 During first and second hour particulate test.
 Operator C. C. Gonzalez

Test	(CO ₂) Reading 1	(O ₂) Reading 2	(CO) Reading 3
25W - 1	0.5	20.1	0.0
-2	0.5	20.1	0.0
Avg.	0.5	20.1	0.0

ORSAT FIELD DATALocation 6E Comments:Date May 20, 1971Time 16E-1 (1129-1229) 36E-2 (1645-1745)
26E-2 (1534-1634)Operator C. C. Gonzalez

Test	(CO ₂) Reading 1	(O ₂) Reading 2	(CO) Reading 3
1 (6 E-1)	0.0	20.8	0.0
2 (6 E-2)	0.2	20.7	0.0
3 (6 E-2)	0.0	20.8	0.0
Avg.	0.1	20.8	0.0

APPENDIX C

COMPLETE OPERATION RESULTS WITH EXAMPLE CALCULATIONS
(Not Applicable - Plant Operating Data in Appendix G)

APPENDIX D

Field Data

PARTICULATE FIELD DATA

1 OF 2VERY IMPORTANT - FILL IN ALL BLANKS

Read and record at the start of each test point or, if single point sampling, read and record every 5 minutes.

Plant Foote MineralsRun No. 25E-1Sample Box No. RAC 2Bar. Press. "Hg 29.3Location 25E-1Meter Box No. RAC 2Assumed Moisture % 4%Date May 18, 1971Probe Length 5'Heater Box Setting, °F 250Operator Blessing and BaxleyProbe Heater Setting 65Prob Tip Dia., In. 3/16

Point	Clock Time	Dry Gas Meter, CF	Pitot in. H ₂ O	Orifice ΔH in. H ₂ O	Dry Gas Temp. °F	Box Temp. °F	Impinger Temp °F	Stack Press in. Hg	Stack Temp °F
E-1	1438	446.00			100	100	250	70	.80
E-1	1440	449.01	1.90	1.84	112	90	100	90	.83
E-1	1447	451.40	1.70	1.70	1.65	120	90	11.0	.84
E-2	1450	453.80	1.70	1.70	1.70	122	100	17.0	100
E-2	1453	455.90	1.60	1.70	1.60	122	100	18.0	100
E-3	1456	457.42	1.60	1.70	1.60	123	100	21.0	100
E-3	1459	459.48	1.70	1.70	1.70	130	102	23.0	100
E-4	1502	462.18	1.70	1.70	1.10	130	102	23.0	100
CHANGE	1528	464.34	1.70	1.70	1.70	114	102	5.0	100
FILTER	1531	466.00	1.75	1.70	1.75	126	102	6.0	75
E-5	1534	468.79	1.70	1.70	1.70	130	102	7.0	70
E-6	1537	470.73	1.75	1.70	1.75	130	102	8.0	70
E-6	1540	472.92	1.70	1.70	1.70	134	110	9.0	75
E-7	1543	475.13	1.70	1.70	1.70	138	110	13.0	75
E-7	1546	477.01	1.70	1.70	1.70	138	110	13.0	75
E-8	1549	479.12	1.75	1.70	1.75	142	110	16.0	90
E-8	1552	481.26	1.70	1.70	1.70	144	110	23.0	90
E-9	1555	483.10	1.70	1.70	1.70	146	110	24.0	90
E-9	1558	485.40	1.70	1.70	1.70	146	110	25.0	90

D

-

(CONTINUED)

Foote Minerals - Run No. 25E-1

Point	Clock Time	Dry Gas Meter, CF	Pitot in. H ₂ O ΔP	Orifice ΔH in H ₂ O		Dry Gas Temp. °F	Pump Vacuum in. Hg	Box Temp. °F	Impinger Temp °F	Stack Press in. Hg	Stack Temp °F
				Desired	Actual						
N-1	1625	485.40	.90	.90	.90	120	110	6.0	250	80	.32
N-1	1628	488.95	.93	.90	.95	122	110	9.0		80	.32
N-2	1631	489.72	1.04	.90	.85	130	110	11.0		80	.55
N-2	1634	491.67	1.04	.90	.90	130	110	12.0		80	.58
N-3	1637	492.46	1.02	.90	.90	130	110	12.0		80	.52
N-3	1640	494.20	1.03	.90	.95	130	110	14.0		90	.55
N-4	1643	495.97	1.04	.90	.85	136	110	15.0		90	.70
N-4	1646	498.10	1.03	.20	.20	140	110	22.0		90	.77
N-5	1649	499.79	1.03	.90	.85	140	112	17.0		95	.60
N-5	1652	501.76	1.04	.92	.95	144	112	13.0		90	.68
N-6	1655	503.03	1.04	.90	.90	144	112	20.0		90	.68
N-6	1658	504.75	1.03	.90	.90	144	112	21.0		80	.75
N-7	1701	506.38	1.04	.90	.90	148	112	22.0		80	.76
N-7	1704	508.00	1.04	.90	.85	148	112	24.0		85	.76
N-8	1707	509.37	1.04	.90	.82	148	112	25.0		80	.78
N-8	1717	511.20	1.70	1.70	1.70	140	110	3.0		70	1.03
N-8	1720	513.35	1.70	1.70	1.70	142	110	9.0		70	1.03
N-9	1723	515.60	1.80	1.70	1.60	144	110	11.0		80	1.03
N-9	1726	517.50	1.80	1.70	1.70	144	112	13.0		35	1.03
N-10	1729	519.40	1.90	1.70	1.70	146	112	15.0		90	1.04
N-10	1732	522.12	1.80	1.70	1.70	146	112	13.0		90	1.03
	76.12	1.41		1.40	1.35	134	113	14.9		85	0.34
										280	

Comments:

NCAP-37 (12/67)

PARTICIPATE FIELD DATA

VERY IMPORTANT - FILL IN ALL BLANKS

Read and record at the start of each test point or, if single point sampling, read and record every 5 minutes.

Plant Foote Minerals

RUT No. 25E-2

Location 25E

Date May 19, 1971

Operator Blessing and Baxley

Scutellate 89% 113. RAC-2

RAC-2

Psychol Encyc 5'

Probe Heater Setting 6

THE JOURNAL OF

Scutellate 89% 113. RAC-2

Meter Box No. RAC-2

Ambient Temp. °F

Bar. Press: "Hg 29.3

Assumed moisture % 2.0

Hastings 250

Prob. Tip Diga., In. 3/16

PARTICULATE FIELD DATA

VERY IMPORTANT - FILL IN ALL BLANKS

Read and record at the start of each test point or, if single point sampling, read and record every 5 minutes.

Ambient Temp °F

Run No. Preliminary Test No. 2

תְּהִלָּה בְּשִׁירָה יְהוָה

Location 25W

卷之三

Date May 17, 1971

Run No.	Preliminary Traverse	Sample Box No.	Bar. Press. "Hg	Meter Box No.	Assumed Moisture %	Heater Box Setting, °F	Probe Length	Probe Heater Setting	Probe Tip Dia., In.	
Location	25W									
Date	May 17, 1971									
Operator	Gonzalez and Baxley									
Inches In Point	Clock Time	Dry Gas Water, CF S ΔP	Pitot in. H ₂ O S ΔP	Orifice ΔH in H ₂ O Desired	Dry Gas Temp. °F Actual	Pump Vacuum In. Hg Gauge	Box Temp. °F	Impinger Temp. °F	Stack Press in. Hg	Stack Temp. °F
1.20	1500		1.6	1.3						
4.00			2.1	1.7						
7.10			2.1	2.0						
10.60			2.1	2.0						
15.00			2.5	2.4						
21.00			2.6	2.5						
38.80			2.5	2.7						
45.00			3.1	2.9						
49.50			3.0	3.1						
53.00			3.0	3.2						
56.00			3.0	3.0						
58.80	1530		2.8	3.1						

Foote Minerals - Run No.25W-1(cont.) - 25 West Stack - 5/18/71

PAGE 2 OF 2

Point	Clock Time	Dry Gas Meter, CF	Pitot in. H ₂ O AP	Orifice ΔH in H ₂ O		Dry Gas Temp. °F	Pump Vacuum In. Hg	Box Temp. °F	Impinger Temp °F	Stack Press in. Hg	Stack Temp °F
				Desired	Actual						
W-1	1625	55.38	-	-	-	95	N.A.	1.5	-	-	-
	1628		0.51	0.57	0.57	95	N.A.	1.5	-	-	220
W-1	1631	58.23	0.54	0.56	0.56	95	N.A.	1.5	-	-	220
	1634		2.30	2.1	2.1	96	N.A.	6.2	-	-	220
W-2	1637	62.34	2.30	2.1	2.1	96	N.A.	6.2	-	-	220
	1640		2.50	2.9	2.9	95	N.A.	8.5	-	-	220
W-3	1643	67.72	2.50	2.9	2.9	96	N.A.	9.0	-	-	220
	1646		2.60	3.0	3.0	95	N.A.	9.0	-	-	220
W-4	1649	74.32	2.60	3.0	3.0	95	N.A.	9.0	-	-	220
	1652		2.90	3.2	3.2	96	N.A.	10.0	-	-	220
W-5	1655	79.68	2.90	3.2	3.2	96	N.A.	10.0	250	Ice	220
	1658		3.10	3.7	3.7	96	N.A.	10.5	Bath	-	220
N-6	1701	86.10	3.10	3.7	3.7	95	N.A.	11.0	-	-	210
	1704		2.70	3.2	3.2	97	N.A.	10.0	-	-	210
W-7	1707	92.40	2.60	3.0	3.0	98	N.A.	10.5	-	-	213
	1715		-	-	-	-	-	-	-	-	-
	1718		2.50	2.9	2.9	98	N.A.	7.0	-	-	220
W-8	1721	97.45	2.60	3.0	3.0	98	N.A.	7.5	-	-	220
	1724		3.00	3.5	3.5	98	N.A.	8.5	-	-	218
W-9	1727	105.25	3.00	3.5	3.5	98	N.A.	8.5	-	-	220
Avg.		105.25	2.47	2.8	2.8	97	N.A.	8.2	250	.90	218

Comments:

Condensate = 0

NCP-37 (12/67)

PARTICULATE FIELD DATA

1 OF 2(2FN-A6
(2FN-BIK
(2FN-P
(2FN-A1VERY IMPORTANT - FILL IN ALL BLANKS

Read and record at the start of each test point or, if single point sampling, read and record every 5 minutes.

Plant Foote MineralsRun No. 25W-2Location #25 West StackDate May 19, 1971Operator SchroederSample Box No. Reston RigMeter Box No. Reston RigProbe Length 5'Probe Heater Setting 65Bar. Press. "Hg 29.3Assumed Moisture % 2Heater Box Setting, °F 250Probe Tip Dia., In. 3/16

Point	Clock Time	Dry Gas Meter, CF in. H ₂ O ΔP	Pitot in. H ₂ O	Orifice ΔH in H ₂ O	Dry Gas Temp. °F	Plenum Vacuum In. Hg	Box Temp. °F	Impinger Temp °F	Stack Press in. Hg	Stack Temp °F
N-1	953	000.00	-	-	-	N.A.	-	250	Ice Bath	
	956	-	1.40	1.4	85	N.A.	3.5	Assumed		190
	959	03.54	1.40	1.4	36	N.A.	6.5	50°		190
	1002	-	1.70	1.6	86	N.A.	9.5			200
N-2	1005	07.35	1.70	1.6	86	N.A.	10.5			200
	1013	-	-	-	-					-
	1016	-	1.90	1.9	86	N.A.	17.5			200
N-3	1019	11.04	1.90	1.9	86	N.A.	21.0			200
	1030	-	-	-	-	N.A.	-			-
	1033	-	2.0	2.0	36	N.A.	4.5			200
N-4	1036	14.78	2.0	2.0	86	N.A.	5.0			200
	1039	-	2.2	2.1	87	N.A.	7.0			200
N-5	1042	19.35	2.2	2.1	88	N.A.	7.0			200
	1045	-	2.6	2.4	88	N.A.	17.0			200

(CONTINUED)

FOOTER MINERALS - Run No. 25W-2 - - - - - #25 West Stack - May 19, 1971

FOOTOE MINERALS - Run No. 25N-2 - #25 West Stack - May 19, 1971

TEST NO. 22W-2 - MAY 15, 1971									
Point	Clock Time	Dry Gas Meter, CF	pitot in. H ₂ O	Orifice ΔH in H ₂ O	Dry Gas Temp. °F	Pump Vacuum in. Hg	Box Temp. °F	Impinger Temp °F	Stack Press in. Hg
N-6	1040	23.80	2.6	2.4	2.1	88	N.A.	20.0	250
	1050	-	-	-	-	-	-	-	-
N-7	1104	28.56	2.6	2.4	2.4	79	N.A.	4.5	200
	1105	-	-	-	-	-	-	-	-
N-8	1111	32.31	2.0	2.4	2.2	90	N.A.	12.5	210
	1114	-	-	-	-	-	-	-	-
N-9	1122	36.70	2.5	2.3	1.9	90	N.A.	21.0	220
	1125	-	-	-	-	-	-	-	-
	1126	-	-	-	-	-	-	-	-
	Avg.	38.70	2.2	2.1	2.1	85	-	10.6	204

Comments: Condensate = 0

CAP-37 · (12/67)

PARTICULATE FIELD DATA

10F 2VERY IMPORTANT - FILL IN ALL BLANKS

Read and record at the start of each test point or, if single point sampling, read and record every 5 minutes.

Plant Foot Minerals

Run No. 6E-2
Location 6E
Date 5/20/71

Operator Blessing

Sample Box No. RAC-2
Meter Box No. RAC-2
Probe Length 5'
Probe Heater Setting 65

Ambient Temp °F 100
Bar. Press. "Hg 29.3
Assumed Moisture % 1.
Hecter Box Setting, °F 250
Probe Tip Dia., In. 1/4

Point	Clock Time	Dry Gas Meter, CF	Pitot in. H ₂ O ΔP	Orifice ΔH in H ₂ O		Dry Gas Temp. °F	Pump Vacuum	Box Temp. °F	Impinger Temp °F	Stack Press in. Hg	Stack Temp °F
				Desired	Actual						
1	1533	651.68	.33	1.40	1.40	116	116	3.0	250	105	.30
1	1536	653.66	.33	1.40	1.40	118	117	3.0		90	.30
1	1539	655.64	.33	1.40	1.35	120	118	3.0		90	.30
1	1542	657.66	.31	1.30	1.30	122	118	3.0		90	.20
2	1545	659.77	.40	1.65	1.65	126	118	3.5		90	.25
2	1548	661.89	.40	1.65	1.60	130	118	3.5		90	.25
3	1551	664.15	.40	1.65	1.70	132	118	4.5		90	.25
3	1554	666.51	.43	1.70	1.70	140	118	5.0		87	.30
4	1557	668.59	.45	1.80	1.80	140	118	5.0		85	.30
4	1600	671.15	.50	1.95	1.95	144	118	6.0		83	.30
5	1603	673.35	.45	1.85	1.85	144	118	6.0		85	.25
5	1606	675.81	.50	1.95	1.95	150	118	6.5		90	.30
6	1609	678.06	.50	1.95	1.95	152	118	7.0		90	.30
6	1612	680.52	.55	2.15	2.15	154	118	7.5		90	.30
7	1615	683.00	.55	2.15	2.15	156	119	8.0		90	.35
7	1618	685.44	.50	1.95	1.95	156	119	8.0		90	.35
8	1621	687.84	.50	1.95	1.95	158	119	8.5		90	.30
8	1624	690.27	.50	1.95	1.95	158	119	9.0		92	.30
9	1627	692.62	.50	1.95	1.95	158	119			165	

Foote Minerals, Run No. 6E-2

Contents:

NCAP-37 · (12/67)

D-17

VERY IMPORTANT - FILL IN ALL BLANKS

Read and record at the start of each test point or, if single point sampling, read and record every 5 minutes.

Plant Foote Minerals

Run No. 6E-1

6E

卷之三

לט

卷之三

Plant Foote Minerals

82111 No 6E-1

1 locution 6F

卷之三

卷之三

卷之三

minutes.		Ambient Temp °F	100
Sample Box No.	RAC-2	Bar. Press. "Hg	29.3
Meter Box No.	RAC-2	Assumed Moisture %	2
Probe Length	5'	Heater Box Setting, °F	250
Probe Heater Setting	65	Probe Tip Dia. In	3/16

Point	Clock Time	Dry Gas Meter, CF	Pitot in. H ₂ O	Orifice ΔH in H ₂ O	Dry Gas Temp. °F	Pump Vacuum in. Hg	Box Temp. °F	Impinger Temp. °F	Stack Press. in. Hg	Stack Temp. °F
0	1129	566.48	.40	1.7	1.7	108	112	2.5	250	110
	1132	568.61	.40	1.7	1.7	112	108	2.5		80
1	1135	570.80	.40	1.7	1.7	116	106	2.5		85
	1138	573.04	.45	1.9	1.9	120	106	2.5		90
2	1141	575.39	.45	1.9	1.9	122	106	2.5		90
	1144	577.62	.40	1.7	1.7	124	106	2.5		90
3	1147	579.85	.40	1.7	1.7	125	105	2.5		90
	1150	582.15	.45	1.9	1.9	130	106	2.5		95
4	1153	584.50	.45	1.9	1.9	132	106	2.5		95
	1156	586.90	.50	2.0	2.0	134	106	2.9		95
5	1159	589.33	.50	2.0	2.0	138	108	3.0		95
	1202	591.74	.50	2.0	2.0	140	108	3.0		95
6	1205	594.15	.50	2.0	2.0	141	108	3.0		95
	1208	596.55	.50	2.0	2.0	142	108	3.0		95
7	1211	598.95	.50	2.0	2.0	144	109	3.0		95
	1214	601.32	.45	1.9	1.9	144	109	3.0		95
8	1217	603.78	.45	1.9	1.9	144	109	3.0		95
	1220	606.20	.55	2.3	2.3	146	109	3.5		95
9	1223	608.65	.55	2.3	2.3	146	110	3.5		95

(CONTINUED)

Foote Minerals, Run No. 6E-1

Point	Cijack Time	Dry Gas Ketter, CF	Pitot in. H ₂ O ΔP	Orifice ΔH in. H ₂ O		Dry Gas Temp. °F		Pump Vacuum In. Hg Gauge	Box Temp. °F	Impinger Temp. °F	Stack Press. in. Hg	Stack Temp. °F
				Desired	Actual	Inlet	Outlet					
0	1242	608.77	.35	1.5	1.5	110	2.8		90	.15	.15	125
	1245	610.60	.30	1.25	1.25	110	2.5		80	.15	.15	125
1	1248	612.81	.35	1.5	1.5	132	110		70	.15	.15	125
	1251	614.80	.35	1.5	1.5	134	110		70	.20	.20	125
2	1254	617.02	.40	1.7	1.7	136	112		75	.20	.20	135
	1257	619.20	.40	1.7	1.7	138	114		80	.20	.20	130
3	1300	621.40	.40	1.7	1.7	140	114		80	.20	.20	140
	1303	623.71	.45	1.8	1.8	140	116		80	.20	.20	140
4	1306	626.05	.45	1.8	1.8	142	116		80	.20	.20	140
	1309	628.52	.50	2.1	2.1	144	118		85	.20	.20	140
5	1312	630.90	.50	2.1	2.1	144	118		87	.30	.30	140
	1315	633.33	.50	2.1	2.1	146	120		90	.30	.30	140
6	1318	636.01	.53	2.2	2.2	146	120		90	.30	.30	138
	1401	638.38	.53	2.2	2.2	150	118		90	.30	.30	142
7	1424	641.12	.59	2.45	2.45	156	118		90	.32	.32	140
	1427	643.65	.65	2.70	2.70	158	118		90	.33	.33	132
8	1430	646.40	.65	2.70	2.70	160	119		90	.33	.33	135
	1433	649.05	.60	2.45	2.45	160	119		90	.34	.34	140
9	1436	651.68	.60	2.45	2.45	162	120		90	.35	.35	145
		85.20	.47	1.96	1.96	112	3.5	250	89	0.22	123	
										213.6=02		

Comments:

NCAP-37 (12/67)

D-13

RESOURCES RESEARCH, INC.

Page 2 of 2

Personnel _____ Entered _____
Checked _____

Normal - ASME Test
Plant Conditions, etc. _____
Plant Reactive Metals, Steubenville, Ohio Date 5/20/71
Sample Location #6 Furnace Exhaust

Stack/Duct Measurement _____ Estimated Mc- _____ Actual - _____

Stack Diameter, inches (D) _____ Estimated Rn(Corrected) _____

Stack Area, Ft², (A) _____ Calculated Rm(Corrected) _____

Final Gas Temp Op Sample Nozzle Dia.,In. (d) _____

Starting Gas Temp ${}^{\circ}\text{F} + 460 = \text{Ts}$ ${}^{\circ}\text{R}$ % Moisture of Gas _____

Barometric Pressure, "Hg, (Pb) _____ Avg. Gas Velocity, fpm(Vs_x60) _____

$$\text{Stack Static Pressure, In. H}_2\text{O} \quad + \quad \text{Vs} = (2.9 \times F_s) \times \sqrt{H} \times \sqrt{T}$$

Condensate, ml (Vw) _____ or 2.46

Flue Gas Volume, as is (V₀) _____ CFM

Type Pitot: Standard Type S _____

PITOT READINGS (A.M. P.M.)

In. from side H₁ (In, H₂O)

D-20

D-2Q

RESOURCES RESEARCH, INC.

Personnel CCG

Page 1 of 1

Entered

• Checked

Plant Conditions, etc. Normal - SO₂ Train

Plant: Reactive Metals, Steubenville, Ohio

Date 5/19/71

Sample Location

SO₂ Train

RESOURCES RESEARCH, INC.

Personnel CCG

Page 1 of 1

Entered

• Checked

Normal - SO_2 Train
Plant Conditions, etc.

Plant Reactive Metals, Steubenville, Ohio

Date 5/20/71

Sample Location

RESOURCES RESEARCH, INC.

Page 1 of 1

Personnel CCG Entered

Entered _____

Checked

Plant Conditions, etc. _____ Normal - SO_2 Train

Plant Reactive Metals, Steubenville, Ohio Date 5/18/71

Sample Location 25W Stack

NOTE: The first impinger (isopropyl alcohol) contained 3 percent H_2O_2 by mistake; therefore, total SO_x was collected instead of the split into SO_2 .

APPENDIX E

1. STANDARD SAMPLING PROCEDURES
2. CLEANUP AND ANALYTICAL PROCEDURES

APPENDIX E.1 STANDARD SAMPLING PROCEDURES

PARTICULATE SAMPLING

In an unstable operation a trial run is conducted. Otherwise, preliminary data are obtained for gas velocity, temperature, and other variables which might affect the isokinetic sampling rate. Two 12-point, equal area traverses, along two axes, were selected as being most appropriate for the conditions encountered at each exhaust stack. Each sampling was designed to cover one complete operating and tapping cycle.

Particulate samples were obtained using the equipment and test procedures as stipulated in "Sample Collection Procedures," published by OAP. The sampling train was basically the same as that designed by the Control Development Program of OAP (formerly the Air Pollution Control Office), "Gas Stack Sampling Improved and Simplified with New Equipment," and described in Paper No. 67-119, presented at the Air Pollution Control Association meeting in June 1967, Cleveland, Ohio.

The sample gases were drawn into the all-glass sampling train through a button-hook stainless steel nozzle with a diameter of 0.1875 inch. A pyrex glass probe was fitted inside the stainless steel sheath with a probe heating element. The glass probe was connected to a glass cyclone and an Erlenmeyer flask to collect the solids from the cyclone. The sampled gases passed from the cyclone through a tared 2-1/2 inch diameter MSA 1106BH glass fiber filter. This filter and the cyclone were enclosed in a heated box which was maintained near 250°F. The

filter holder was connected to an impinger train consisting of four Greenburg-Smith impingers with the high velocity tip removed from the first impinger. The second impinger was used with the tip while the third and fourth impingers were modified as the first. The first two impingers each contained a measured volume (100 ml) of distilled, deionized water. The third impinger was used dry and the fourth impinger contained approximately 175 grams of silica gel. The sampling train exit was connected, in line, to a vacuum gauge, a leakless vacuum pump, a dry gas meter, and a calibrated orifice. The calibrated orifice differential was measured with an inclined-vertical manometer. Velocity variations at the sampling point were constantly monitored by a pitot tube connected to the probe sheath. The sampling train, with probe and nozzle attached, was leak tested prior to each test.

Isokinetic sampling was maintained by appropriate adjustment of the sampling rate as indicated by the pressure drop across the orifice following the dry gas meter. The necessary orifice pressure differential was determined by using the nomographs presented in APCA Paper No. 67-119. This nomograph related stack gas velocity, temperature, and moisture content to the flow rate required for isokinetic sampling.

The ASME train run in conjunction with the OAP train consisted of a stainless steel filter holder containing a pre-weighed alundum filter. The sample was drawn through a stainless steel probe and nozzle into a set of water filled Greenburg-Smith impingers. Isokinetic sampling rates were not determined during the test but were precalculated from initial pitot and temperature readings. Only the material collected by the alundum filter is normally considered as particulate.

SULFUR DIOXIDE SAMPLING

Sulfur dioxide emission tests were conducted at the same location as the particulate tests. The sample gas was drawn through a glass wool filter into a probe followed by a coarse frit midget impinger and a second glass wool filter. The filter led to three midget impingers in an ice bath followed in turn by a silica gel tube drier, vacuum gauge, valve, leakless pump with by-pass valve, dry gas meter, rate meter, and pitot tube with manometer.

The midget bubbler contained 15 ml of 80 percent isopropyl alcohol. The first two midget impingers contained 15 ml of 3 percent H_2O_2 solution and the third was operated dry. A dry gas meter with vacuum gauge and a pump followed the impingers. Temperatures, vacuum and gas meter readings were taken and tabulated in order to calculate standard volumes. After sampling, the train was purged with clean air in order to carry over any SO_2 trapped in the isopropyl

ORSAT SAMPLING

An integrated gas sample was obtained with a mylar bag and a peristaltic pump with adjustable flow rate. The gases were filtered and cooled prior to reaching an all plastic and glass flow meter where the sampling rate was monitored. Gas samples were taken during the same period during which velocities, temperatures, and particulate samples were obtained. Analyses were performed at the site immediately after each sampling was collected.

APPENDIX E.2
CLEANUP AND ANALYTICAL PROCEDURES

CLEANUP (OAP PARTICULATE TRAIN)

Probe, Nozzle, Cyclone, and Front Half of Filter Holder

The nozzle, probe, cyclone, flask, and front half of the filter holder were washed with reagent grade acetone. Washings were collected in a container and transported to the laboratory for analysis. A rubber policeman was used with the acetone to remove any particles adhering to the cyclone walls or the flask. The reagent acetone used for washing was tested to determine the blank or residue upon evaporation.

Filter

The tared circular MSA type 1106BH filter paper was carefully removed from the fritted glass support and transferred to a glass petri dish for later weighing.

Impingers

Water in the first three impingers (the original water plus the condensate) was measured, then emptied into a polyethylene container. The impingers were then water washed; the washings were combined with the condensate and the original water.

Acetone Train Wash

The rear half of the filter holder, including the fritted glass support, the impingers, and impinger connections up to but excluding the fourth impingers, were washed with acetone. These washings were collected in a polyethylene bottle and sealed for later analysis.

Silica Gel

Silica gel was transferred (dry) from the fourth impinger to an airtight container and sealed. The impinger was then washed with acetone, the acetone being discarded because it contained fine silica gel particles.

CLEANUP (SO₂ TRAIN)

The impinger containing 80 percent isopropyl alcohol was discarded and the impingers containing 3 percent H₂O₂ saved. These contained SO₂ gas in the form of H₂SO₄. A glass jar was used as a sample container for transportation to the laboratory for analysis.

ANALYTICAL PROCEDURES (OAP PARTICULATE TRAIN)

Acetone Washings

The acetone washings from the nozzle, probe, cyclone and flask; from the front and back of the filter; and from the impinger train were analyzed separately by evaporation and drying at ambient temperatures.

Filter Particulate

The filter and particulate collected thereon were dried for 24 hours in a desiccator at ambient temperature and weighed. Tare weight of the filter was then deducted.

Impinger Water

Water collected in the impingers, along with the water washings of the impingers, was extracted with ether and chloroform. The extracts were transferred to a tared dish and evaporated to dryness at room temperature. After extraction, the remaining water and solvent were evaporated to dryness on a steam bath and this additional net weight was added to the total weight of particulate matter.

Analysis Orsat Measurements

Orsat measurements for determination of carbon dioxide, oxygen and carbon monoxide oxygen were made using a Burrell Industrial Gas Analyzer.

Analysis (SO₂ Train)

SO₂ samples were analyzed by the Shell Development method except that barium perchlorate was used instead of barium chloride (as in the EPA proposed source testing Method 7) because of the sharper titration end point obtainable with the former reagent.

APPENDIX F
LABORATORY REPORT

Ready to sent to Calif.

G.F. Filters

Total wt

SAMPLES FOOTE MINERALS STEOBENVILLE

Dish	CR	LOCATION and SAMPLE NO.	Dish + G.F. Filter + Part.	SAMPLE WEIGHT	TIT. ALIQ.	Reading Blank	MG in ALIQ.			Total WT grams
12	2FN-T	25W-1	Dish	30.7352	30.7346	30.7342	30.7352	30.7344	30.7342	0.4453
				30.2889	30.2889	30.2889	30.2889	30.2889	30.2889	0.2900
				0.4463	0.4457	0.4453	0.4463	0.4455	0.4453	0.1553
13	2FN-AG	25W-2	Dish	38.5412	38.5406	38.5400	38.5402	38.5401		0.3283
				38.2118	38.2118	38.2118	38.2118	38.2118		0.1318
				0.3294	0.3288	0.3282	0.3284	0.3283		0.1465
14	2FN-BIK	" "	Dish	24.8564	24.8564	24.8558	24.8557	24.8558		0.4142
				24.4416	24.4416	24.4416	24.4416	24.4416		0.2935
				0.4148	0.4148	0.4142	0.4141	0.4142		0.1207
15	2FN-P	"	Dish	31.1196	31.1193	31.1187	31.1186	31.1187		0.4612
				30.6575	30.6575	30.6575	30.6575	30.6575		0.2941
				0.4621	0.4618	0.4612	0.4611	0.4612		0.1671
16	2FN-A1	"	Dish	29.1948	29.1948	29.1943	29.1940	29.1941		0.2415
				28.9526	28.9526	28.9526	28.9526	28.9526		0.1871
				0.2422	0.2422	0.2417	0.2414	0.2415		0.0544
17	2FNA	25E-1	Dish	26.3041	26.3048	26.3040	26.3040	26.3040		0.4905
				25.8072	25.8072	25.8072	25.8072	25.8072		0.2911
				0.4969	0.4976	0.4968	0.4968	0.4968		0.2057
18	2FNO	"	Dish	27.5707	27.5712	27.5700	27.5700	27.5700		1.1149
				26.4551	26.4551	26.4551	26.4551	26.4551		0.5845
				1.1156	1.1161	1.1149	1.1149	1.1149		0.5303
19	2FNU	25E-2	Dish	29.5515	29.5518	29.5510	29.5515	29.5515		0.5154
				29.0356	29.0356	29.0356	29.0356	29.0356		0.2252
				0.5159	0.5162	0.5154	0.5154	0.5154		
20	2FN-AS	"	Dish	29.7810	29.7817	29.7803	29.7808	29.7808		0.3753
				29.4055	29.4055	29.4051	29.4051	29.4051		0.1793
				0.3755	0.3762	0.3753	0.3753	0.3753		0.1960
21	2FN-W	"	Dish	31.1986	31.1988	31.1982	31.1980	31.1981		0.4680
				30.7301	30.7301	30.7301	30.7301	30.7301		0.2714
				0.4685	0.4687	0.4681	0.4679	0.4680		0.1766
22	2FN-V	"	Dish	28.4426	28.4434	28.4437	28.4438	28.4438		0.5021
				27.9415	27.9415	27.9411	27.9415	27.9415		0.2936
				0.5011	0.5019	0.5022	0.5023	0.5021		0.2085
23	2FN-A4	6E-1	Dish	29.7057	29.7068	29.7057	29.7057	29.7057		0.2980
				29.4077	29.4077	29.4077	29.4077	29.4077		0.1720
				1.2980	1.2991	1.2980	1.2980	1.2980		0.1200
24	2FN-A2	- 2	Dish	29.2026	29.2029	29.2027	29.2026	29.2026		0.3813
				28.8213	28.8213	28.8213	28.8213	28.8213		0.1837
				0.3813	0.3816	0.3814	0.3813	0.3813		0.1976
25	2FN-J	IGNORE:	Dish	29.5751	29.5774	29.5754	29.5754	29.5754		0.3319
				29.2435	29.2435	29.2435	29.2435	29.2435		0.2311
				0.3316	0.3339	0.3319	0.3319	0.3319		0.0424

Project No. 859489

Collection Date 5/17 - 5/21

Analysis Date 6 - 8 - 71

2FN J No 1
2FN J - .0424

F-1

TOTAL WT
WATER IMP
(PHS)

SAMPLES FOOTE MINERALS - STRUBENVILLE, OHIO

NET

CR NO.	LOCATION and SAMPLE NO.	Total Volume	SAMPLE WEIGHT	TIT. ALIQ.	Reading Blank	MG in ALIQ.			Total WT grams
H ₂ O ✓ 2a	25F - 1	300	90.9042 90.8860	90.9036 90.8860	90.9037 90.8860	90.9036 90.8860	90.9037 90.8860	90.9037 90.8860	0.0169
H ₂ O ✓ 2b	25F - 1	300	90.9702 90.9532	90.9708 90.9537	90.9712 90.9537	90.9702 90.9537	90.9708 90.9537	90.9706 90.9537	0.0169
H ₂ O ✓ 2c	25E - 1	125	88.4748 88.4759	88.4748 88.4759	88.4748 88.4759	88.4746 88.4754	88.4748 88.4754	88.4748 88.4754	0.0001
H ₂ O ✓ 2d	25E - 2	125	90.8382 90.8377	90.8378 90.8377	90.8378 90.8377	90.8378 90.8377	90.8378 90.8377	90.8378 90.8377	0.0001
H ₂ O ✓ 6a	25W - 1	460	91.20180 91.12160	91.3014 91.1216	91.3040 91.1216	91.3032 91.1216	91.3034 91.1216	91.3034 91.1216	0.1806
H ₂ O ✓ 8a	25W - 2	300	86.5743 86.5632	86.5750 86.5632	86.5740 86.5632	86.5750 86.5632	86.5752 86.5632	86.5752 86.5632	0.0112
H ₂ O ✓ 7a	25W - 1	125	89.2841 89.2490	89.2849 89.2490	89.2841 89.2490	89.2843 89.2490	89.2844 89.2490	89.2844 89.2490	0.0350
H ₂ O ✓ 7a	25W - 2	125	89.3820 89.3809	89.3822 89.3809	89.3820 89.3809	89.3821 89.3809	89.3821 89.3809	89.3821 89.3809	0.0008
H ₂ O ✓ 6a	#6 EXH - 1	380	89.7632 89.7270	89.7657 89.7270	89.7657 89.7270	89.7632 89.7270	89.7632 89.7270	89.7632 89.7270	0.0396
H ₂ O ✓ 13a	#6 EXH - 2	180	91.3403 91.3310	91.3403 91.3310	91.3403 91.3310	91.3402 91.3310	91.3400 91.3310	91.3400 91.3310	0.0085
H ₂ O ✓ 13a	#6 EXH - 1	125	90.8402 90.8345	90.8596 90.8345	90.8392 90.8345	90.8390 90.8345	90.8390 90.8345	90.8390 90.8345	0.0041
H ₂ O ✓ 13a	#6 EXH - 2	125	89.8600 89.8588	89.8602 89.8588	89.8600 89.8588	89.8698 89.8588	89.8694 89.8588	89.8694 89.8588	0.0002
H ₂ O ✓ 13a	131	300	90.0705 90.0694	90.0700 90.0694	90.0696 90.0694	90.0711 90.0694	90.0708 90.0694	90.0702 90.0694	0.0008
H ₂ O ✓ 13a	131	125	89.8734 89.5172	89.8777 89.5172	89.8737 89.5172	89.8739 89.5172	89.8736 89.5172	89.8736 89.5172	0.0004

Project No. 259489

Collection Date _____

Analysis Date 6-18-71

TOTAL WT
ACETONE AFT:

SAMPLES FOOTE MINERALS - STEUBENVILLE, OHIO

Project No. 559 489

Collection Date _____

Analysis Date 7-9-71

TOTAL WT
ACETONE BEFORE

SAMPLES FOOTE MINERALS - STEUBENVILLE, OHIO

CR NO.	LOCATION and SAMPLE NO.	SAMPLE WEIGHT g. / ml. x p	TIT. ALIQ.	Reading Blank	MG in ALIQ.			Total wt grams	N.T.
31	25 E - 1	87.5433 87.3142 0.2291	87.5434 87.3142 0.2292	87.5433 87.3142 0.2291					- .0002
32	- 2	88.4997 88.3200 0.1791	88.4997 88.3200 0.1791	88.4997 88.3200 0.1791				0.2291	- .0003
33	25 W - 1	87.8803 87.7312 0.1491	87.8807 87.7312 0.1491	87.8803 87.7312 0.1491					- .0002
34	- 2	88.6180 88.5109 0.1071	88.6180 88.5109 0.1071	88.6178 88.5109 0.1069	88.6177 88.5109 0.1068	88.6185 88.5109 0.1078	88.6185 88.5109 0.1076	0.1491	- .0078
35	# 6 Ex H - 1	91.7570 91.1802 0.5768	91.7570 91.1802 0.5775	91.7570 91.1802 0.5775	91.7570 91.1802 0.5776	91.7570 91.1802 0.5776	91.7570 91.1802 0.5776	0.5776	- .0002
36	# 6 Ex H - 2	92.9532 92.2884 0.6642	92.9532 92.2884 0.6648	92.9532 92.2884 0.6649	92.9532 92.2884 0.6649	92.9532 92.2884 0.6649	92.9532 92.2884 0.6649	0.6648	0.6648

Project No. 859 (59)

Collection Date _____

Analysis Date 7-9-71

Barium Perchlorate

Sa2

SAMPLES

FOOTE MINERALS

STEUBENVILLE, OHIO

Project No. _____

Collection Date

Analysis Date

Date June 10, 1971
te F-5

1 ml = .811 mg SO_2

NET N.T. GAIN
SILICA GEL

SAMPLES FOOTE MINERALS — STEUBENVILLE, OHIO.

Project No. 859489

Collection Date 5/17/71 - 5/21/71

Analysis Date 6/3/71

Al. Thimbles

ASME

SAMPLES FOOTE MINERALS - STEUBENVILLE, OHIO

Project No. 859489

Collection Date

Analysis Date 6-7-71

TOTAL WT.
ASME ~~HO~~ IN
PAGE 11 OF 11
CHIC

SAMPLES

FOOTE MINERALS — STEUBENVILLE, OHIO

Project No. 859489

Collection Date 5-19-71

Analysis Date 7-9-71

APPENDIX G
TEST LOGS

APPENDIX G
TEST LOGS

<u>Date</u>	<u>Samples Performed</u>
5/17/71	Arrive. Equipment unpacked and set up ready at 25 East and West.
5/18/71	First complete particulate samples (OAP & ASME) at locations 25 E & W. Orsat and two SO ₂ .
5/19/71	Second test (OAP & ASME) at each location; 25 E and W. Two Orsats and one SO ₂ .
5/20/71	Two complete tests (OAP & ASME) at No. 6 stack. Two Orsats and 2 SO ₂ . Some members of crew return home.
5/21/71	Rest of the crew packed up equipment and returned home in afternoon

Following are the furnace operating conditions at the time of the atmospheric emissions studies conducted at the Foote Mineral Company Steubenville Plant from May 18 to May 20, 1971.

Furnace No. 25

First Test - May 18, 1971

Furnace Product - L. C. FeCr Si (36-40)

Test Time - 2:38 P.M. to 5:30 P.M.

Furnace Mix - Quartzites, Chrome Ores, Carbon Reducing Agents, and Flux

Furnace Delays - None

Furnace Tapped - 3:03 P.M. to 3:18 P.M.; 5:02 P.M. to 5:22 P.M.

Remarks: This test was conducted during a period when the furnace was operating under normal conditions.

Furnace No. 25

Second Test - May 19, 1971

Furnace Product - L. C. FeCr Si (36-40)

Test Time - 9:59 A.M. to 11:43 A.M.

Furnace Mix - Quartzites, Chrome Ores, Carbon Reducing Agents, and Flux

Furnace Delays - None

Furnace Tapped - 11:09 A.M. to 11:27 A.M.

Remarks: This test was conducted during a period when the furnace was operating at abnormal conditions. The furnace is normally stoked with two stoking machines - one each for the west and east sides of the furnace. Before the test started (9:45 A.M.), the stoking machine which services the east side of the furnace had to be removed from service due to mechanical failure. As a result of this failure, only the west side of the furnace was stoked; the east side developed heat "blows" and tended to run at a much higher temperature than normal. This condition existed until 10:45 A.M.

Furnace No. 6

First Test - May 20, 1971

Furnace Product - Ore Lime Melt

Test Time - 11:30 A.M. to 1:43 P.M.

Furnace Mix - Chrome Ores and Lime

Furnace Delays:

12:22 P.M. to 12:38 P.M. - Both Nos. 5 & 6 Fces. Down - Clean Spout

12:45 P.M. to 12:55 P.M. - Both Nos. 5 & 6 Fces. Down - Tap No. 5

1:32 P.M. to 1:40 P.M. - No. 6 Only Down - Tap

Remarks: This test was conducted during a period when the furnace was operating under normal conditions.

Furnace No. 6

Second Test - May 20, 1971

Furnace Product - Ore Lime Melt

Test Time - 3:38 P.M. to 5:45 P.M.

Furnace Mix - Chrome Ores and Lime

Furnace Delays:

5:00 P.M. to 5:07 P.M. - No. 6 Fce. Only Down - Tap

Remarks: This test was conducted during a period when the furnace was operating under normal conditions.

APPENDIX H
RELATED REPORTS

NOTE: Not applicable - This is the first report in a series.

APPENDIX I
PROJECT PARTICIPANTS AND TITLES

R. N. Allen, P.E., Project Leader
N. A. Blessing, Chemist
C. C. Gonzalez, Chemist
W. E. Schroeder, Chemist
L. W. Baxley, Technician
B. U. Kwon, M.S., (particle size determination)

APPENDIX J
CALCULATION OF EXCAPING
FUMES AND DUST

APPENDIX J
CALCULATION OF ESCAPING FUMES AND DUST
(Furnace No. 6)

It was estimated that an area approximately 10 feet by 10 feet, in cross-sectional dimensions, represented the fume concentrations as measured by a high volume sampler. Visual estimates of the dust flow indicated six feet per second passed this cross-sectional area. The total gas flow would, therefore, be 36,000 cubic feet per minute. This figure was approximately half way between two other estimates by independent technical observers.

During the sampling of Furnace 6 for particulate matter, the test operator was asked to keep a record of the emissions visually observed escaping. Each time he recorded data, it was noted whether these concentrations were high, medium, or low. While testing the exhaust stack for particulate emissions, a high volume sampler was employed to determine actual concentrations during relatively low and high periods of positive emission. Two high concentration periods were measured at almost identical rates of 65 pounds per hour. Two low concentrations periods were measured with rates of 9 and 20 pounds per hour of emission. High emission rates were considered, therefore, to be 65 pounds per hour; medium rates were 40 pounds per hour; and low rates were assumed to be 15 pounds per hour. The percentage at each concentration was tabulated and calculated to determine the average total flow for one hour test period. The data and results are recorded in the following tables.

CALCULATION OF SAMPLE EMISSIONS

<u>Test</u>	<u>Spl. Wt. grains</u>	<u>Volume Spl. cu ft</u>	<u>grains/cu ft</u>	<u>Lbs/hr</u>
1	8.95	300	.0298	9.20
2	34.95	164	.213	65.7
3	8.52	129	.0659	20.3
4	28.03	135	.208	64.2

HIGH VOLUME SAMPLES

<u>Sample No.</u>	<u>Time</u>	<u>Minutes Run</u>	<u>Rate, cfm</u>	<u>Total cu ft</u>	<u>Gross Wt, gm</u>
			<u>Start</u>	<u>End</u>	<u>Tare Wt, gm</u>
1	1415	6	55	300	4.0719
					3.4917
					0.5802
2	1544	4	55	164	5.7763
					3.5110
					2.2653
3	1611	3	55	129	4.0628
					3.5107
					0.5521
4	1735	3	55	135	5.3217
					3.5049
					1.8168

CALCULATION OF FLUE GAS VOLUME

$$\text{Area} = 10' \times 10' = 100 \text{ ft}^2$$

Velocity = 6' per second

$$\text{Volume} = 6 \text{ ft/sec} \times 60 \text{ sec/min} \times 100 \text{ ft}^2 = 36,000 \text{ cfm}$$

Date = 5/20/71

OBSERVATION PERIODS

<u>During Run</u>	<u>High Concentration @ 65#/hr</u>	<u>Medium Concentration @ 40#/hr</u>	<u>Low Concentration @ 15#/hr</u>
1	11 = 58%	3 = 16%	5 = 26%
2	16 = 70%	7 = 30%	--

CALCULATIONS, 1-hour Basis - Escaping Dust

Run 1: 37.7 #/hr High
 6.4 " Medium
 3.9 " Low

 48.0 " Total
 54.8 " Stack Emission

 102.8 " Overall

Run 2: 45.5 #/hr High
 12.0 " Medium

 57.5 #/hr Total
 62.2 " Stack Emission

 119.7 " Overall

Estimate of Percent Fugitive
 Dust Emission
 Therefore - 53%

- 52%

APPENDIX K

PARTICLE SIZING SAMPLES

PARTICLE SIZE DISTRIBUTION OF METAL FUME

INTRODUCTION

Determinations of particle size distribution of fume emissions at the Ferroalloy Plant of the Foote Mineral Company, Steubenville, Ohio, were conducted from May 18 through May 20. Emissions were evaluated at the east and west exhaust stacks of Furnace 25, and at the exhaust stack of Furnace 6. There were no emission control devices on either furnace.

METHODS

Samples for the evaluation of particle size distribution were gathered using both the Andersen Stack Sampler and the Brink Cascade Impactor. The first nine samples were collected with the Anderson unit. This had been the EPA recommended procedure because larger particles were expected when there were no emission control devices after the furnace. However, the majority of particles deposited on the filter, as shown in Table 1 on the following page. This data could not be used to determine mass median diameters without resorting to extrapolations. The Andersen sampler was therefore discarded in favor of the Brink impactor, which was utilized in gathering the three subsequent samples.

The Andersen sampler and Brink sampler, both with attached 47 millimeter glass fiber filters, were mounted on probes and connected to vacuum pumps by rubber tubing. Metering valves were installed on the inlet side of the pumps to adjust the air flow through the samplers. Magnehelic gauges were inserted in the system to measure pressure drops (ΔP) across the samples. Figure 1 illustrates a typical particle sizing train.

Results

Table 1

Anderson Stack Sampler Data
Furnace No. 25
Sample Rate = 0.5 cfm

<u>Stage No.</u>	<u>Dpc</u> μ	Sample No. (Percent Collection)					
		1	2	3	4	8	9
0	-	-	-	-	-	2.9	5.5
1	20.00	-	-	-	-	0.8	4.2
2	12.50	-	0.9	-	1.9	-	3.5
3	8.50	2.9	2.6	2.8	3.6	0.4	0.0
4	5.60	3.4	4.4	3.4	3.6	0.8	-
5	3.72	4.2	8.8	4.1	5.0	2.9	2.1
6	1.80	4.5	7.9	2.8	5.0	4.9	8.4
7	1.16	14.4	9.7	6.2	5.0	7.4	5.6
8	0.78	5.3	10.5	10.4	7.9	8.6	7.7
Filter	0.1	65.3	55.2	70.3	68.0	71.3	63.0

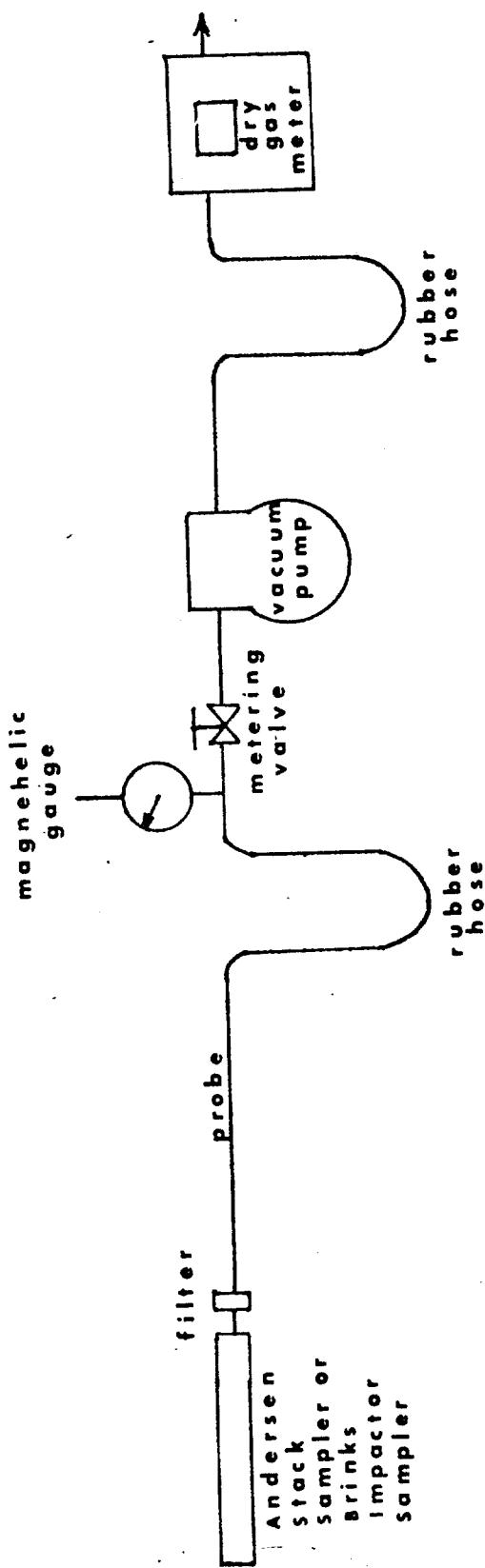


FIGURE 1. PARTICLE SIZING TRAIN

Prior to collecting samples in the field, each impactor was calibrated to determine air flow rates at various operating conditions. Rates were determined by assembling the particle sizing train as shown in Figure 1. Air was drawn through the sampler for 10 minutes at each pressure drop (ΔP) of two inches of Hg, five inches of Hg, and 10 inches of Hg. The corresponding volume of air flow for each sample was measured by the dry gas meter. A calibration curve was constructed by plotting the pressure drop across the sampler versus the air flow rate.

As high temperatures were encountered, the plugged impactor was inserted into the duct for at least five minutes, to allow time for thermal equilibrium. The impactor was then removed from the duct, the plug removed, and reinserted into the duct for sample collection. The sampling durations ranged from two minutes to 10 minutes with an air flow of 0.5 cfm through the samplers. The impactor was grounded, either to the stack or building, to prevent electrostatic deposition of particles.

RESULTS

Graphical results are shown in Sub-appendix 1 and the field data sheets are included in Sub-appendix 2. The characteristic diameter of an aerosol particle for each Brink impactor stage (i.e., D_{pc}) has been calculated for an air flow rate of 0.5 cfm through the impactor, assuming particles of unit density (1 gram/cubic centimeter), using the equation described by * J. A. Brink, Jr. The characteristic diameters are as follows:

<u>Stage No.</u>	<u>D_{pc}, microns</u>
1	3.40
2	2.00
3	1.36
4	0.69
5	0.42

* Industrial Engineering and Chemistry, Vol. 50, April 1958, pp 645-648.

Graphical presentation of the data, that is, log-probability plots of cumulative percent less than stated micron size versus the Dpc for each stage in microns, is shown for the Brink impactor samples and one typical Andersen impactor sample in the following section. A graphically determined mass median diameter (MMD) and Geometric Standard deviation (σ_g) for each sample is presented in the following Table 2.

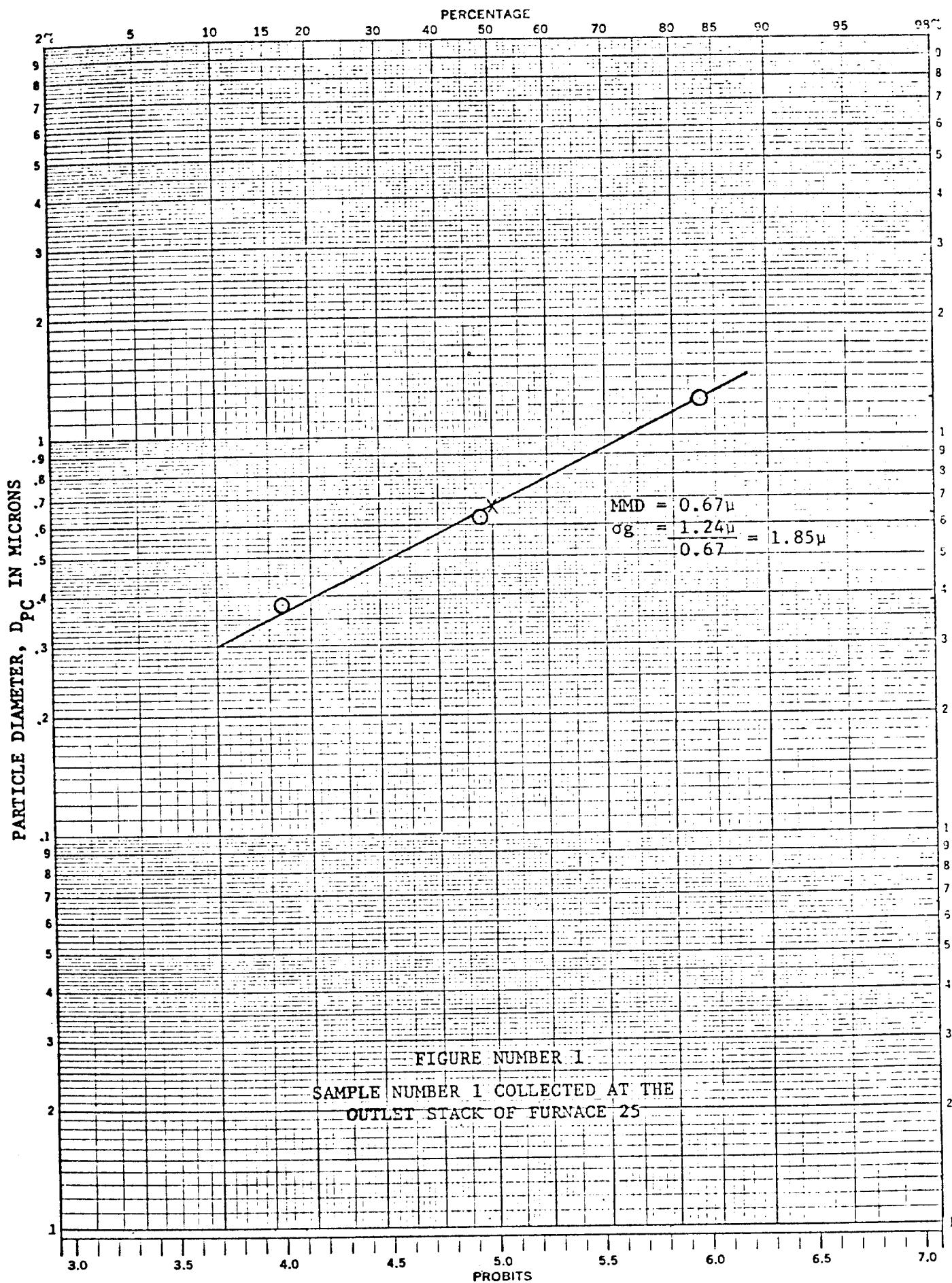
TABLE 2

<u>Date</u>	<u>Sample No.</u>	<u>Location of Sample</u>	<u>Duration of Sampling</u> (Min.)	<u>MMD</u> (microns)	<u>σ_g</u> (microns)
5-18-71	1	Outlet Stack Furnace 25	5	0.67	1.85
5-19-71	2	Outlet Stack Furnace 25	5	0.68	1.84
5-19-71	3	Outlet Stack Furnace 25	5	0.62	2.00

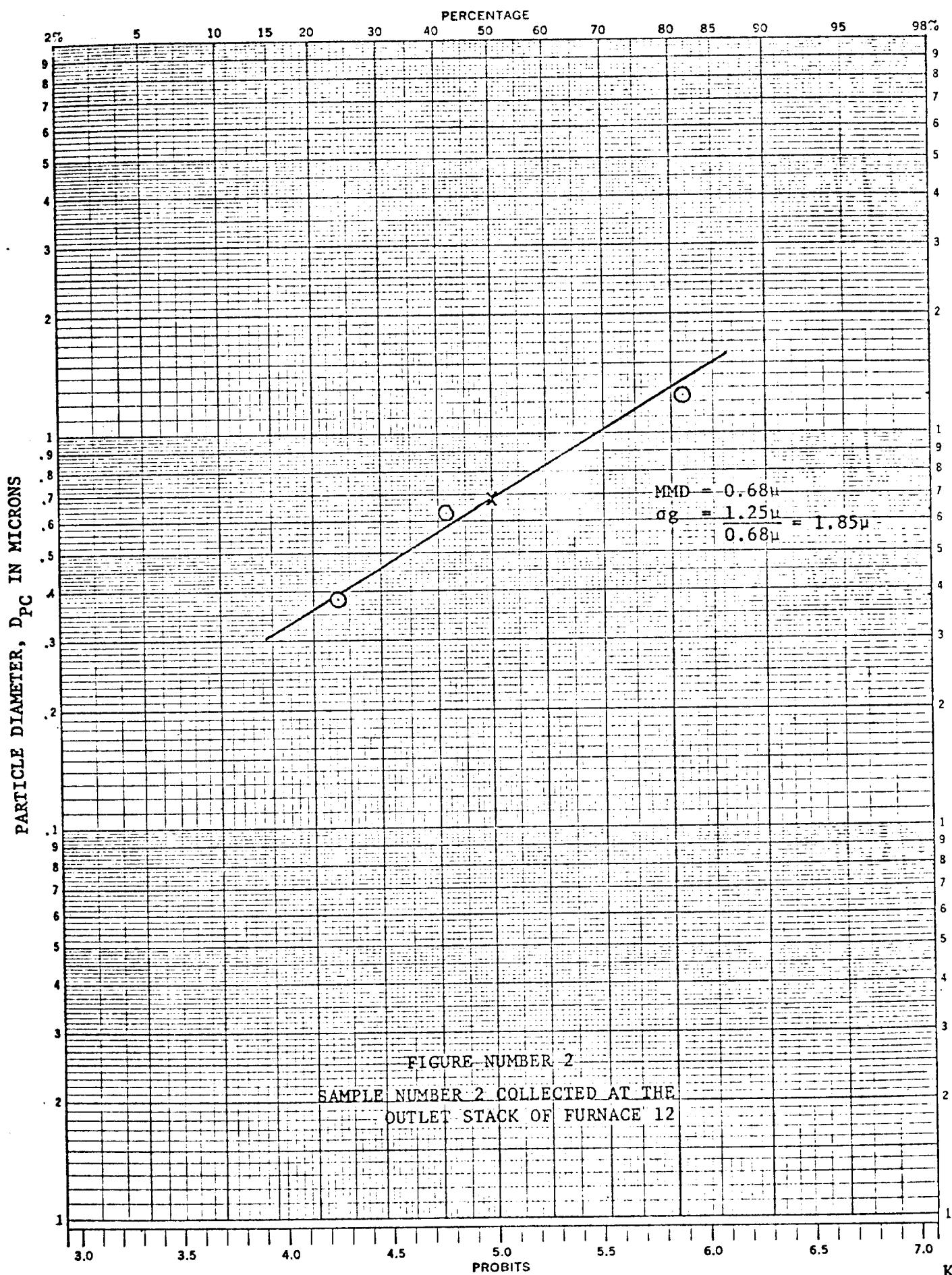
SUB-APPENDIX K-1

GRAPHICAL PRESENTATION OF RESULTS

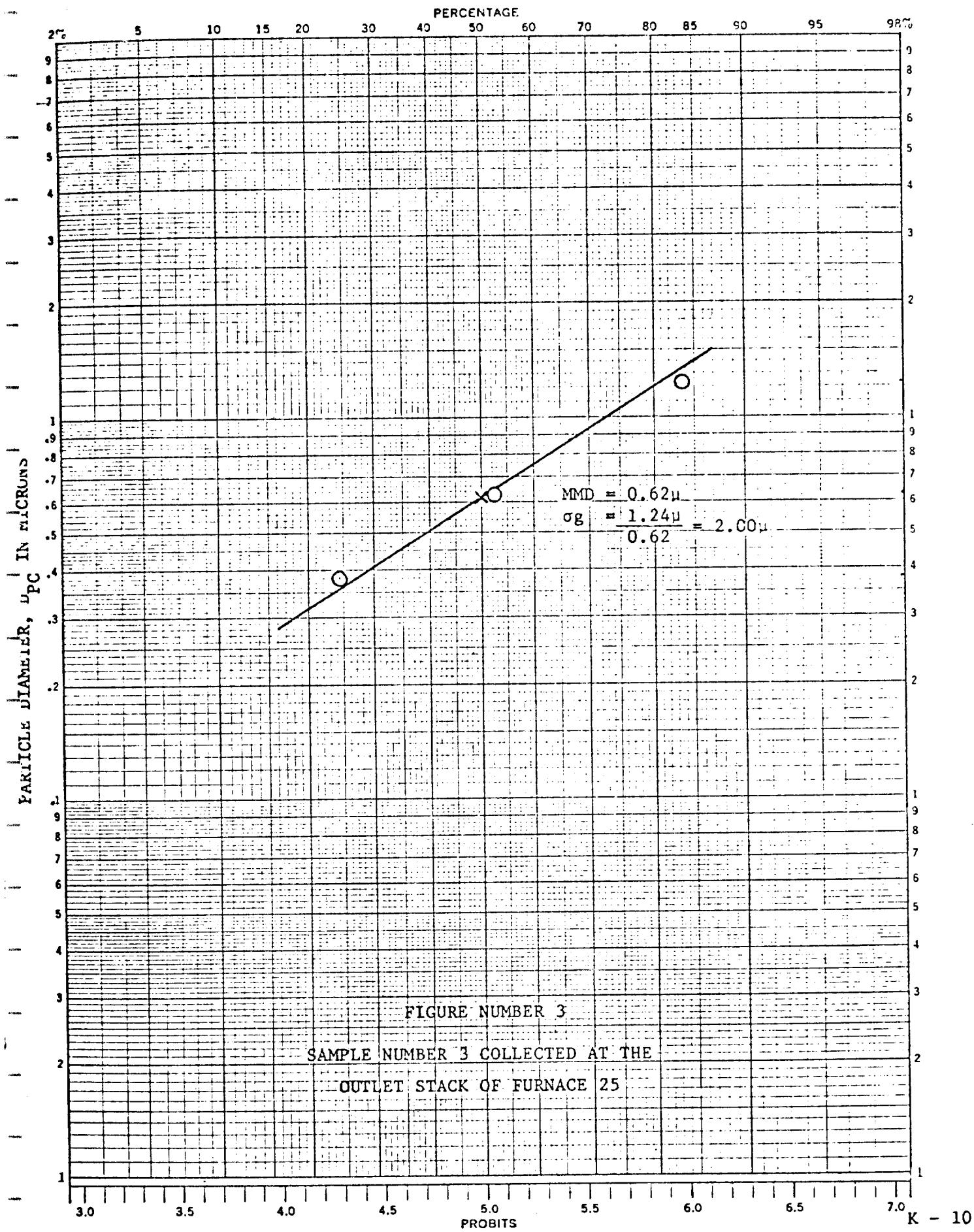
CUMULATIVE PERCENT LESS THAN STATED MICRON SIZE



CUMULATIVE PERCENT LESS THAN STATED MICRON SIZE



CUMULATIVE PERCENT LESS THAN STATED MICRON SIZE



THIS PAGE DELIBERATELY BLANK

SUB-APPENDIX K-2

FIELD DATA



Anderson
data sheets 20

Stack No. 26-Far

Sample No. #1

220°F

Date 5-18

STAGE	POST. WT.	PRE. WT.	WT. GAIN	%	CUM. % LESS THAN Dpc
0	29.7193	29.7184			
1	20.2763	20.2755			
2	21.3394	21.2272			
3	21.7500	21.7493	0.0007	2.9	
4	21.6515	21.6506	0.0009	3.4	
5	12.8373	12.8362	0.0011	4.2	
6	12.0400	12.0392	0.0012	4.5	
7	12.5400	12.5382	0.0032	14.4	
8	21.2844	21.2730	0.0014	5.3	
Filter	0.2328	0.2255	0.0173	65.3	
					.0264

heated the head for over 5 mins before beginning air filter

ten min run at 0.5 cfm; placed in stack 1.5 ft. 1 ft.

appears to be blown off mesh on underside of 7th stage probably from large ^{debris} piles on the 8th Stage; 6th stage underside has small 1/4 in. dia. deposition ^{debris} in orifice holes; guess is that orifices have deposited ^{debris} and ^{debris} by the 7th stage, can see deposition on the top ^{stage} surface around orifice.

majority of mesh collecting on final filter

Stack No. #25-Far

260° F

Date 5-18-19

Sample No. #2

2"

STAGE	POST. WT.	PRE. WT.	WT. GAIN	%	CUM. % LESS THAN Dpc
0		30.2184			
1		20.1049			
2	0.0001	21.3510	21.3511	0.9	
3	0.0003	21.8287	21.8290	2.6	
4	0.0005	22.3194	22.3199	4.4	
5	0.0010	12.6150	12.6160	8.8	
6	0.0009	12.2841	12.2850	7.9	
7	0.0011	13.1838	13.1849	9.7	
8	0.0012	20.8885	20.8897	10.5	
Filter	0.0063	0.2263	0.2326	55.2	
	0.0114				

0.5 cfm for 2 min

2600° F.

2"

Stack No. 25-For

Date 5-12-19

Sample No. #3

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u>	<u>%</u>	<u>CUM. %</u> <u>LESS THAN Dpc</u>
0	<u>30.535</u>				
1	<u>20.4750</u>				
2	<u>21.2356</u>				
3	<u>21.8816</u>	<u>21.8812</u>	<u>0.0004</u>	<u>2.8</u>	
4	<u>21.9624</u>	<u>21.9619</u>	<u>0.0005</u>	<u>3.4</u>	
5	<u>12.2019</u>	<u>12.2013</u>	<u>0.0006</u>	<u>4.1</u>	
6	<u>12.2249</u>	<u>12.2245</u>	<u>0.0004</u>	<u>2.8</u>	
7	<u>11.8049</u>	<u>11.8070</u>	<u>0.0009</u>	<u>6.2</u>	
8	<u>20.6139</u> ^{DM}	<u>20.6164</u>	<u>0.0015</u>	<u>10.4</u>	
Filter	<u>0.2371</u>	<u>0.2269</u>	<u>0.0102</u>	<u>70.3</u>	
			<u>0.0145</u>		

0.5 cfm for 2 min

260° f1

Stack No. #26-Fox

2"

Date 5-2-19

Sample No. #4

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u>	<u>%</u>	<u>CUM. %</u> <u>LESS THAN Dpc</u>
0	30.1127				
1	20.2809				
2	21.1133	21.1130	0.0003		1.9
3	21.5148	21.5143	0.0005	3/3	3.6
4	22.0159	22.0154	0.0005	3/3	3.6
5	12.2162	12.2155	0.0007	4/7	5.0
6	12.4628	12.4621	0.0007	4/7	5.0
7	12.9537	12.9530	0.0007	4/7	5.0
8	20.7511	20.7500	0.0011	7.3	7.9
Filter	0.2401	0.2306	0.0095	63/3	68.0
				0.01570	

0.5 cfm, 2 min

Stack No. #5

Sample No. 5

Date 5-10

3 min sample
at 0.5 gfm

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u> kg	<u>%</u>	<u>CUM. %</u> <u>LESS THAN D₅₀</u>
0	30.1881	30.1876	0.5		
1	20.1249	20.1245	0.4		
2	21.2382	21.2382	0.0		
3	21.7310	21.7306	0.4		
4	22.4317	22.4314	0.3		
5	11.7534	11.7526	0.8		
6	11.8910	11.8906	0.4		
7	11.5054	11.5089	0.5		
8	21.0490	21.0480	1.0		
Filter	0.2274	0.2243	3.1		

Stack No. 25-for
Sample No. #8

Date 5-19

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u> mg	<u>%</u>	<u>CUM. %</u> LESS THEN Dpc
0	30.2510	30.2503	0.7	2.8	2.88
1	20.3471	20.3469	0.2	0.8	0.89
2	21.0604	21.0607	-(0.3)		
3	21.3246	21.3245	0.1	0.4	0.45
4	22.2783	22.2781	0.2	0.8	0.89
5	11.6131	11.6124	0.7	2.8	2.89
6	11.8996	11.8984	1.2	4.9	4.9
7	11.4904	11.4886	1.8	7.4	7.4
8	21.0371	21.0350	2.1	8.6	8.6
Filter	0.2417	0.2243	17.4	71.3	71.0

5 min run at 0.5 cfm
24.4
84.4

Stack No. #25 for

Date 5-19

Sample No. #9

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u> <small>mg</small>	<u>%</u>	<u>CUM. %</u> <u>LESS THAN Dpc</u>
0	29.9100	29.9092	0.8		5.5
1	20.2560	20.2554	0.6		4.2
2	21.2515	21.2510	0.5		3.5
3	21.7240	21.7240	-		
4	22.2703	22.2708	-0.5		
5	11.8003	11.8005	0.3		2.1
6	11.7435	11.7423	1.2		8.4
7	12.3854	12.3846	0.8		5.6
8	21.1606	21.1595	1.1		7.7
Filter	0.2399	0.2309	9.0		63.0

for 2.5 min at 0.5 cfm
(4.3)

Stack No. 26-Far

Sample No. 1

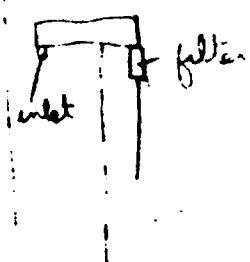
Brinks
Beta sheets

Date 5-18

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u>	<u>%</u>	<u>CUM. %</u> <u>LESS THEN Dpc</u>
0					
1					
2				Mg	
3	3.2336	3.2320	1.6	μ	1.23 16.7 83.3
4	3.4784	3.4750	3.4	μ	0.63 35.3 48.0
5	3.2521	3.2491	3.0	μ	0.38 31.3 16.7
6					
7					
8					
Filter	0.0424	0.0404	1.6	μ	0.10 16.7

sample for 10 min at 40/min

preheat sampler for over 5 min



MMD = .74

Stack No. 15

Sample No. 2

250°F

Date 5-9-71

Bromine

5 min sample at 4 min

CUM. %

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u>	<u>%</u>	<u>CUM. %</u> <u>LESS THAN Dpc</u>
0					
1					
2					
3	<u>3.2329</u>	<u>3.2318</u>	<u>1.1</u>	<u>18.4</u>	<u>81.6</u>
4	<u>3.4775</u>	<u>3.4751</u>	<u>2.4</u>	<u>40.0</u>	<u>41.6</u>
5	<u>3.2499</u>	<u>3.2488</u>	<u>1.1</u>	<u>18.4</u>	<u>23.2</u>
6					
7					
8					
Filter	<u>0.0414</u>	<u>0.0401</u>	<u>1.4</u>	<u>23.2</u>	
			<u>6.0</u>		

Sampled at 1:25 p.m.

Stack No. 25

Sample No. 3

B. mites
Sampled for 5 min.
at 46/min

Date 5-19-71

<u>STAGE</u>	<u>POST. WT.</u>	<u>PRE. WT.</u>	<u>WT. GAIN</u>	<u>%</u>	<u>CUM. % LESS THAN Dpc</u>
0					
1					
2					
3	3.2322	3.2312	0.0010	16.4	13.6
4	3.4762	3.4743	0.0019	31.1	52.5
5	3.2500	3.2483	0.0017	28.0	24.5
6					
7					
8					
Filter	0.0860	0.0395	- 0.0015	24.5	

6.1 mg

Sampled at 2:05 p.m.

APPENDIX L
CHEMICAL ANALYSIS OF EMISSIONS

PREFACE

The following report, covering chemical analysis of emissions from reactive metal smelting operations at Steubenville, Ohio, has been prepared by the technical staff of TRW Systems Group, One Space Park, Redondo Beach, California.

Principal Contributors: D. F. Carroll
M. L. Kraft
W. B. Hewitt

Approved By: J. R. Ogren

CHEMICAL ANALYSIS OF EMISSIONS
FROM
REACTIVE METAL SMELTING OPERATIONS

1. INTRODUCTION

Particulate fumes and gaseous emissions are generated during the smelting and pouring of a commercially important class of ferro alloy materials called reactive metals. The particulate portion of these emissions has been collected on glass fiber filters, strategically placed in the air stream of an exhaust system. Fourteen such filters from the Foote Mineral Corporation (Steubenville, Ohio) were analyzed microscopically, by X-ray diffraction, atomic absorption, electron beam X-ray microanalysis, and optical-emission spectroscopy.

The analytical results are presented in the following sections where it is shown that the particulate specimens from the two furnaces are distinctly different from the standpoints of chemical composition and crystallographic structure. The samples from Furnace #25 consist principally of non-crystalline fused silica (SiO_2) with impurities. Impurities present in concentrations > 1 weight per cent are Mg, Cr, and Zn in decreasing order. In contrast, the samples from Furnace #6 contain crystalline material with the inverse spinel structure such as typified by Fe_3O_4 . Instead of consisting mostly of SiO_2 as seen for the Furnace #25 specimens, the specimens from Furnace #6 consist of chromium, silicon, magnesium, iron, and zinc all in the 4 to 18 weight per cent range along with chemically combined oxygen. This is, the specimens from Furnace #6 consist of metal oxides.

2. TEST RESULTS

2.1 Optical Examination

The specimens were examined at magnifications up to 100X. Figure 1 shows Specimen 25W-1 and shows the manner in which all specimens were divided for individual analysis. The specimens from Furnace #6 were yellow-brown and distinctly different from the gray-colored Furnace #25 specimens.

2.2 X-Ray Diffraction Analysis

X-ray diffraction occurs when a crystalline substance is exposed to a beam of X-rays. The angle between the diffracted beam and the incident beam is always 2θ , or twice the angle of incidence. By using monochromatic X-rays of wavelength λ , the interplanar spacing d , of various planes in a crystal can be found by using Bragg's Law, $\lambda = 2d \sin \theta$. An electronic detector or photographic film is used to record θ angles and the intensities of the diffracted beams. Every crystalline substance has an unique X-ray pattern comprised of many θ angles (usually converted to d -spacings) and associated intensity values. Over 22,000 X-ray diffraction patterns have been published to date.

The diffraction samples were prepared by removing the powders from the individual filters, thoroughly mixing each powder manually in a plastic container with a wooden tongue depressor, and pressing into 1/2-inch diameter pellets under 80,000 psi. This method of specimen removal from the quartz (SiO_2) filter in no way disturbed the filter. No filter particles mixed with the specimens removed. In fact, a small quantity of powder remained on the filter after removal of the specimens. These pellets were analyzed on a G.E. XRD-5 X-ray unit. The instrumental settings used are listed in Table 1.

The diffraction patterns from all samples were weak, therefore, a chromium tube was used as a source of X-rays in order to reduce background radiation due to X-ray fluorescence. The use of the chromium X-ray tube and pulse height analysis maximized the signal/noise ratio.

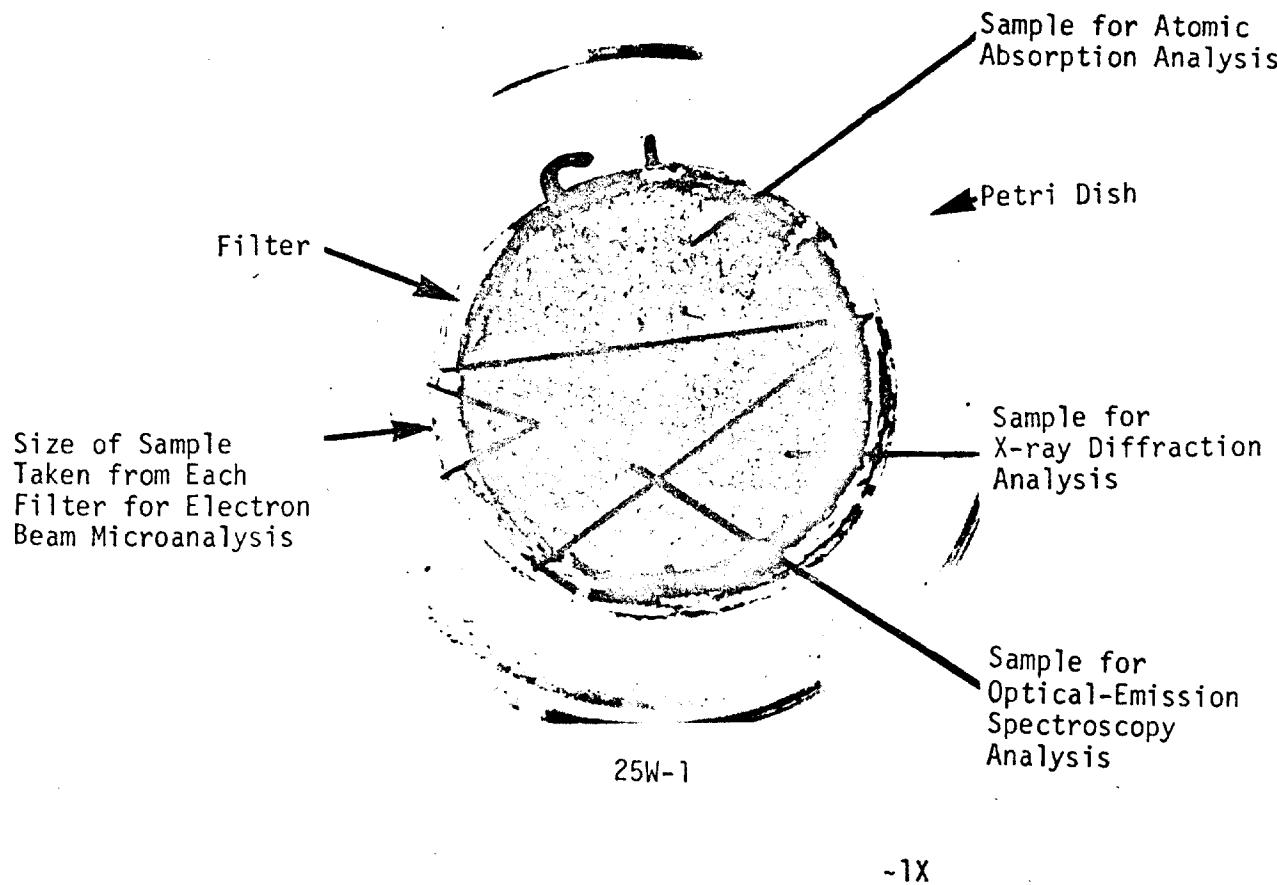


Figure 1. Photograph of condensate from ferrochrome operation showing areas analyzed.

TABLE 1.
X-RAY DIFFRACTOMETER SETTINGS

X-ray Source: Chromium Tube; 50KVP, 20 ma, no filter

Beam Slit: 1°

Soller Slit: Medium Resolution

Exit Slit: 0.1°

Table Speed: $2^{\circ}/\text{Min}$; Chart Speed $2''/\text{min.}$

Detector: Flow proportional

Scale: Linear 100

Pulse Height Selector: $E_1 = 2V$ with Gain $\times 16.$
 $\Delta E = 6V$

The diffraction results are summarized in Figure 2 which shows the sample identification, and the d-spacings and relative intensities of the diffracted beams. These patterns were then compared with tables of known diffraction patterns. *

The X-ray results can be summarized as follows:

1. All specimens were largely non-crystalline as evidenced by an absence of a diffraction pattern or very weak diffuse patterns with very few lines. No X-ray diffraction patterns were obtained from Specimens 25W-1, 25W-2, 25E-1, and 25E-2; they were completely non-crystalline. **
2. Another eight samples had weak patterns, but the patterns could not be correlated in a meaningful way with any known pattern from the diffraction file. In a few instances, a force-fit might have been possible but the choices were hydrated crystals such as $\text{Ca}_3\text{Al}_8(\text{PO}_4)_8(\text{OH})_6 \cdot 15\text{H}_2\text{O}$. It seemed unlikely that a highly hydrated and complex crystal would have formed during the few microseconds available for emissions to condense from the gaseous high temperature effluent. These eight patterns were, therefore, classified as unknown.
3. Recognizable patterns were obtained from both specimens from Furnace #6. The patterns belong to the naturally occurring class of compounds called spinels. *** It was not possible to positively tell which particular spinel oxide was present but the best fit to the X-ray data include:

* Joint Committee on Powder Diffraction Standards, Powder Diffraction File, Swarthmore, Pennsylvania, 1969.

** The specimen numbers designate the Furnace, #25 or #6, and duct, west or east, from which specimens, 1 or 2, were taken simultaneously. For example, specimen 25W-1 is Sample #1 taken from the west (W) duct of Furnace #25. Specimen 25g-1 was taken from the east duct of Furnace #25 at the same time specimen 25W-1 was taken.

*** The spinel group includes a large number of oxides of the general formula AB_2O_4 . The more familiar members of the spinel group are MgAl_2O_4 , ZnFe_2O_4 , CdFe_2O_4 , FeAl_2O_4 , CoAl_2O_4 , NiAl_2O_4 , MnAl_2O_4 , and ZnAl_2O_4 . Inverse spinels have the same X-ray pattern, are more common in nature, and include $\text{FeO} \cdot \text{Fe}_2\text{O}_3$ also written as Fe_3O_4 .

4742.3.71-139
23 September 1971
Page 7

SPECIMEN

25W-1 8
25W-2
25W-2
25W-2
25W-2
25W-2
25E-1 4
25E-1
25E-1
25E-1
25E-2
25E-2
25E-2
25E-2
6E-1
6E-2

*NO PATTERN DETECTABLE

POWDER DIFFRACTION FILE DATA

	19-629	3-0873	1-1110	4.5	4.0	3.5	3.0	2.5	2.0	1.5
Fe_3O_4										
$FeO(FeAl_{1-x}Al_x)_2O_3$ (CHROMITE)										
$NiMn_2O_4$										

Figure 2. Summary of X-Ray Diffraction Data
CRYSTALLOGRAPHIC d-SPACING - ANGSTROM UNITS

4742.3.71-139
23 September 1971
Page 8

Chromite: $\text{FeO} \cdot [\text{Cr}_x \text{Al}_{1-x}]_2 \text{O}_3$ $0.64 < x < 1$

and

Magnetite: $\text{FeO} \cdot \text{Fe}_2\text{O}_3$

4. The eight unidentified weak patterns were scrutinized to see if any possibility exists that the compounds reported previously might be present. No evidence was found to indicate the presence of CaO , SiC , FeSi , FeSi_2 , MnO , or any of the numerous crystallographic allotropes of either SiC or SiO_2 (quartz).

2.3 Atomic Absorption Analysis

Atomic Absorption (A.A.) means that a cloud of atoms in the un-ionized and unexcited state is capable of absorbing radiation at wavelengths that are specific in nature and characteristic of the element in consideration. The atomic absorption spectrophotometer used in these analyses consists of a series of lamps which emit the spectra of the elements determined, a gas burner to produce an atomic vapor of the sample, a monochromator to isolate the wavelengths of interest, a detector to monitor the change of absorption due to the specimen, and a readout meter to visualize this change in absorption.

The sample pellet previously used for the X-ray diffraction analysis was carefully pulverized, weighed into a tared 150 ml beaker, and extracted for two hours with hot aqua regia (4 ml HCl + 16 ml HNO_3).* The resulting solution was then filtered and the residue washed several times with cold water. The filtrate from the above extraction was concentrated on a low heat hot plate, transferred to a 25 ml volumetric flask, and the elemental concentrations determined by Atomic Absorption Spectroscopy (A.A.). The residues from the above extractions were ignited in a muffle furnace at 900°C and fused with 0.4 g Na_2CO_3 . The resulting fusion product was dissolved in water, made acid with H_2SO_4 , and taken to dryness on a high heat hot plate. The resulting residue was taken up to 100 ml of dilute acid (1 ml H_2SO_4 in 100 ml H_2O) and filtered. The SiO_2 content was determined gravimetrically by igniting and weighing the filtered solids residue.** The filtrate was concentrated on a low heat hot plate, transferred to a 100 ml volumetric flask, and the elemental concentration determined by A.A.

* R. J. Thompson, G. B. Morgan and L. J. Purdue, "Analysis of Selected Elements in Atmospheric Particulate Matter by Atomic Absorption," Atomic Absorption NEWS Letter, Vol. 9, No. 3, 1970.

** N. H. Furman Ed., Standard Methods of Chemical Analysis, 6th Edition, Vol. 1, D. Van Nostrand Co., Inc., Princeton, New Jersey, pp. 950-957, 1962.

The individual elemental concentrations obtained from the aqua regia leach and the filtrate from the Na_2CO_3 fusion were then summed for each sample; these results are compiled in Table 2. Samples 6E-1 and 6E-2 were found to be insoluble in the Na_2CO_3 flux and as a consequence of this, it was not possible initially to obtain SiO_2 or further elemental analysis on these two samples using the above method. These two samples were found to be soluble in a potassium pyrosulfate flux. The samples were, therefore, fused with approximately 0.5 grams of potassium pyrosulfate, and the resultant fused samples were then dissolved in dilute HCl. The solution was then filtered; the filtrate made up to 100 cc volume, and subsequently analyzed by Atomic Absorption Spectroscopy (A.A.). The residue on the filter paper was then ignited in a muffle furnace at 900°C and the residue back weighed as SiO_2 . The results of these analyses were added to the results found for the acid extracted portion of the sample, and are tabulated in Table 2. Since the optical-emission spectroscopy analyses discussed later showed that Specimens 6E-1 and 6E-2 contained considerable quantities of calcium, A.A. analyses for calcium were run on these two specimens also. The instrumental parameters used for each element are listed in Table 3. Nitrous oxide-acetylene flames were used for Cr and Mg to eliminate inter-element interferences.* Because of the small amount of sample collected (30-120 mg) and the desirability of determining the toxic elements (Mn, Cd, Pb, As, Hg, Be) in low concentration levels, it was found necessary to use the entire sample for each of the above analyses.

The choice of these elements was based on the combined considerations of (i) expected presence in condensate, (ii) toxicity, and (iii) availability of atomic absorption lamps. The results are summarized as follows:

1. The samples from Furnace #25 invariably contain at least 66 wt% SiO_2 with an average value of 73.6 wt%. In contrast, the specimens from #6 contained only ~6 wt% SiO_2 as found in a supplemental optical-emission analysis. The SiO_2 did not come from the filter paper because the sample was removed from the quartz (SiO_2) paper prior to analysis.

* Walter Slavin, Atomic Absorption Spectroscopy, Interscience Publishers, New York, New York, pp 79-189, 1968.

TABLE 2. ATOMIC ABSORPTION ANALYSIS RESULTS
 ELEMENTAL CONCENTRATION (WT%)

Sample #	Ca	Cr	Mn	Cd	Pb	Hg*	Be	V	Mg	Fe	Zn	Al	SiO ₂
25W-1	--	2.11	0.059	0.002	0.0013	<0.05	<0.001	<0.03	4.7	0.45	1.68	<0.02	72.1
25W-2	--	0.74	0.065	<0.0003	<.0008	"	"	"	7.8	0.18	0.43	0.18	81.4
25W-2	--	1.68	0.081	"	.0012	"	"	"	7.2	0.37	1.56	0.079	74.4
25W-2	--	0.94	0.056	0.0007	<.0008	"	"	"	6.5	0.30	0.44	0.050	76.3
25W-2	--	1.22	0.028	<0.0003	"	"	"	"	7.1	0.44	0.39	<0.02	72.9
25E-1	--	2.08	0.104	0.0005	0.0008	"	"	"	7.6	0.43	0.84	0.11	70.5
25E-1	--	1.66	0.079	"	<.0008	"	"	"	11.3	0.40	0.50	0.11	67.5
25E-1	--	1.82	0.084	.0004	"	"	"	"	11.9	0.39	0.52	0.16	66.6
25E-2	--	1.15	0.064	<0.0003	"	"	"	"	8.2	0.22	0.78	0.14	75.7
25E-2	--	1.86	0.087	"	"	"	"	"	6.7	0.42	0.53	0.21	77.1
25E-2**	--	0.96	0.071	"	"	"	"	"	8.1	0.24	0.42	0.095	75.2
25E-2**	--	0.68	0.037	0.0005	"	"	"	"	5.1	0.27	0.30	0.024	SPILLED
6E-1	10.6	12.4	0.107	0.003	0.0067	"	"	"	7.6	4.9	3.9	0.59	12.2
6E-2	10.7	18.1	0.102	0.0018	0.0043	"	"	"	7.3	5.4	1.6	0.50	8.3

* Only the acid leach sample was analyzed. Hg would be lost during fusion.

** Part of the sample was lost due to a spill. The results here, therefore, represent only what was present in the acid leach.

TABLE 3
INSTRUMENTAL PARAMETERS

ELEMENT	WAVELENGTH ° (Å)	FUEL OXIDIZER SYSTEM	SLIT WIDTH (Å)	HOLLOW CATHODE CURRENT (MA)
Cr	3579	N ₂ O acetylene	2	10
Mn	2801	air acetylene	2	10
Cd	2288	air acetylene	7	4
Pb	2833	air acetylene	7	4
Hg	2537	air acetylene	7	4
Be	2348	N ₂ O acetylene	7	12
V	3184	N ₂ O acetylene	7	15
Mg	2852	N ₂ O acetylene	7	4
Fe	2483	air acetylene	2	12
Zn	2138	air acetylene	7	8
Al	3093	N ₂ O acetylene	7	13
Ca	4227	N ₂ O acetylene	7	8

2. The chromium content ranged from 0.68 to 2.08 wt% from Furnace #25 and the average value was 1.3 wt%. The value from Furnace #6 is much higher at 7.2 wt%.
3. The average manganese content was 0.067 wt% in Furnace #25 and 0.062 wt% in #6. The values are virtually the same.
4. The magnesium content varied considerably among different samples. It was highest, 11.6 wt% in the two 25E-1 specimens. These specimens also had the lowest SiO₂ levels (67.5 and 66.6 wt%) from among the samples from Furnace #25.
5. The iron content in Furnace #6 was a full factor of 10 higher than in Furnace #25. The average values in Furnace #6 and #25 are 3.75 and 0.34 wt%, respectively.
6. The zinc content in Furnace #25 varied from 0.30 to 1.68 wt% with an average of 1.04 wt%. The corresponding value for Furnace #6 is 2.20 wt%.
7. The average aluminum content from Furnace #25 was 0.10 wt%, and hence, lower than the 0.545 wt% found in the two samples from Furnace #6.
8. Mercury, beryllium, and vanadium, all toxic elements, were below the detectability limits of 0.05 wt%, 0.001 wt%, and 0.03 wt%, respectively.
9. The cadmium levels from Furnace #25 varied from below 0.0003 wt% to 0.002 wt%. The condensate from Furnace #6 contained an average of 0.0024 wt% cadmium which was somewhat higher than that for Furnace #25 but still relatively low. Cadmium is a toxic element.
10. Calcium analyses of Specimens 6E-1 and 6E-2 from Furnace #6 were decided on only after it was seen from the optical-emission spectroscopy results that the calcium levels were very high compared to the Furnace #25 specimens. The A.A. analyses for

calcium yielded 10.6 wt% and 10.7 wt% for the two specimens. Since the A.A. technique is more exacting than the optical-emission technique, it is the A.A. calcium values which should be considered as being the true calcium concentrations in the Furnace #6 specimens.

11. The total concentration of elements from Furnace #25 samples is virtually 100% when all the metal values are converted to equivalent oxide percentages.* This means that all the major elements in the emissions from this furnace were detected and, in addition, a few minor but toxic elements (V, Hg, Be, Cd) were also detected.
12. The total concentration of elements from Furnace #6 after conversion to equivalent oxide percentages is 70%, a somewhat less satisfactory mass balance situation than for Furnace #25. This lack-of-closure should not be taken to signify the presence of an additional but undetected element. No additional element of any consequence was detected in either the electron microprobe or optical-emission techniques. It is concluded that all the major elements are accounted for in Table 2 and that the lack-of-closure in samples from Furnace #6 is due to errors associated with the extreme difficulty encountered in dissolving the samples.

2.4 Electron Beam X-Ray Microanalysis

The electron microprobe is an advanced piece of equipment which uses a small beam of electrons to produce characteristic X-ray emissions from a sample volume with a radius of ~1 micron. Curved crystal X-ray spectrometers are used to analyze the resultant characteristic X-ray spectra. In these analyses, the electron beam was defocused to a diameter of 200 microns (0.008 inch) to cover a larger segment of the sample.

* Equivalent oxide percentages are obtained by multiplying the weight percent metal in Table 2 by the ratio Mo/M_m where Mo is the molecular weight of the metal oxide and M_m is that of the metal. The oxide formulae were taken to be Al_2O_3 , ZnO , Fe_3O_4 , MgO , A_2O_3 , and CaO . Thus for Ca, the equivalent oxide percentage is $10.65 \times (40+16)/40$. Justification for this conversion is based on electron microprobe results.

The electron beam impinged in vacuum upon the untouched sample surface as shown in Figure 1. An examination was made of the complex spectrum of X-rays given off by the specimen under electron beam excitation, and it was found that the entire spectrum could be identified uniquely on the basis of the elements shown in Table 4. All portions of the X-ray spectrum in the wavelength range 1-100A covering all elements except H, He, Li, and Be were taken into account.

The silicon and oxygen signals did not originate from the silica filters although the latter were present in the electron microprobe chamber. The electron beam penetrated about 2 microns (and absolutely no more than 20 microns) into the sample from the top surface. The total sample thickness was about 0.02 inch (~500 microns). Thus, the silica filter material was ~500 microns away from the effective sensing depth of the electron beam.

The major outcome of the electron microprobe analyses was that the main elements were identified for the atomic absorption analysis already discussed. Thus, Fe, Cr, Si, Al, Ca, Mg, and Zn were found on the untouched samples and were, therefore, selected along with other elements, for A.A. analyses.

A second outcome of the electron microprobe analyses was the detection of oxygen at roughly the 50% level in the samples from both furnaces. This means that the metals are present as oxides and is the basis for the conversion of the metal percent values in Table 2 to equivalent oxide percents.* The 50% oxygen value was strictly applicable only to the top 2-20 microns of the untouched samples where the analyses were made. However, it was assumed that the sample was essentially a mixture of oxides throughout its depth. Such an assumption seemed reasonable when the source of the samples was taken into account.

* The 50% value was obtained in a 10^{-6} torr vacuum. Thus oxygen was not an occluded atmospheric gas but was present as an oxide.

TABLE 4. ELECTRON BEAM X-RAY MICROANALYSIS RESULTS FROM
QUALITATIVE ANALYSES

Sample	Elements Positively Identified in X-ray Spectra
25E-1	Fe, Cr, O, Si, Al, Ca, Mg, Zn, Na, Ba, K, -, -, -
25E-2	Fe, Cr, O, Si, Al, Ca, Mg, -, -, -, -, Ni, -, -
6E-2	Fe, Cr, O, Si, Al, Ca, Mg, Zn, Na, -, -, Ni, Cl, S

The concentrations are not given in the electron microprobe table (Table 4) because, although the elements shown were present throughout the depth of the samples, their concentrations (particularly the metals) varied with depth (i.e., the samples were non-uniform). Thus, the atomic absorption analyses were used to determine the quantitative analyses on properly composited samples while the electron microprobe qualitatively identified the elements.

2.5 Optical-Emission Spectroscopy

Optical-emission spectroscopy or arc-spark spectroscopy consists of electrical excitation of the electrons of the elements in the sample. When the electrons return to their ground state, light is emitted. The emitted light is passed through a prism or diffraction grating to separate it into its component wavelengths. The spectrum is then analyzed electronically or optically on a photographic plate. Each line occurring at a definite wavelength position on the spectrum designates a specific element, and the intensity of light at that wavelength is proportional to the quantity of that element present.

Portions of the samples were subjected to optical-emission analyses to provide (i) a check on the analytical procedures (particularly the lack-of-closure in the atomic absorption analyses from Furnace #6), and (ii) a more sensitive approach to trace element analysis than that provided by electron beam X-ray microanalysis. The spark emission results for the major elements agreed well with the atomic absorption and electron beam results and, in addition, identified numerous trace impurities not found in the other approaches. The results are compiled in Table 5.

TABLE 5. OPTICAL EMISSION ANALYSES (WT%)

Sample No.	Si	Mg	Al	Fe	Cr	Zn	Ca	Na	Ni	Mn	V	Cu	Co	Ti	B	Pb
25W-1	40	4	2	1	1	0.2	0.8	0.005	0.05	<0.005	<0.001	0.001	0.005	0.005	0.03	
25W-2	40	6	2	1	0.2	0.1	0.4	0.005	0.05	<0.005	<0.001	0.001	0.005	0.005	0.01	
25W-2	40	6	2	1	0.4	0.2	0.6	0.005	0.05	<0.005	<0.001	0.001	0.002	0.005	0.01	
25W-2	40	6	2	1	0.4	0.1	0.4	0.005	0.05	<0.005	<0.001	0.001	0.001	0.005	0.01	
25E-1	40	6	2	1	0.4	0.2	0.6	0.005	0.05	<0.005	<0.001	0.001	0.001	0.005	0.01	
25E-1	40	6	2	1	1	0.2	0.8	0.005	0.05	<0.005	<0.001	0.001	0.001	0.01	0.01	
25E-1	40	10	2	1	0.4	0.2	0.8	0.005	0.05	<0.005	<0.001	0.001	0.002	0.01	0.01	
25E-1	40	6	2	0.5	1	0.4	0.2	0.8	0.005	0.05	<0.005	<0.001	0.001	0.002	0.02	0.01
25E-1	40	8	3	0.5	1	0.4	0.2	0.8	0.005	0.05	<0.005	<0.001	0.001	0.002	0.005	0.01
25E-2	40	6	2	1	1	0.4	0.2	0.4	0.005	0.05	<0.005	<0.001	0.001	0.002	0.005	0.01
25E-2	40	6	2	1	1	0.4	0.2	0.4	0.005	0.05	<0.005	<0.001	0.001	0.002	0.005	0.01
25E-2	40	4	2	1	0.4	0.2	0.4	0.005	0.05	<0.005	<0.001	0.001	0.002	0.005	0.01	
6E-1	3	10	3	10	10	2	20	1.0	0.3	0.05	<0.03	<0.001	0.03	0.03	0.005	0.01
6E-2	3	10	2	10	10	3	20	1.0	0.3	0.05	<0.03	<0.001	0.03	0.03	0.005	0.01

Note: Values of Major Elements \pm 30% of Reported Value.
 Elements listed below not detected. The limits of detection are given.

Hg 200 PPM
 As 200 PPM
 Te 200 PPM
 Sb 50 PPM
 Be 2 PPM
 Cd 100 PPM

4742.3.71-139
 23 September 1971
 Page 17