

FROM

P. R. ATKINS
T. B. BONNEY
V. W. RIEKE

TO SEE DISTRIBUTION LIST

July 26, 1973

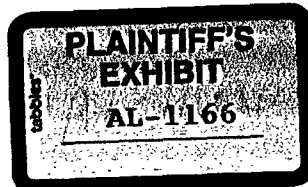
RE: AIR SAMPLING REQUIREMENTS

The recent activities of EPA and OSHA and their State counterparts have resulted in increased demands for in-plant, source and ambient air sampling. Extensive testing is necessary to provide a basis for choosing the proper air pollution control techniques and designing the necessary equipment. Compliance testing is required after each control project is completed. Source testing is, or soon will be, required semi-annually or more often for all "significant" emission points. These results will have to be reported to the state agencies. Ambient monitoring, in addition to our existing fluoride sampling program, may be required in some states.

Similarly, OSHA will increase its demands for in-plant sampling. At the present time only one comprehensive OSHA standard exists for which there are sampling requirements. This is the asbestos standard. On the other hand, on the present priority list there are about 450 chemical and physical agents for which comprehensive standards will be written. A look at those standards whose promulgation is imminent assures us that environmental monitoring will be a requirement in most of them.

It is obvious that these sampling requirements are extensive and necessitate the formulation of a detailed manpower and resource allocation plan. Some plants may be capable of performing all the required sampling work on schedule, but many will need to make some changes. In order that we can better determine the company-wide needs, and coordinate the company's efforts in this area, please provide me with the following information by August 17, 1973:

1. A list of all routine sampling activities that occur at your plant and the schedule for these tests. Note the pollutants that are tested and briefly describe the sampling methods used.
2. A list of all emission sources that presently are not sampled routinely but in your opinion need to be sampled. Note the pollutants which should be measured. If any major problems exist which might make sampling difficult at any emission point, note this.



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3. A list of all the remaining emission sources that you feel don't necessarily need to be sampled.
4. A description of the ambient sampling program presently maintained at your plant and your comments on how it should be improved.
5. The names, background and experience of all personnel that conduct sampling tests at your plant. Estimate the manhours required from these individuals and from supervisory personnel.
6. A list of "problem areas" which you feel will require sampling equipment or expertise beyond the capabilities of your staff.

The information that you submit will be used in conjunction with input from Environmental Engineering, Health and Environment and operating departments to develop a detailed plan that will insure that the company will be able to meet all the sampling requirements at every operating location. Beginning in late summer, a series of four-day short courses in air sampling technology will be offered for sampling personnel. Information from this survey will aid in the development of this short course.

Your cooperation in providing as much information as possible will be appreciated.

PATRICK R. ATKINS

bg *TB Bonney*
VW Ruck

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FROM R. E. PODHORA/A. A. RAMBIKUR
ENVIRONMENTAL CONTROL
POINT COMFORT OPERATIONS

TO DR. P. R. ATKINS
PITTSBURGH OFFICE

February 27, 1974

cc. → Mr. T. B. Bonney - Pittsburgh
Mr. V. W. Rieke - Pittsburgh
Mr. A. A. Rambikur - Point Comfort
Mr. C. L. Green - Point Comfort
Dr. L. F. Brennecke - Point Comfort

RE: AIR SAMPLING REQUIREMENTS

In response to your letter of July 26, 1973, the following are data and comments you requested.

1. Table I shows the Point Comfort plant routine sampling activities, including the pollutant, frequency of sampling and sampling method used. Included in this table are the source numbers for reference to our TACB emissions inventory for further details.
2. Table II shows emission sources that presently are not sampled, but in our opinion need to be sampled.
3. The following emission sources do not necessarily need to be sampled at this time.

	<u>Source Number</u>
a. Five stacks (POC of natural gas)- Oil & Gas Plant	111
b. One Elevated Flare - Oil & Gas Plant	112
c. Smelting power individual engine exhaust stacks	114
d. Alumina reactivator - Power Department	115
e. Cryolite mist eliminator	169
f. H ₂ emergency vent stack	177
g. Chlor-Alkali boilers	181
*h. R-110 low pressure boiler	182
*i. R-110 boilers 1-5	193 - 197

*-Provided we remain on natural gas fuel.

4. The ambient air sampling program presently maintained is described below.

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- a. High volume air samples are obtained at three locations:

- (1) Witco pumps (Smelting dock area).
- (2) Witco gate, next to the company fence along Highway 35.
- (3) Mr. A. A. Rambikur property, Point Comfort City.

These are 24-hour samples, 4 days per week.

- b. One Leigh Instrument monitors ambient F^- 24 hours/day (located at A. A. Rambikur property (City of Point Comfort)).
- c. One MSA Lira Instrument monitors ambient carbon monoxide 24 hours/day. Located at A. A. Rambikur property.

The ambient air sampling program could be improved to include the up-wind data and expanded to include ambient air monitoring around the Chlor-Alkali plant for chlorine and mercury and F^- around the Al F_3 Plant. We may need to monitor for CO in the potrooms and total oxidants to confirm State's data in Point Comfort.

5. An estimate of manhours is shown below. See Table III for personnel data.

<u>Type of Test</u>	<u>Man Hours/Test</u>	
	<u>Technicians</u>	<u>Supervisory</u>
a. Compliance dust tests		
Tests requiring 2 men	6-10 MH	0.5 - 1
Tests requiring 3 men	9-12 MH	0.5 - 1
b. Engineering tests, i.e., flows and/or particle size determination.		
2 Men	6-10	0.5 - 1
3 Men	9-12	0.5 - 1
c. Hg Sampling		
Ambient - Cell House	6	0.5
Hg Fume Stack and H_2 Line	7	0.5
d. Asbestos	2-4	0.25 - 0.5
e. Smoke Opacity	1-2	0.25 - 0.5
f. Detector Tubes	1	0.25 - 0.5

	<u>Technicians</u>	<u>Supervisory</u>
g. Ind. hygiene	1 - 8	0.5 - 1
h. Potroom Scrubber Towers		
Inlet	8	0.5
Outlet	8	0.5
i. Potroom monitor (24 hour)	1 (set up)	0.5
	1-2 (periodic inspection and adjustment)	
j. Lab analyses (for #8 and #9)	4-7	0.5 - 1
Elapsed time - 17 hours.		6.25 - 8

Compliance sampling, ambient monitoring, mercury sampling, asbestos monitoring, and engineering tests occupy the time of two Chemical Engineering Technicians, one-half the time of a Laboratory Assistant and one-half the time of three Day Analysts.

6. The Point Comfort air sampling team has ample expertise to handle most problems with occasional advise or recommendations from environmental health lab personnel.


R. E. PODHORA

REP:vh

P.S. I must apologize for the lateness of this response. Due to the press of other matters, your request for information was put aside and neglected.

A. A. RAMBIKUR

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TABLE I
ROUTINE SAMPLING ACTIVITIES

<u>Area: Smelting</u>				
<u>Location</u>	<u>Source No.</u>	<u>Frequency</u>	<u>Pollutant</u>	<u>Sampling Method</u>
1. Power Department				
Exhaust heat boilers (24)	113	2/Year	Smoke	Visual opacity, Lear-Siegler Transmissometer
2. Potroom Scrubbers	116			
a. Inlet		2 sets towers/ quarter	F ⁻ & Part.	Thimbles ASME
b. Outlet		2 towers/ month	F ⁻ & Part.	Petrey filters & impingers/ NaOH Soln.
3. Monitors	117	2/Qtr.	F ⁻ & Part.	Petrey filters & impingers/ NaOH Soln.
4. Flotation Cyclone	118	2/Year	Particulate	Thimbles--ASME
5. Flotation Bag Col.	122	2/Year	Particulate	Thimbles--ASME
6. Flotation Drum Drier	190	2/Year	Particulate	Thimbles--ASME
7. Paste Plant EP	123	2/Year	Particulate	Thimbles--ASME
8. Paste Plant Bag Col.	125	2/Year	Particulate	Thimbles--ASME
9. Paste Plant #1 Air Classifier	191	2/Year	Particulate	Thimbles--ASME
10. Paste Plant #2 Air Classifier	192	2/Year	Particulate	Thimbles--ASME
11. Alumina Unloading, Building 40	126	2/Year	Alumina Dust	Thimbles--ASME
12. Alumina Unloading Building 41	127	2/Year	Alumina Dust	Thimbles--ASME
13. Ingot Casting				
a. Furnace Stacks	130	2/Year	Particulate, Cl ⁻ , Cl ₂	Thimbles--ASME, Impingers
b. Skim Shed	131	2/Year	Particulate	Thimbles--ASME
14. Potlining--Shot blast collector - 54E	132	2/Year	Particulate	Thimble--ASME
15. Marinite Collector	134	2/Year	Asbestos	MSA Personal Sampler and Filter.

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TABLE I - continued

Area: Refining

<u>Location</u>	<u>Source No.</u>	<u>Frequency</u>	<u>Pollutant</u>	<u>Sampling Method</u>
1. Lime Kiln Cold end (Venturi)	141	2/year	Lime dust	Thimbles--ASME
2. Lime Storage	144	2/year	Lime dust	Thimbles--ASME
3. Bauxite Crushing (bags bad 10-24-73)	148	2/year	Bauxite dust	Thimbles--Asme
4. Calcination				
a. R-50 - EP-1	151	2/year	Alumina Dust	Thimbles--ASME
b. R-50 - EP-2	185	2/year	Alumina dust	Thimbles--ASME
*c. R-55 - EP-2	186	Often	Alumina dust	Thimbles--ASME
*d. R-55 - EP-3	187	Often	Alumina dust	Thimbles--ASME
5. Bulk Loading				
a. R-52	159	2/year	Al ₂ O ₃ Briquette Dust	Thimbles--ASME
b. R-53	158	2/year	Briquette Dust	Thimbles--ASME
6. Spar Grinding Col. R81	160	2/year	Spar Dust	Thimbles--ASME
7. Spar Drying R81-- *Aerodyne-air tumbler	161	Often	Spar Dust	Brinks and Anderson Impactor--Thimbles--ASME
8. a. Spar storage R-82 top	163	2/year	Spar Dust	Thimbles--ASME
b. Spar storage R82 grd.	168	2/year	Spar Dust	Thimbles--ASME
9. AlF ₃ wet scrubbers (converters)	165	2/year	Particulate, HF	Thimble--ASME, Impingers
10. #2 Hydrate drier air tumbler.	166	2/year	Hydrate Dust	Thimbles--ASME
11. #2 Nuisance air tumbler.	167	2/year	Hydrate Dust or AlF ₃	Thimbles--ASME
12. AlF ₃ Storage R86A	183	2/year	AlF ₃ dust	Thimble--ASME
a. #1 Hydrate drier air tumbler	188	Often	Hydrate	Thimble--ASME
b. #1 Nuisance air tumbler	189	Often	Hydrate or AlF ₃	Thimble--ASME

*-Numerous Engineering Tests

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TABLE I - continued

Area: Refining

<u>Location</u>	<u>Source No.</u>	<u>Frequency</u>	<u>Pollutant</u>	<u>Sampling Method</u>
13. Cryolite				
a. Fluid drier venturi	170	2/Year	Cryolite dust	Thimble - ASME
b. Fine collector	171	2/Year	Cryolite dust	Thimble - ASME
c. Pelletizer Col.	172	2/Year	Cryolite Dust	Thimble - ASME
d. Pelletizer drier venturi	173	2/Year	Cryolite Dust	Thimble - ASME
14. Chlor-Alkali				
a. Hg vapor stack	178	1-2/Week	Hg	EPA with Alcoa impinger train.
b. H ₂ line to R-110 powerhouse	-	1-2/Week	Hg	EPA with Alcoa impinger train.
c. Cell house ambient air.	-	1-2/Week	Hg	Cell house traverse with mini-impingers.

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TABLE II

EMISSION SOURCES THAT NEED TO BE SAMPLEDArea: Refining

<u>Location</u>	<u>Source No.</u>	<u>Pollutant</u>	<u>Remarks</u>
1. Raw Materials			
a. Bauxite unloading (fugitive)	145	Bauxite Dust	Sometime in future, dust collection must be provided.
b. Bauxite transfer (fugitive)	146	Bauxite dust	Sometime in future, dust collection must be provided.
c. Bauxite storage (fugitive)	147	Bauxite dust	Sometime in future, dust collection must be provided.
d. Bauxite Storage			
R-25N	149	Bauxite dust	Revise existing collector.
R-25S	145	Bauxite dust	Revise existing collector.
2. Chlor-Alkali Diamond Snift vent stack	179	CCl_4 & Cl_2	3" diameter vent line off the absorber and normally runs at 80 - 90 psi.
3. Chlor Alkali Hypo Unit	180	NaOH Mist, Cl_2	Intermittently used for Cl_2 NaOH Mist continually.
4. Paste Plant Fume Stack	124	Carbon dust and Hydro- carbons	Thimbles - ASME, Alcoa 4070

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TABLE III

PERSONNEL: BACKGROUND, EXPERIENCE

<u>Name & Statistics</u>	<u>Background</u>	<u>Environmental Experience</u>
<u>Tom Flores</u> Age: 38 Co. Sen. 18-1/2 years College: 2 years Reports to A. A. Rambikur	Potrooms, Casting Lab, Quantometer Lead Analyst, 10 Years.	Since February, 1968, Chem. Engr. Technician. Development of dust testing, air sampling, industrial hygiene sampling. Qualified smoke reader. Qualified asbestos analyst.
<u>E. A. Brogger</u> Age: 37 Co. Sen. 15 years College: No Assigned to Lab	Lab - Process Control Bath Lab, Oil Lab, Quantometer	Since mid-1968, water analyses, Hg sampling, ambient and stack Bay solids/Hg, urine analyses, vegetation samples, industrial hygiene hi-volume air, Leigh instrument, Lira CO-monitor.
<u>R. L. Nolen</u> Age: 39 Co. Sen. 15 years College: 4 Hours Chem. Assigned to Lab	Paste Plant lab, Power Maint. Lab - Process Control, Chlor-Alkali Lab, vac. relief and Lead Analyst.	Since June, 1969, water analyses, Hg sampling - ambient and stack Bay solids/Hg, urine analyses, industrial hygiene, hi-vol. air, Leigh instrument, Lira CO-Monitor.
<u>L. F. Wagner</u> Age: 41 Co. Sen. 17 years College: No Assigned to Lab	Texas Hiway Dept. Surveyor, Supply School, U. S. Army. Surveyor - Alcoa Const. 7th potline, Refining Plant, Warrick Operations, Lab 1959, Jr. Analyst, Bath Lab.	Since June, 1969, water analyses, Hg sampling, ambient and stack Bay solids/Hg, urine analyses, industrial hygiene, hi-vol air., Leigh Instrument, Lira CO-Monitor.
<u>L. W. Onken</u> Age: 31 Co. Sen. 8-1/2 years College: 1 + years Reports to A. A. Rambikur	Lab - Process Control Chlor-Alkali Lab, Quantometer, AlF ₃ task force, Ft. Meade Plant, Florida.	Since October, 1973, Chem. Engr. Technician. Dust testing. Qualified smoke reader. Qualified asbestos analyst.
<u>E. A. Kozelsky</u> Age: 27 Co. Sen. 6 Years College: 1 + years Assigned to Lab	U. S. Marines, Supply. Lab--Process Control sampler. Storeroom. Lab--Process Control	Since January 15, 1974, effluent sampler. Dust test sampling assistant.
<u>R. J. Putnam</u> Age: 26 Co. Sen. 5 + Years College: 2 Years Assigned to Smelting	Roughneck--oil fields. Computer - bank Office - Invoice. Lab Assistant. Cathodic Protection.	November, 1969, Process Engr. Tech. (Smelting). Potroom scrubber tower testing. Potroom monitor testing. See H. E. Bonds' letter 1-9-74. Re: Environmental Tests & Inspection (not included)

409673 0066

September 13, 1973

FROM J. D. BREAZEAL
ENGINEERING & MAINTENANCE DIVISION
DAVENPORT WORKS

TO P. R. ATKINS
HEALTH & ENVIRONMENT DEPARTMENT
PITTSBURGH OFFICE - 7

C. C. to:
V. W. Rieke - Pittsburgh Office 7
T. B. Bonney - Pittsburgh Office 7
R. J. Reitz - Davenport Works
K. Williams - Davenport Works

RE: AIR SAMPLING REQUIREMENTS

In response to your July 26, 1973 memo to R. J. Reitz, Safety and Environment Steering Committee Chairman, on the captioned subject we are forwarding the attached "Information" sheets. These attachments contain information on the six subjects requested, numbered to correspond to the questions in your July 26 letter.

Our in-plant and stack sampling program is progressing at a slow rate, due primarily to a lack of trained manpower. We hope to take full advantage of any short courses in sampling technology offered by the company as mentioned in your letter.

J. D. BREAZEAL

JDB/11

Attachments (4)

409673 0074



TO: P. R. ATKINS
September 7, 1973

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Information - Sampling Requirements

I. List of all routine sampling activities:

No in-plant or stack sampling is done on a routine basis. Sources and areas are only checked when there is a specific need or request.

Operations or areas that have been sampled at least once are as follows:

Stack Sampling

Ingot Melting & Holding Furnaces - Particulate, Beryllium,

Hcl & Cl₂ Gas

Solid Waste Incinerator - Particulate

Foil Rolling Mill Exhausts - Particulate

Hill Acme Polisher -
Scrubber Exhaust - Particulate

Skim House Rotary Barrel Stacks - Particulate, Beryllium

Skim House Dust Collector Stack - Particulate, Beryllium

In-plant Sampling

Ingot Plant - Beryllium, Cadmium, Chlorine, Hcl, SO₂

Hot Line - Oil Mist Vapors

Cold Mills - Oil Mist Vapors

Foil Mills - Oil Mist Vapors

Preheat & Reheat Furnaces - Fluoride Fume, Carbon Monoxide

1A. Sampling Procedures - Stack Samples

- a. Pitot tube measurements taken to determine velocity profile, CFM, and sampling position.
- b. Sample collected using glass probe, Petrey filter holder and glass fiber filter, impingers (where required), gas meter, and vacuum pump.
- c. Analysis of samples.

c-1. Beryllium samples are forwarded to Industrial Hygiene Laboratory--Alcoa Technical Center,

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TO: P. R. ATKINS
September 7, 1973

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for analysis.

- c-2. Samples from Ingot Plant operations are analyzed according to "Method for Sampling and Analyzing Stack Effluents from Chlorine - Fluxing Operations" received from T. B. Bonney (copy attached).
- c-3. Particulate emissions from other sources determined from weight gain of dry glass fiber filters.

II. List of sources that have not been sampled but that need to be checked:

Stack Samples

Paint Line	- Hydrocarbons
Foil Rolling Mills	- Hydrocarbons
Cold Rolling Mills	- Hydrocarbons
Laminator Bay Stacks	- Hydrocarbons
60" Process Line (Solvent Cleaning)	- Hydrocarbons
84" Process Line (Solvent Cleaning)	- Hydrocarbons
#4 Tension Level Line (Solvent Cleaning)	- Hydrocarbons
Melting & Holding Furnaces	- Opacity or Ringleman number

In-plant Sampling - first (or additional samples required)

Ingot Plant - Check for asbestos, beryllium, nuisance ducts, Cl_2 & HCl gas, metallic chloride fumes, fluoride fumes (from fluxes, skim pots, & processing) sulfur dioxide, carbon dioxide, cadmium (2021 alloy) copper oxide dusts, phosphate ester fumes.

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TO: P. R. ATKINS
September 7, 1973

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Hot Rolling Area - Soluble oil mist and fumes, phosphate
ester fumes.

Plate Mill Area - Oil fumes from sawing operation, 1,1,1,-
trichloroethane vapors, ozone.

Furnace Department - Carbon monoxide, smoke.

Sheet Finishing - Solvent vapors.

Paint Line - Fluoride mists, chromate mists, solvent fumes.

Cold Mills - Oil mists & fumes, nuisance dusts in filter houses.

I. P. S. - Solvent vapors, vapors from TEC chemical stripable
coating-799.

Foil Mill - Solvent fumes, oil mists, paper dusts, carbon monox-
ide, nuisance dusts in filter house.

Machine Shop - Ozone in welding area.

III. List of remaining sources that don't need sampling:

Foil Annealing Furnaces - 8	Preheat Furnaces - 9
Reheat Furnaces - 19	Soaking Pit Exhausts - 26
Ageing Furnaces - 2	

IV. Description of ambient sampling program: --none, with present regu-
lations, there is no apparent need for an ambient sampling program.

V. Names, background, and experience of sampling personnel:

Actual stack samples are collected and analyzed by one or more
chemical analyst from Chemical Laboratory. These are hourly employees
with no specific training other than that obtained by experience in
the laboratory.

Supervision: K. W. Williams, Laboratory Supervisor, B.A. Degree,
major in biology, no formal training in stack sampling,
15+ years experience in Davenport Chemical Laboratory.

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TO: P. R. ATKINS
September 7, 1973

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M. K. Sonksen, B.S.M.E., no formal training in stack sampling procedures.

Man-hours required vary depending on the testing required.

Example: 3 sets of Ingot stack samples require 16 man-hours for sampling, 8 hours for analytical work, 2-4 hours laboratory supervision, and 1-2 hours engineering time. In the last half of 1972 a total of 640 hours were spent in collecting and analyzing stack samples, not including supervisory or engineering time.

VI. "Problem Areas" that will require expertise or equipment beyond Davenport's capabilities:

1. Collection and analysis of hydrocarbon samples.
2. Determination of quantity and identification of solvent vapor fumes.
3. Beryllium analysis - currently done by Technical Center, Merwin.
4. Determination of fluoride fumes.
5. Need for a trained technician to arrange and supervise sampling and write up reports on results.

M. K. SONKSEN

MKS;11

409673 0078

2K

METHOD FOR SAMPLING AND ANALYZING STACK EFFLUENTS
FROM CHLORINE-FLUXING OPERATIONS

1. Scope

The method covers sampling and analysis of particulate and gaseous effluents from stacks exhausting chlorine-fluxing operations. The components are principally HCl, Cl₂, Al₂O₃ and AlCl₃ (or Al₂O₃ with adsorbed HCl), as gases, aerosols and particulate. Some of these are not specifically identified but all are accounted for. There is a separation of particulate from gaseous components and these are analyzed separately. For convenience, the particulates are reported as Al₂O₃ and HCl.

2. Summary of Method

- 2.1 A measured volume of the effluent stream is sampled through a filter paper to separate particulates, then through a solution of sodium hydroxide to absorb chlorine and hydrogen chloride gases.
- 2.2 The filter retaining particulates is analyzed for aluminum oxide gravimetrically, and for total chloride as HCl by titration after leaching from the filter into solution.
- 2.3 The liquid impinger sample is analyzed for chlorine by titration of an aliquot portion. Another aliquot is analyzed for chlorides, and the titration obtained includes the chlorine present in the sample. Subtraction of the HCl equivalent of Cl₂ present from the chloride titration gives the chlorides present as HCl.

NOTE: The Mohr titration is presented in this method for chloride determination. The Volhard titration is also acceptable and may be used at the chemist's discretion. The chloride specific ion electrode is also recommended and its use is briefly described in an appendix.

3. Sampling Apparatus

- 3.1 Sampling Probe. Use a borosilicate glass tube (Vycor is required for temperatures above 500°C) of 10-12 mm I.D. and long enough to reach the appropriate sampling point with about one foot outside the stack to cool the gases enough to prevent charring the filter. This tube should have a 90° bend of ca. 2-inch radius on the inlet end.
- 3.2 Petrey Filter Holder. Holds 12.5 cm dia. filter paper. Whatman #42 is used.
- 3.3 Greenburg-Smith Standard Impingers. 500 ml capacity. Two required.
- 3.4 Metering Device. Sprague dry gas meter or equivalent. Must be calibrated and include a dial thermometer and vacuum gauge for correcting volumes to standard conditions.
- 3.5 Vacuum Source. Willson or Gast pump, air ejector, or equivalent, capable of pulling 1/2-1 CFM through the sample train.

409673 0079

- 3.6 Pitot Tube and Draft Gauge for determining stack velocity.
- 3.7 Thermometer or Thermocouple (range to 1500°F) for determining dust or stack temperatures.
- 3.8 Walkie-Talkies for communicating with sampling coordinator at the furnace.
- 3.9 Stop-Watch
- 3.10 Data Sheets

4. Analytical Reagents

- 4.1 Sodium Hydroxide (NaOH), 1N; dissolve 40 g of NaOH pellets in H₂O and dilute to 1000 ml.
- 4.2 Sulfuric Acid (H₂SO₄), 0.01N; add 3 ml of H₂SO₄ (sp.gr. 1.84) to H₂O and dilute to 1000 ml.
- 4.3 Sulfuric Acid (H₂SO₄), 2N; add 60 ml of H₂SO₄ to H₂O and dilute to 1000 ml.
- 4.4 Potassium Chromate indicator (K₂CrO₄); dissolve 10 g K₂CrO₄ in 100 ml H₂O.
- 4.5 Potassium Iodide (KI), crystals
- 4.6 Hydrochloric Acid (HCl), 4N; add 50 ml HCl (sp.gr. 1.19) to 100 ml H₂O.
- 4.7 Sodium Thiosulfate (Na₂S₂O₃·5H₂O), 0.1N; dissolve 24.819 g Na₂S₂O₃·5H₂O in H₂O and dilute to one liter.
- 4.8 Silver Nitrate (AgNO₃), 0.1N; dissolve 16.989 g AgNO₃ in H₂O and dilute to 1 liter. Standardize with Sodium Chloride.
- 4.9 Starch Indicator (Soluble Starch); mix 1 g soluble starch into a paste in 5 ml H₂O with a mortar and pestle. Rinse into 100 ml hot H₂O and boil 3 min. (Substitutes may also be used for indicators in iodimetry. Thyodene is recommended.)
- 4.10 Ammonium Hydroxide (NH₄OH); sp.gr. 0.899.
- 4.11 Methyl Red Indicator Solution, 0.04%; dissolve .1 g of Methyl Red in 18.6 ml 0.02N NaOH, dilute with H₂O to 250 ml.
- 4.12 Phenolphthalein Indicator, 0.1%; dissolve 0.1 g in 60 ml alcohol and dilute with H₂O to 100 ml.
- 4.13 Nitric Acid (HNO₃); sp.gr. 1.41.

4.14 Ammonium Nitrate Wash Solution; dilute 25 ml NH_4OH , sp.gr. 0.899, with H_2O , neutralize using Methyl Red with 1:1 HNO_3 , add 1 ml excess NH_4OH (sp.gr. 0.899) and dilute to 1000 ml.

4.15 Sodium Chloride (NaCl) 0.1N; dissolve 5.845 g NaCl in H_2O and dilute to 1000 ml.

5. Sampling Procedure

5.1 Determine velocity and volumetric flow rate of stack gases (Ref. Method 4070.5.2)

5.2 Choose sampling location. The ideal location is 5 to 10 diameters downstream from any bend, branch, or blower, and 3 to 5 diameters upstream from similar causes of turbulence. Since this is usually non-existent, an optimum location is determined by surveying several profiles at available locations. The most uniform profile marks the best location. From the profile obtained and the average velocity calculated choose for sampling that point which is nearest the average velocity.

5.3 Collecting the Sample.

5.3.1 Determine the sampling rate by referring the average velocity above to the chart. (Fig. 9.2)

5.3.2 Duration of the test is determined by the operation. Sampling should continue through a complete cycle, including fluxing and skimming.

5.3.3 Assemble the equipment (See Fig. 9.1)

Load the filter holder with 12.5 cm diameter Whatman #42 filter paper and connect to two standard impingers, each containing 100 ml 1N NaOH and protected from dust by covering the ground joint with masking tape. Place the gas meter next between the sample train and the suction source, and control the flow rate with a screw clamp on the meter outlet hose.

5.3.4 Sampling procedure.

5.3.4.1 Check system for leaks before sampling by blocking the nozzle and applying about 10 in. mercury vacuum. Close the clamp K and, if the vacuum holds, release the nozzle slowly and proceed with sampling. A drop in vacuum indicates a leak, which must be corrected.

5.3.4.2 Insert sampling probe at the proper location (5.2), directing the nozzle into the gas stream with the clamp K closed. Start the vacuum source and open the clamp to the desired flow rate (5.3.1). This rate should be observed and adjusted as frequently as necessary throughout the entire sampling period, noting time, temperature, and pressure for each change. At the end of the sampling period, the filter holder and impingers are sealed to avoid contamination or loss while transporting to the laboratory.

6. Analysis of Samples

6.1 Filter sample

6.1.1 Transfer paper to a beaker, brushing or washing any residues from inside the holder (3.2) and sampling probe (3.1), adding enough water to cover the paper. Add 3 drops Methyl Red indicator and NH_4OH (4.10) until just alkaline. Bring to a boil and filter through Whatman #40 paper, or equivalent, into a 250 ml volumetric flask. Wash with hot NH_4NO_3 wash solution (4.14). Cool, dilute to 250 ml, mix, and set aside for determination of HCl.

6.1.2 Place the paper containing washed precipitate in a tared crucible and ignite at 1000°C for one hour. Cool and weigh. Record the increase in weight as Al_2O_3 (retain the residue for analysis of minor constituents, if needed).

6.1.3 Calculation of Al_2O_3 concentration

$$\text{Al}_2\text{O}_3, \text{mg/m}^3 = \frac{W_n}{V} \quad \text{where:}$$

W_n = net weight of Al_2O_3 in mg

V = Volume of gas sample in cubic meters S.T.P.
(25°C , 760 Torr.)

6.1.4 Transfer a 50 ml portion of filtrate (6.1.1) into a casserole, add 3 drops phenolphthalein indicator (4.12) and H_2SO_4 (4.2) until just acid. Add a few drops of K_2CrO_4 indicator (4.4) and titrate with 0.1N AgNO_3 (4.8) to the first color change from pure yellow. Deduct a determined blank.

6.1.5 Calculation of HCl concentration

$$\text{Total wt. HCl, mg} = T_n \times N \times 36.5 \times \text{A.F.}$$

$$\text{Conc. HCl, mg/m}^3 = \text{wt. HCl, mg/V}$$

$$\text{Conc. HCl, ppm} = \text{mg HCl/m}^3 \times 0.670$$

T_n = net ml AgNO_3 titer

N = normality AgNO_3

36.5 = m.e.wt HCl

A.F. = aliquot factor $\left(\frac{250 \text{ ml total volume}}{50 \text{ ml aliquot}} \right)$

V = sampled vol. in m^3 S.T.P.

0.670 = conversion factor mg HCl/ m^3 to ppm

6.2 Impinger Samples

6.2.1 Transfer contents and washings of both impingers into 250 ml volumetric flasks and make up volume with water.

6.2.2 Chlorine. Pipet a 10 ml aliquot (the aliquot amount depends on concentration in the sample) in a 250 ml Erlenmeyer flask and add 5 g KI crystals (4.5) and 5 ml 4N HCl (4.6). *Set 1:2*
Titrate with 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ solution (4.7), using starch indicator (4.9) as the end point approaches. Deduct a determined blank.

6.2.3 Calculation of Chlorine concentration

$$\text{wt. Cl}_2, \text{mg} = T_n \times N \times 35.5 \times \text{A.F.}$$

$$\text{conc. Cl}_2, \text{mg/m}^3 = \text{Cl}_2, \text{mg/V, m}^3$$

$$\text{ppm Cl}_2 = \text{Cl}_2, \text{mg/m}^3 \times 0.689$$

where: T_n = net ml $\text{Na}_2\text{S}_2\text{O}_3$ titer N = normality $\text{Na}_2\text{S}_2\text{O}_3$

35.5 = m.o.w Cl_2

V = sampled vol. m^3 S.T.P. A.F. = aliquot factor (250/
0.689 = conversion $\text{Cl}_2, \text{mg/}$
to

6.2.4 Total Chloride. Pipet a ¹⁰5-ml aliquot (aliquot amount depends on concentration in the sample) into a 250 Erlenmeyer flask and add 25 ml NH_4OH (4.10) to reduce chlorine to chloride. Add 5 glass beads and boil vigorously to remove excess ammonia. Add 3 drops phenolphthalein indicator (4.12) and H_2SO_4 (4.2) until just acid. Add a few drops of K_2CrO_4 indicator (4.4) and titrate with 0.1N AgNO_3 (4.8) to the first color change from yellow. Deduct a determined blank.

6.2.5 Calculation of Total Chloride as HCl. The same calculation procedure as in 6.1.5 is used with the possible exception of the aliquot factor. In the example used a 5 ml aliquot of the total 250 ml sample solution is used. The factor therefore would be 250/5 or 50.

6.2.6 Calculation of Hydrogen Chloride. Convert the wt. of chlorine found in 6.2.3 to its HCl equivalent and subtract from the total chloride weight found in 6.2.5.

conversion Cl_2 to HCl: $\text{Cl}_2, \text{mg} \times 1.03 = \text{HCl}, \text{mg}$

where: $\frac{\text{m.e.w. HCl (36.5)}}{\text{m.e.w. Cl}_2 (35.5)} = 1.03$

HCl in sample, mg = Total Chloride as HCl, mg - HCl equiv. of chlorine, mg

Concentration HCl, $\text{mg}/\text{m}^3 = \text{HCl}, \text{mg}/\text{V}, \text{m}^3$

Concentration HCl, ppm = $\text{HCl}, \text{mg}/\text{m}^3 \times 0.670$

where: V = sampled volume m^3 S.T.P.

0.670 = conversion $\text{mg HCl}/\text{m}^3$ to ppm HCl

6.3 See appendix 8.1 for use of specific ion electrode

7. Report of Data

Date of test, sample number, sampling location

Operation, alloy processed, duration of fluxing

Average stack temperature, $^{\circ}\text{C}$

Stack gas volume, $\text{m}^3/\text{min.}$ at S.T.P. (25°C and 760 torr.)

Results: Aerosols - total Al_2O_3 mg/m^3

HCl mg/m^3

Gas - Chlorine mg/m^3 and ppm

HCl

Mass Emission - total mg/m^3