AP-42 Section Number: 12.15

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Title: Source Testing at a Lead Acid Battery Manufacturing Company- ESB, Canada, Ltd., Mississauga, Ontario

EPA-76-3

US EPA

1976

Report No. 76-BAT-3

STORAGE BATTERY
PRODUCTION
AP-42 Section 7.15
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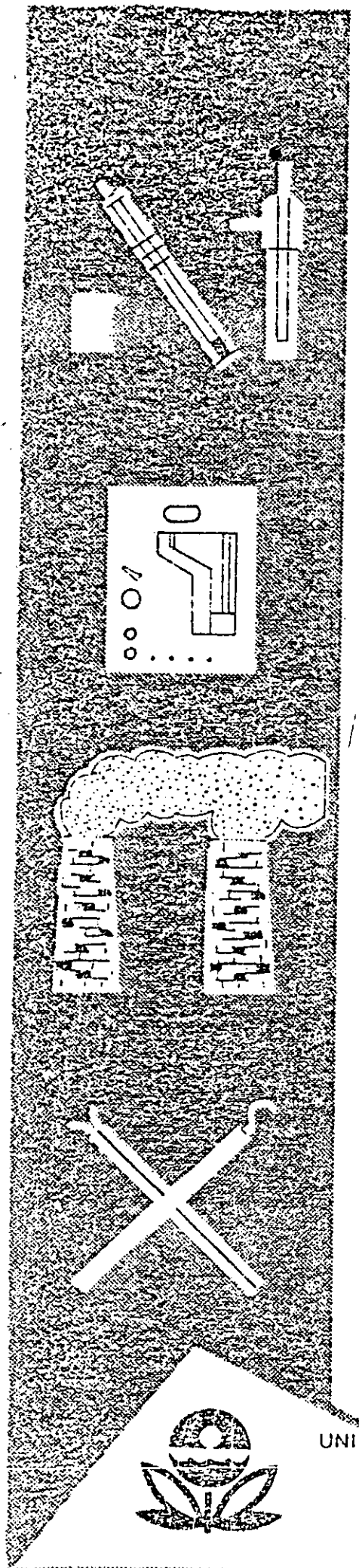
5



AIR POLLUTION EMISSION TEST

ESB CANADA LIMITED

MISSISSAUGA, ONTARIO

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

SOURCE TESTING
AT A
LEAD ACID BATTERY MANUFACTURING COMPANY

ESB Canada Limited
Mississauga, Ontario

Test Conducted By
Monsanto Research Corporation
Dayton, Ohio

August 16-20, 1976

Report Prepared by
Robert Martin
Environmental Protection Specialist
U.S. Environmental Protection Agency
Research Triangle Park, N. C.

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I. INTRODUCTION

During the week of August 16, 1976, testing was conducted by Monsanto Research, under contract by the Environmental Protection Agency, on two control devices at the ESB Canada Limited manufacturing plant in Mississauga, Canada. In addition, sampling was conducted to verify the reproducibility of a modified method of sample collection applicable to this industry.

The sources of interest were those from the three-process operation which are basically the assembly process and the ball mill process where lead oxide is produced from lead ingots or pigs.

The emissions of interest were lead concentration, visible emissions, particle size, and trace metals.

This report contains the data collected along with a description of the process.

II. SUMMARY AND DISCUSSION OF RESULTS

Pb Sampling and Results

Please refer to Figure 2, Page 17, in order to visualize the location of sampling points in relation to the process flow. Table I, Page 4, summarizes the results of sampling for lead concentration at these locations.

Sampling was performed using Method 22 as explained in the analytical section of this report. This method is a modification of Method 5 incorporating dilute (0.1N) nitric acid in the impingers and placing the filter behind the impinger train. On the outlets (points A & D) two sampling trains were run simultaneously in order to obtain data verifying the reproducibility of this sampling method. Previous sampling at another lead acid battery facility had been conducted to compare this method with Method 5 and those results supported the modification.

(Baghouse Controlling Three Process Operation, Points C and D) - The inlet (point C) and outlet (point D) were sampled simultaneously during periods of normal process operation. As seen on the data summary, the inlet concentrations are much higher than the outlet indicating good efficiency across the baghouse. Bear in mind that these are lead concentrations and should not be confused with particulate loadings.

Also of interest is the catch as divided by impinger and filter. These data tend to substantiate our previous findings that the impingers capture the majority of the lead and emissions.

(PbO Mill - Point A) - Three simultaneous dual tests were conducted on the baghouse controlling the PbO process. These results can also be seen on

the summary. The emissions were somewhat higher than those from the three process operation but this was as expected due to the nature of the process. The inlet was not sampled because of unsuitability of duct configuration but it can safely be assumed that these emissions are quite high as this baghouse is a product recovery device rather than an industrial hygene ventilation system as is Point D.

Visible Emissions

Visible emissions were recorded by a qualified observer during each test and no emissions were observed during these periods.

Particle Size - Particle size samples were taken at each of the test sites. These data can be seen in the test section concerning particle sizing. Inlet, Point C, was sampled using a Brink's collector and outlets A and D were sampled using Anderson apparatus.

Trace Metals

Trace metals samples were obtained at points A and D in order to obtain a qualitative sample that would indicate if any other metals were present in significant amounts. These results were negative.

Table 1. Pb - RESULTS - ESB CANADA LIMITED, MISSISSAUGA, ONTARIO

		Run	VM STD	*µg/Pb A FH	B Filt.	C Total	µg/CF Ax50 / VM	Std. Bx50 / VM	Cx50 / CF
Three Process PbO Mill	inlet	1C	40.99	695	1.3	696.3	848	1.6	849
		2C	42.13	800	1.0	801	949	1.2	950
		3C	42.23	475	1.0	476	562	1.2	564
	outlet	1D1	35.46	5.4	-	5.4	7.6	-	7.6
		1D2	35.32	7.2	1.7	8.9	10.2	2.4	12.6
		2D1	34.43	2.2	-	2.2	3.2	-	3.2
		2D2	33.694	.9	.5	1.4	1.3	.7	2.1
		3D1	34.994	6.4	-	6.4	9.1	-	9.1
		3D2	33.741	.8	-	.8	1.2	-	1.2
		4A1	30.74	24.8	1.9	26.7	40.3	3.1	43.4
		4A2	34.48	22	-	22	32.0	-	32.0
		5A1	31.33	24.0	.3	24.3	38.3	.5	38.8
		5A2	35.31	45	.5	45.5	63.7	.7	64.4
		6A1	31.60	22.4	-	22.4	35.4	-	35.4
		6A2	35.32	22.4	-	22.4	31.7	-	31.7

*The numbers presented in this column are the results of analysis of an aliquot of 1/50 of the total sample.

PARTICULATE SUMMARY IN ENGLISH UNITS

DESCRIPTION	UNITS	1C	2C	3C	AVERAGE
DATE OF RUN		8-18-76	8-18-76	8-18-76	
Stack Area	FT2	4.909	4.909	4.909	
Net Time of Run	MIN	60.0	60.0	60.0	
Barometric Pressure	IN.HG	29.96	29.91	29.91	
Avg Orifice Pres Drop	IN.H_2O	1.580	1.670	1.670	
Vol Dry Gas-Meter Cond	DCF	42.21	43.92	44.09	
Avg Gas Meter Temp	DEG.F	88.1	94.4	94.8	
Vol Dry Gas-Std Cond	DSCF	41.02	42.20	42.27	
Total H_2O Collected	ML	16.0	15.1	13.9	
Vol H_2O Vapor-Std Cond	SCF	.76	.72	.66	
Percent Moisture by Vol		1.8	1.7	1.5	
Mole Fraction Dry Gas		.982	.983	.985	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.95	28.82	28.82	
Molecular Wt-Stk Gas		28.75	28.64	28.65	
Avg Stack Temperature	DEG.F	81.0	85.6	87.7	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	IN.HG	29.59	29.54	29.54	
Avg Stack Gas Velocity	FPS	63.529	65.580	65.726	64.948
Stk Flowrate, Dry, Std CN	DSCFM	17795.	18214.	18207.	18072.
Actual Stack Flowrate	ACFM	18710.	19317.	19357.	19128.
Percent Ioskinetic		100.0	100.5	100.7	100.4

PARTICULATE SUMMARY IN ENGLISH UNITS

DESCRIPTION	UNITS	4A1	4A2	5A1	AVERAGE
DATE OF RUN		8-19-76	8-19-76	8-19-76	
Stack Area	FT2	.994	.994	.994	
Net Time of Run	MIN	64.0	64.0	64.0	
Barometric Pressure	IN.HG	29.97	29.97	29.97	
Avg Orifice Pres Drop	IN.H_2O	.805	.943	.807	
Vol Dry Gas-Meter Cond	DCF	31.34	35.02	31.94	
Avg Gas Meter Temp	DEG.F	81.4	80.1	81.9	
Vol Dry Gas-Std Cond	DSCF	30.79	34.49	31.34	
Total H_2O Collected	ML	16.0	15.2	13.7	
Vol H_2O Vapor-Std Cond	SCF	.76	.72	.65	
Percent Moisture by Vol		2.4	2.0	2.0	
Mole Fraction Dry Gas		.976	.980	.980	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.82	28.82	28.82	
Molecular Wt-Stk Gas		28.56	28.60	28.60	
Avg Stack Temperature	DEG.F	143.0	143.0	145.0	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	IN.HG	29.93	29.93	29.93	
Avg Stack Gas Velocity	FPS	41.246	44.965	42.093	42.768
Stk Flowrate, Dry, Std CN	DSCFM	2110.	2309.	2155.	2191.
Actual Stack Flowrate	ACFM	2460.	2682.	2510.	2551.
Percent Ioskinetic		99.9	102.2	99.6	100.5

PARTICULATE SUMMARY IN ENGLISH UNITS

DESCRIPTION	UNITS	5A2	6A1	6A2	AVERAGE
DATE OF RUN		8-19-76	8-19-76	8-19-76	
Stack Area	FT2	.994	.994	.994	
Net Time of Run	MIN	64.0	64.0	64.0	
Barometric Pressure	IN.HG	29.97	29.97	29.97	
Avg Orifice Pres Drop	IN.H_2O	.995	.837	.986	
Vol Dry Gas-Meter Cond	DCF	35.87	32.25	35.83	
Avg Gas Meter Temp	DEG.F	80.2	82.5	79.5	
Vol Dry Gas-Std Cond	DSCF	35.33	31.61	35.34	
Total H_2O Collected	ML	14.7	8.0	13.0	
Vol H_2O Vapor-Std Cond	SCF	.70	.38	.62	
Percent Moisture by Vol		1.9	1.2	1.7	
Mole Fraction Dry Gas		.981	.988	.983	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.82	28.82	28.82	
Molecular Wt-Stk Gas		28.61	28.69	28.63	
Avg Stack Temperature	DEG.F	144.0	146.0	144.0	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	IN.HG	29.93	29.93	29.93	
Avg Stack Gas Velocity	FPS	46.199	41.983	46.047	44.743
Stk Flowrate, Dry, Std CN	DSCFM	2371.	2164.	2369.	2301.
Actual Stack Flowrate	ACFM	2755.	2504.	2746.	2668.
Percent Ioskinetic		102.0	100.0	102.1	101.3

PARTICULATE SUMMARY IN ENGLISH UNITS

DESCRIPTION	UNITS	1D1	1D2	2D2	AVERAGE
DATE OF RUN		8-18-76	8-18-76	8-18-76	
Stack Area	FT2	4.909	4.909	4.909	
Net Time of Run	MIN	60.0	60.0	60.0	
Barometric Pressure	IN.HG	29.96	29.91	29.91	
Avg Orifice Pres Drop	IN.H_2O	1.020	1.100	1.020	
Vol Dry Gas-Meter Cond	DCF	35.29	35.07	35.14	
Avg Gas Meter Temp	DEG.F	,68.9	67.2	81.7	
Vol Dry Gas-Std Cond	DSCF	35.49	35.33	34.45	
Total H_2O Collected	ML	11.0	12.0	11.4	
Vol H_2O Vapor-Std Cond	SCF	.52	.57	.54	
Percent Moisture by Vol		1.4	1.6	1.5	
Mole Fraction Dry Gas		.986	.984	.985	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.82	28.82	28.82	
Molecular Wt-Stk Gas		28.65	28.65	28.65	
Avg Stack Temperature	DEG.F	81.7	81.7	82.5	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	IN.HG	29.89	29.84	29.84	
Avg Stack Gas Velocity	FPS	74.810	72.458	74.777	74.015
Stk Flowrate, Dry, Std CN	DSCFM	21217.	20487.	21122.	20942.
Actual Stack Flowrate	ACFM	22033.	21340.	22033.	21799.
Percent Ioskinetic		101.8	105.0	99.3	102.0

PARTICULATE SUMMARY IN ENGLISH UNITS

DESCRIPTION	UNITS	2D2	3D1	3D2	AVERAGE
DATE OF RUN		8-18-76	8-18-76	8-18-76	
Stack Area	FT2	4.909	4.909	4.909	
Net Time of Run	MIN	60.0	60.0	60.0	
Barometric Pressure	IN.HG	29.91	29.91	29.91	
Avg Orifice Pres Drop	IN.H_2O	1.070	1.030	1.690	
Vol Dry Gas-Meter Cond	DCF	34.41	35.47	34.36	
Avg Gas Meter Temp	DEG.F	82.0	77.5	80.6	
Vol Dry Gas-Std Cond	DSCF	33.71	35.05	33.81	
Total H_2O Collected	ML	14.4	14.5	16.2	
Vol H_2O Vapor-Std Cond	SCF	.68	.69	.77	
Percent Moisture by Vol		2.0	1.9	2.2	
Mole Fraction Dry Gas		.980	.981	.978	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.80	28.82	28.82	
Molecular Wt-Stk Gas		28.58	28.61	28.58	
Avg Stack Temperature	DEG.F	89.0	88.0	91.0	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	IN.HG	29.84	29.84	29.84	
Avg Stack Gas Velocity	FPS	71.791	71.752	72.457	72.000
Stk Flowrate, Dry, Std CN	DSCFM	19949.	19987.	20013.	19983.
Actual Stack Flowrate	ACFM	21144.	21132.	21340.	21205.
Percent Ioskinetic		102.9	106.7	102.8	104.1

PARTICULATE SUMMARY IN METRIC UNITS

DESCRIPTION	UNITS	1C	2C	3C	AVERAGE
DATE OF RUN		8-18-76	8-18-76	8-18-76	
Stack Area	M2	.456	.456	.456	
Net Time of Run	MIN	60.0	60.0	60.0	
Barometric Pressure	MM.HG	760.98	759.71	759.71	
Avg Orifice Pres Drop	MM.H_2O	40.132	42.418	42.418	
Vol Dry Gas-Meter Cond	DM3	1.20	1.25	1.25	
Avg Gas Meter Temp	DEG.C	31.2	34.7	34.9	
Vol Dry Gas-Std Cond	ONM3	1.16	1.19	1.20	
Total H_2O Collected	ML	16.0	15.1	13.9	
Vol H_2O Vapor-Std Cond	NM3	.02	.02	.02	
Percent Moisture by Vol		1.8	1.7	1.5	
Mole Fraction Dry Gas		.982	.983	.985	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.95	28.82	28.82	
Molecular Wt-Stk Gas		28.75	28.64	28.65	
Avg Stack Temperature	DEG.C	27.2	29.8	30.9	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	MM.HG	751.59	750.32	750.32	
Avg Stack Gas Velocity	M/S	19.364	19.992	20.033	19.796
Stk Flowrate, Dry, Std CN	DNM3/M	504.	516.	516.	512.
Actual Stack Flowrate	AM3/M	530.	547.	548.	542.
Percent Ioskinetic		100.0	100.5	100.7	100.4

PARTICULATE SUMMARY IN METRIC UNITS

DESCRIPTION	UNITS	4A1	4A2	5A1	AVERAGE
ATE OF RUN		8-19-76	8-19-76	8-19-76	
tack Area	M2	.092	.092	.092	
t Time of Run	MIN	64.0	64.0	64.0	
Barometric Pressure	MM.HG	761.24	761.24	761.24	
vg Orifice Pres Drop	MM.H_2O	20.439	23.955	20.498	
l Dry Gas-Meter Cond	DM3	.89	.99	.90	
Avg Gas Meter Temp	DEG.C	27.4	26.7	27.7	
Vol Dry Gas-Std Cond	DNM3	.87	.98	.89	
tal H_2O Collected	ML	16.0	15.2	13.7	
l H_2O Vapor-Std Cond	NM3	.02	.02	.02	
Percent Moisture by Vol		2.4	2.0	2.0	
le Fraction Dry Gas		.976	.980	.980	
rcent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
rcent CO by Vol, Dry		.0	.0	.0	
rcent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.82	28.82	28.82	
Molecular Wt-Stk Gas		28.56	28.60	28.60	
g Stack Temperature	DEG.C	61.7	61.7	62.8	
et Sampling Points		1	1	1	
Stack Pressure, Absolute	MM.HG	760.22	760.22	760.22	
g Stack Gas Velocity	M/S	12.572	13.705	12.830	13.036
k Flowrate, Dry, Std CN	DNM3/M	60.	65.	61.	62.
Actual Stack Flowrate	AM3/M	70.	76.	71.	72.
rcent Ioskinetic		99.9	102.2	99.6	100.5

PARTICULATE SUMMARY IN METRIC UNITS

DESCRIPTION	UNITS	5A2	6A1	6A2	AVERAGE
DATE OF RUN		8-19-76	8-19-76	8-19-76	
Stack Area	M2	.092	.092	.092	
Net Time of Run	MIN	64.0	64.0	64.0	
Barometric Pressure	MM.HG	761.24	761.24	761.24	
Avg Orifice Pres Drop	MM.H_2O	25.273	21.260	25.044	
Vol Dry Gas-Meter Cond	DM3	1.02	.91	1.01	
Avg Gas Meter Temp	DEG,C	26.8	28.1	26.4	
Vol Dry Gas-Std Cond	DNM3	1.00	.90	1.00	
Total H_2O Collected	MI	14.7	8.0	13.0	
Vol H_2O Vapor-Std Cond	NM3	.02	.01	.02	
Percent Moisture by Vol		1.9	1.2	1.7	
Mole Fraction Dry Gas		.981	.988	.983	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.82	28.82	28.82	
Molecular Wt-Stk Gas		28.61	28.69	28.63	
Avg Stack Temperature	DEG.C	62.2	63.3	62.2	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	MM.HG	760.22	760.22	760.22	
Avg Stack Gas Velocity	M/S	14.081	12.796	14.035	13.638
Stk Flowrate, Dry, Std CN	DNM3/M	67.	61.	67.	65.
Actual Stack Flowrate	AM3/M	78.	71.	78.	76.
Percent Ioskinetic		102.0	100.0	102.1	101.3

PARTICULATE SUMMARY IN METRIC UNITS

DESCRIPTION	UNITS	1D1	1D2	2D1	AVERAGE
DATE OF RUN		8-18-76	8-18-76	8-18-76	
Stack Area	M2	.456	.456	.456	
Net Time of Run	MIN	60.0	60.0	60.0	
Barometric Pressure	MM.HG	760.98	759.71	759.71	
Avg Orifice Pres Drop	MM.H_2O	25.908	27.940	25.908	
Vol Dry Gas-Meter Cond	DM3	1.00	.99	1.00	
Avg Gas Meter Temp	DEG.C	20.5	19.6	27.6	
Vol Dry Gas-Std Cond	DNM3	1.00	1.00	.98	
Total H_2O Collected	ML	11.0	12.0	11.4	
Vol H_2O Vapor-Std Cond	NM3	.01	.02	.02	
Percent Moisture by Vol		1.4	1.6	1.5	
Mole Fraction Dry Gas		.986	.984	.985	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.82	28.82	28.82	
Molecular Wt-Stk Gas		28.66	28.65	28.65	
Avg Stack Temperature	DEG.C	27.6	27.6	28.1	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	MM.HG	759.21	757.94	757.94	
Avg Stack Gas Velocity	M/S	22.802	22.805	22.792	22.560
Stk Flowrate, Dry, Std CN	DNM3/M	601.	580.	598.	593.
Actual Stack Flowrate	AM3/M	624.	604.	624.	617.
Percent Ioskinetic		101.8	105.0	99.3	102.0

PARTICULATE SUMMARY IN METRIC UNITS

DESCRIPTION	UNITS	2D2	3D1	3D2	AVERAGE
DATE OF RUN		8-18-76	8-18-76	8-18-76	
Stack Area	M2	.456	.456	.456	
Net Time of Run	MIN	60.0	60.0	60.0	
Barometric Pressure	MM.HG	759.71	759.71	759.71	
Avg Orifice Pres Drop	MM.H_2O	27.178	26.162	42.926	
Vol Dry Gas-Meter Cond	DM3	.97	1.00	.97	
Avg Gas Meter Temp	DEG.C	27.8	25.3	27.0	
Vol Dry Gas-Std Cond	DNM3	.95	.99	.96	
Total H_2O Collected	ML	14.4	14.5	16.2	
Vol H_2O Vapor-Std Cond	NM3	.02	.02	.02	
Percent Moisture by Vol		2.0	1.9	2.2	
Mole Fraction Dry Gas		.980	.981	.978	
Percent CO_2 by Vol, Dry		.0	.0	.0	
Percent O_2 by Vol, Dry		.0	.0	.0	
Percent CO by Vol, Dry		.0	.0	.0	
Percent N_2 by Vol, Dry		100.0	100.0	100.0	
Molecular Wt-Dry Stk Gas		28.80	28.82	28.82	
Molecular Wt-Stk Gas		28.58	28.61	28.58	
Avg Stack Temperature	DEG.C	31.7	31.1	32.8	
Net Sampling Points		1	1	1	
Stack Pressure, Absolute	MM.HG	757.94	757.94	757.94	
Avg Stack Gas Velocity	M/S	21.882	21.870	22.085	21.946
Stk Flowrate, Dry, Std CN	DNM3/M	565.	566.	567.	566.
Actual Stack Flowrate	AM3/M	599.	598.	604.	600.
Percent Ioskinetic		102.9	106.7	102.8	104.1

LOCATION OF SAMPLING POINTS

Point C, See Figure 2

This point was located ideally more than 8 diameters upstream and more than 2 diameters downstream of obstructions. Ports were cut on a horizontal plane and vertically from the bottom allowing access from one section of scaffolding placed on the plant floor inside the building. Twelve sampling points were used and sampling was conducted for 5 minutes at each point for a total of 60 minutes per test.

Point D, See Figure 3

This point was also ideally located and sampling was conducted simultaneously with the above.

Point A

This sampling location was also ideally located and 12 points were selected for sampling. Four of these points fell within 1 inch of the stack wall so only 8 points were sampled. Each of these points was sampled for 8 minutes giving a total time of 64 minutes per test.

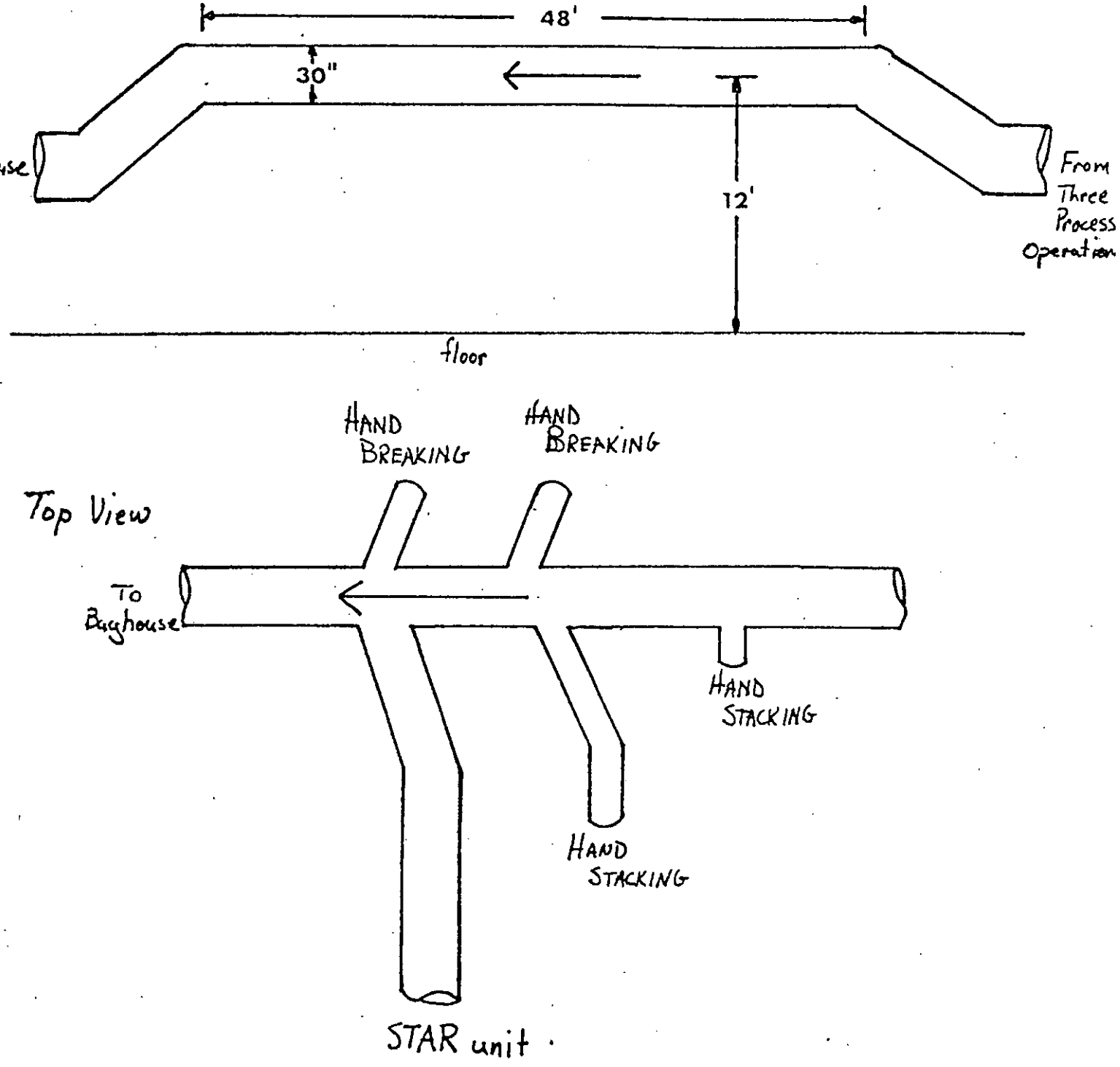

Figure 1.

Inlet to Baghouse and Three-Process Operation Exhaust

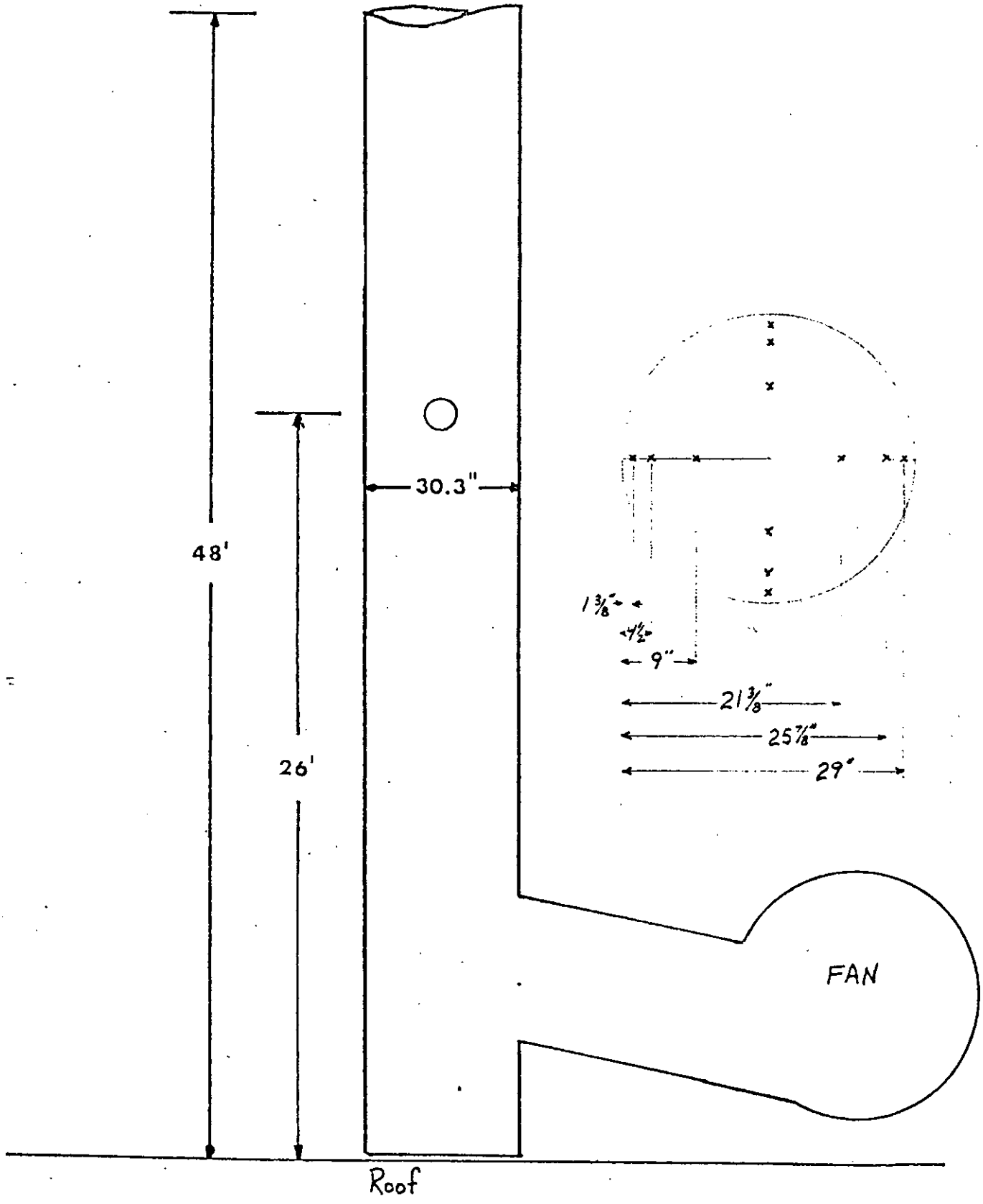

Figure 3.
Baghouse Stack (Three-Process)

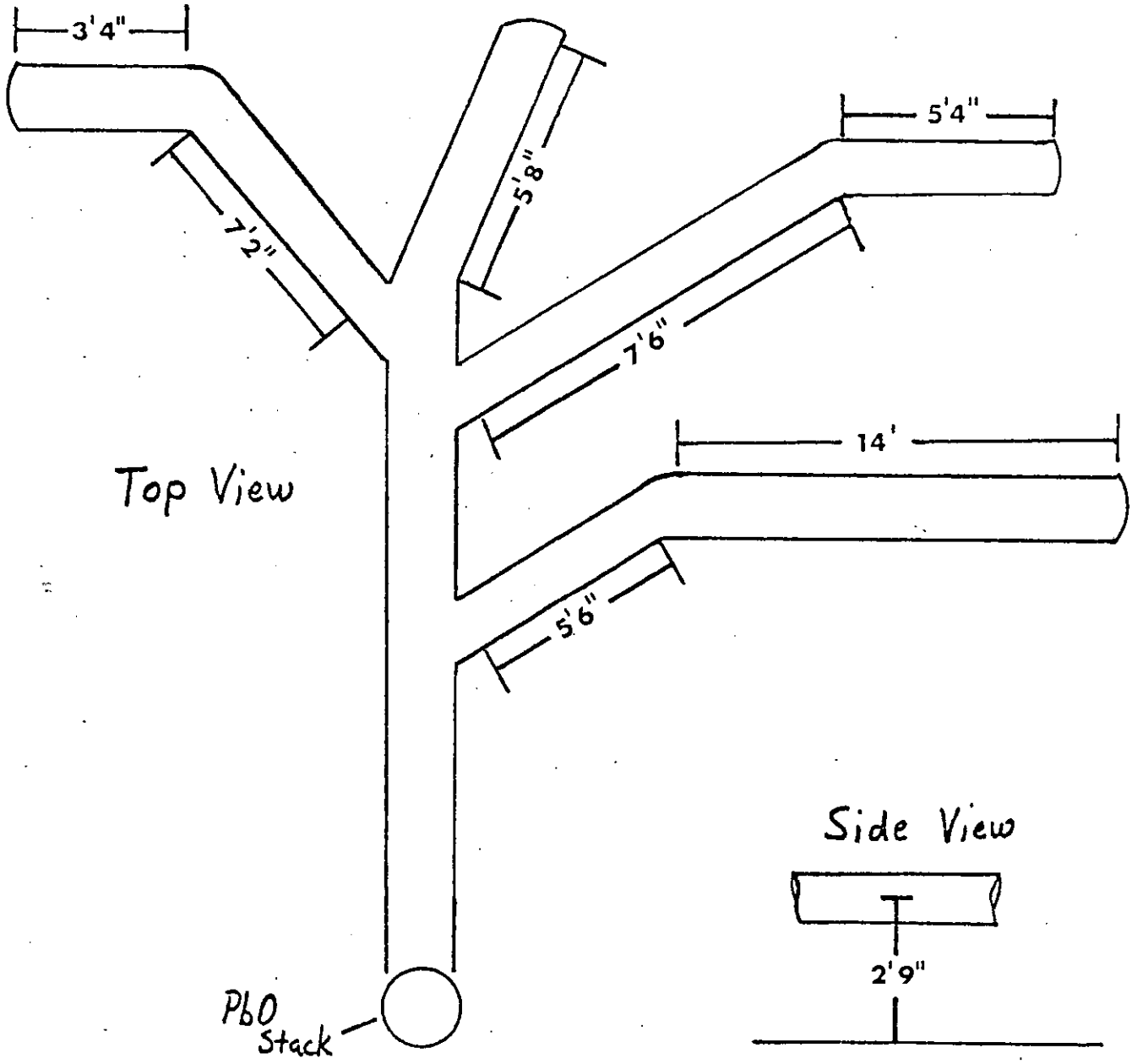

Figure 4.

Inlets to PbO Stack

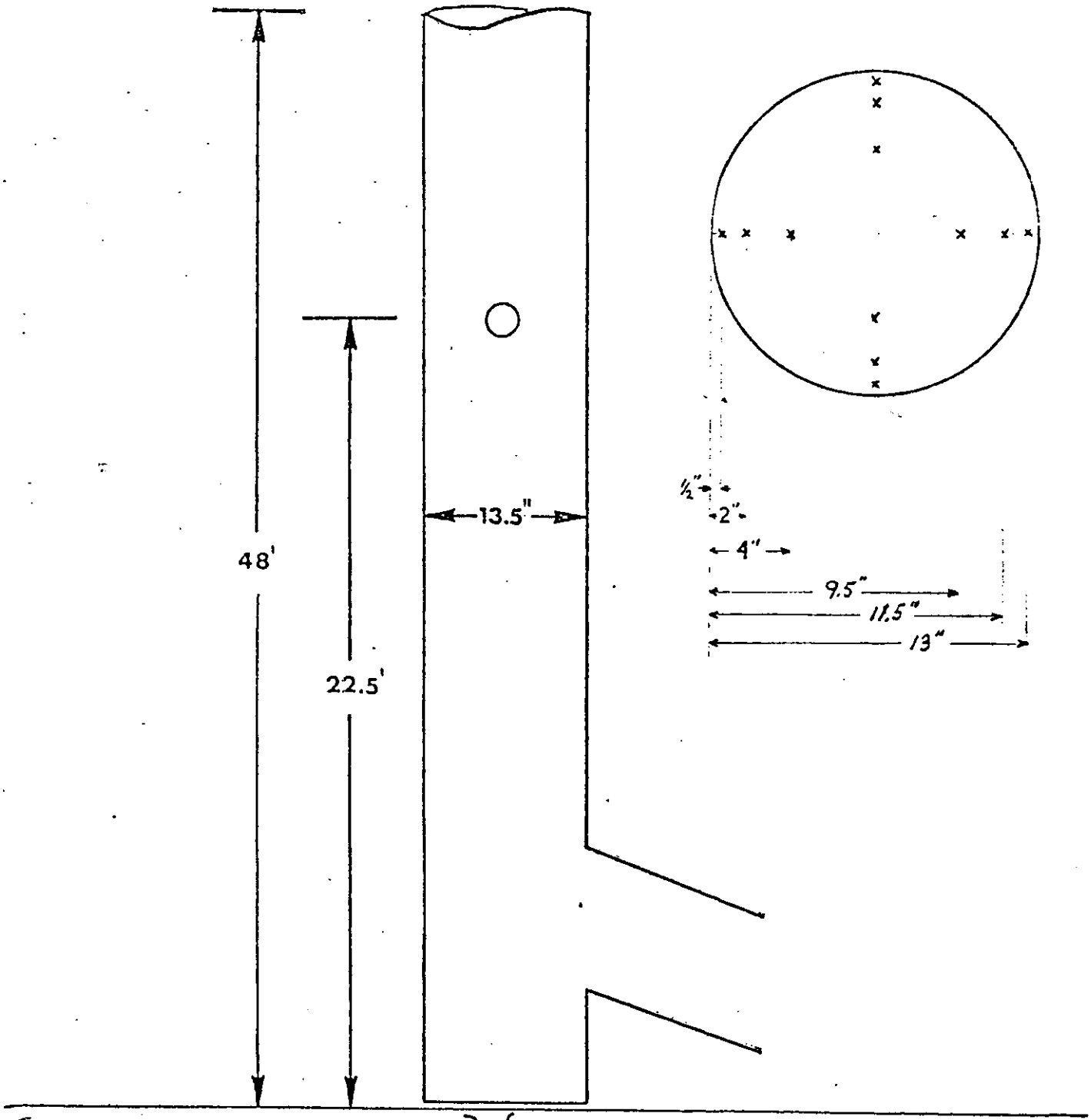

Figure 5.

PbO Stack

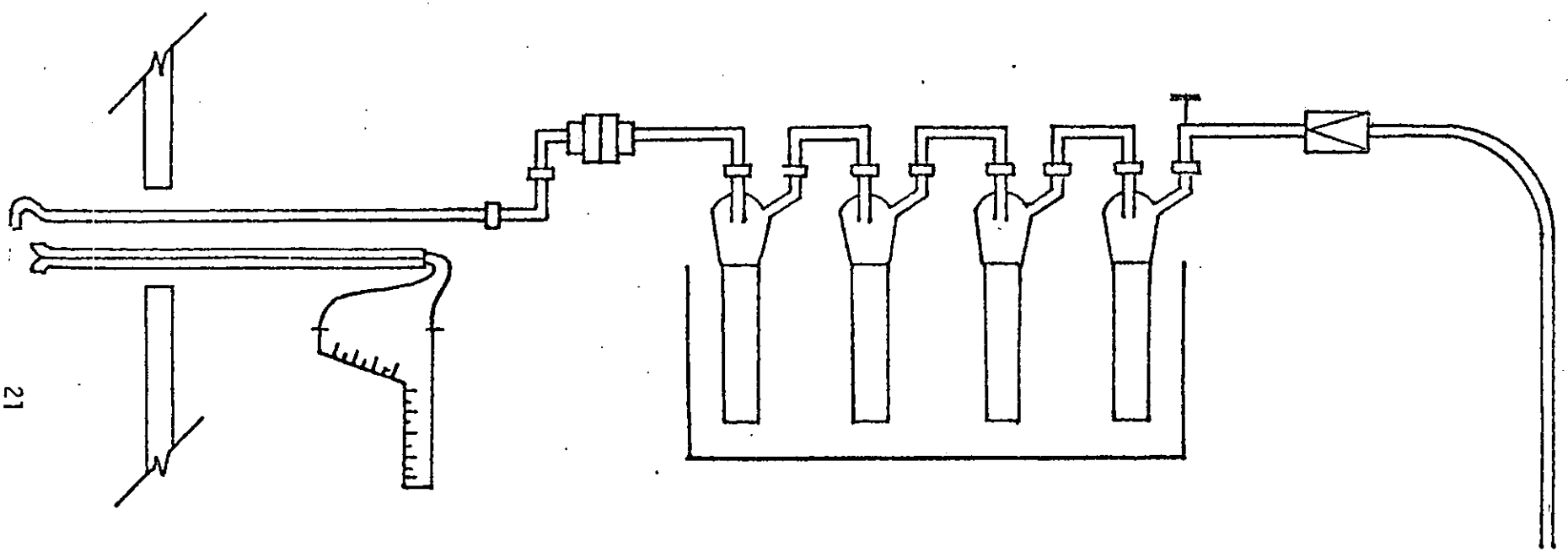

FIGURE 6

STANDARD MEHTOD 5 TRAIN

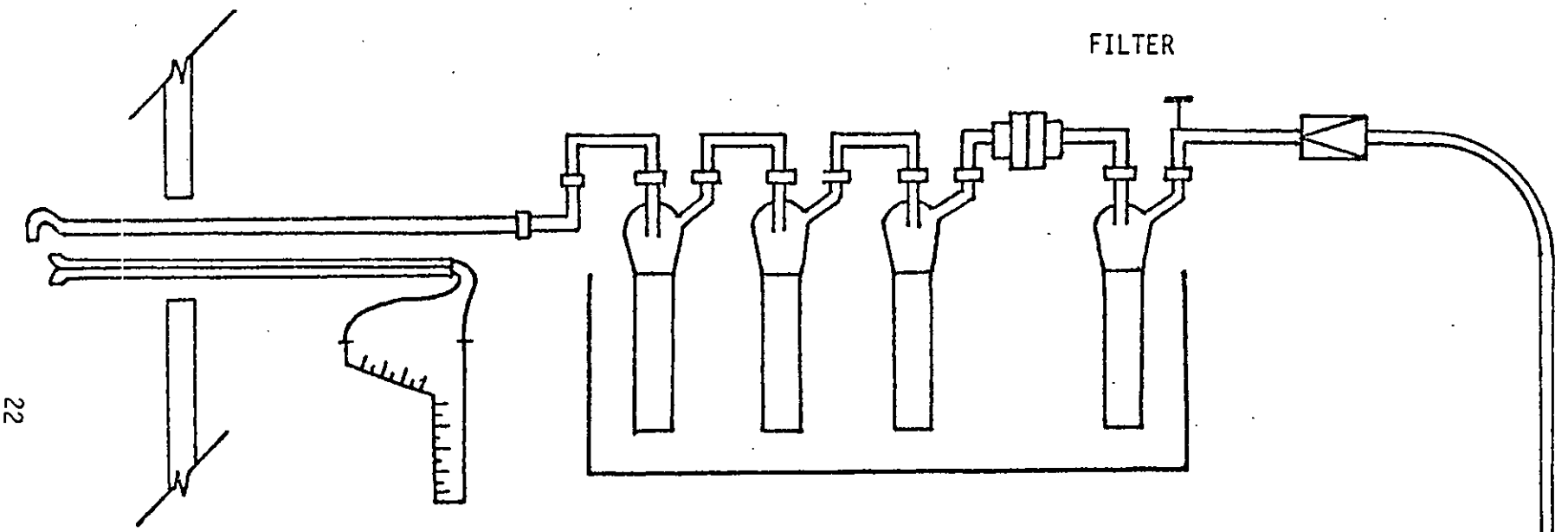

FIGURE 7

MODIFIED Pb TRAIN

PARTICLE SIZE RESULTS

The following pages contain particle size results as analyzed and reported by Monsanto Research. The outlet samples were collected using an Anderson sampling apparatus and the inlet of the baghouse (C) was collected with Brinks equipment.

Andersen Particle Size Data
ESB Canada Ltd.
Mississauga, Ontario

Run 1A

Stage	DPC (microns)	Wt. of Material (mg)	Wt. %	Cum. Wt. %
nozzle	>5.42	4.04	54.30	100.00
0	5.42	0.1	1.34	45.70
1	3.38	0.3	4.03	44.36
2	2.26	0.1	1.34	40.33
3	1.58	0.5	6.72	38.99
4	0.98	0.7	9.41	32.27
5	0.51	0.9	12.10	22.86
6	0.31	0.5	6.72	10.76
7	0.20	0.1	1.34	4.04
F	<0.20	0.2	2.70	2.70

Run 2A

Stage	DPC (microns)	Wt. of Material (mg)	Wt. %	Cum. Wt. %
nozzle	>5.32	3.74	78.90	100.00
0	5.32	0	0	21.10
1	3.38	0.2	4.22	21.10
2	2.23	0.1	2.11	16.88
3	1.57	0.2	4.22	14.77
4	0.98	0.2	4.22	10.55
5	0.50	0.1	2.11	6.33
6	0.31	0.1	2.11	4.22
7	0.20	0	0	2.11
F	<0.20	0.1	2.11	2.11

Run 3A

Stage	DPC (microns)	Wt. of Material (mg)	Wt. %	Cum. Wt. %
nozzle	>5.32	1.84	49.19	100.00
0	5.32	0	0	50.81
1	3.32	0.1	2.67	50.81
2	2.25	0.4	10.70	48.14
3	1.55	0.4	10.70	37.44
4	0.96	0.4	10.70	26.74
5	0.50	0	0	16.04
6	0.31	0.5	13.37	16.04
7	0.20	0.1	2.67	2.67
F	<0.20	0	0	0

Andersen Particle Size Data
ESB Canada Ltd.
Mississauga, Ontario

Run 1D

Stage	DPC (microns)	Wt. of Material (mg)	Wt. %	Cum. Wt. %
nozzle	>4.26	2.34	85.40	100.00
0	4.26	0.1	3.65	14.60
1	2.67	0.1	3.65	10.95
2	1.75	0	0	7.30
3	1.25	0.1	3.65	7.30
4	0.78	0	0	3.65
5	0.40	0	0	3.65
6	0.24	0	0	3.65
7	0.16	0	0	3.65
F	<0.16	0.1	3.65	3.65

Run 2D

Stage	DPC (microns)	Wt. of Material (mg)	Wt. %	Cum. Wt. %
nozzle	>4.20	2.54	86.40	100.00
0	4.20	0	0	13.60
1	2.62	0.1	3.40	13.60
2	1.74	0	0	10.20
3	1.23	0	0	10.20
4	0.77	0.2	6.80	10.20
5	0.39	0.1	3.40	3.40
6	0.24	0	0	0
7	0.16	0	0	0
F	<0.16	0	0	0

Run 3D

Stage	DPC (microns)	Wt. of Material (mg)	Wt. %	Cum. Wt. %
nozzle	>4.33	2.14	78.10	100.00
0	4.33	0.2	7.30	21.90
1	2.73	0	0	14.60
2	1.80	0	0	14.60
3	1.26	0	0	14.60
4	0.79	0.3	10.95	14.60
5	0.41	0	0	3.65
6	0.25	0	0	3.65
7	0.17	0.1	3.65	3.65
F	<0.17	0	0	0

4 NOVEMBER 1976

CASCADE IMPACTOR PARTICLE SIZE DISTRIBUTION FOR RUN 1C

INPUT VARIABLE	UNITS	INPUT DATA
SAMPLING TIME	MIN	30.0
PRESSURE DROP	IN HG	1.66
STATIC PRESSURE	IN H2O	-4.15
PARTICLE DENSITY	G/CC	9.53
BAROMETRIC PRESSURE	IN HG	29.99
GAS MOL WT		28.8
GAS TEMPERATURE	DEG F	156.0
GAS VISCOSITY	POISE	0.00016
GAS DENSITY	G/CC	0.00100

STATE	WT OF MATERIAL	DPC	MG/ACF	WT PCNT	CUM WT PCNT
CYCLONE	20.600		9.84	47.95	100.00
1	3.787	1.25	1.81	8.82	52.05
2	5.383	0.72	2.57	12.53	43.23
3	5.137	0.47	2.45	11.96	30.70
4	4.048	0.22	1.93	9.42	18.74
5	3.802	0.12	1.82	8.85	9.32
FILTER	0.200		0.10	0.47	0.47

4 NOVEMBER 1976

CASCADE IMPACTOR PARTICLE SIZE DISTRIBUTION FOR RUN 1C

INPUT VARIABLE	UNITS	INPUT DATA
SAMPLING TIME	MIN	30.0
PRESSURE DROP	CM HG	4.27
STATIC PRESSURE	CM H2O	-10.54
PARTICLE DENSITY	G/CC	9.53
BAROMETRIC PRESSURE	CM HG	76.17
GAS MOL WT		28.8
GAS TEMPERATURE	DEG C	68.9
GAS VISCOSITY	POISE	0.00018
GAS DENSITY	G/CC	0.00100

STAGE	WT OF MATERIAL	DPC	MG/ACM	WT PCNT	CUM WT PCNT
CYCLONE	20.600		0.28	47.95	100.00
1	3.787	1.25	0.05	8.82	52.05
2	5.383	0.72	0.07	12.53	43.23
3	5.137	0.47	0.07	11.96	30.70
4	4.048	0.22	0.05	9.42	18.74
5	3.802	0.12	0.05	8.85	9.32
FILTER	0.200		0.00	0.47	0.47

CASCADE IMPACTOR PARTICLE SIZE DISTRIBUTION FOR RUN 2C

INPUT VARIABLE	UNITS	INPUT DATA
SAMPLING TIME	MIN	20.0
PRESSURE DROP	IN HG	1.66
STATIC PRESSURE	IN H2O	-4.10
PARTICLE DENSITY	G/CC	9.53
BAROMETRIC PRESSURE	IN HG	29.99
GAS MOL WT		28.8
GAS TEMPERATURE	DEG F	188.0
GAS VISCOSITY	POISE	0.00018
GAS DENSITY	G/CC	0.00100

STATE	WT OF MATERIAL	DPC	MG/ACF	WT PCNT	CUM WT PCNT
CYCLONE	15.400		10.80	49.60	100.00
1	4.172	1.23	2.92	13.44	50.40
2	3.533	0.71	2.48	11.38	36.96
3	2.487	0.46	1.74	8.01	25.58
4	1.762	0.22	1.24	5.68	17.57
5	3.693	0.11	2.59	11.89	11.89
FILTER	0.000		0.00	0.00	0.00

4 NOVEMBER 1976

CASCADE IMPACTOR PARTICLE SIZE DISTRIBUTION FOR RUN 2C

INPUT VARIABLE	UNITS	INPUT DATA
SAMPLING TIME	MIN	20.0
PRESSURE DROP	CM HG	4.27
STATIC PRESSURE	CM H2O	-10.41
PARTICLE DENSITY	G/CC	9.53
BAROMETRIC PRESSURE	CM HG	76.17
GAS MOL WT		28.8
GAS TEMPERATURE	DEG C	86.7
GAS VISCOSITY	POISE	0.00018
GAS DENSITY	G/CC	0.00100

STAGE	WT OF MATERIAL	DPC	MG/ACM	WT PCNT	CUM WT PCNT
CYCLONE	15.400		0.31	49.60	100.00
1	4.172	1.23	0.08	13.44	50.40
2	3.533	0.71	0.07	11.38	36.96
3	2.487	0.46	0.05	8.01	25.58
4	1.762	0.22	0.03	5.68	17.57
5	3.693	0.11	0.07	11.89	11.89
FILTER	0.000		0.00	0.00	0.00

CASCADE IMPACTOR PARTICLE SIZE DISTRIBUTION FOR RUN 3C

INPUT VARIABLE	UNITS	INPUT DATA
SAMPLING TIME	MIN	30.0
PRESSURE DROP	IN HG	1.68
STATIC PRESSURE	IN H2O	-3.70
PARTICLE DENSITY	G/CC	9.53
BAROMETRIC PRESSURE	IN HG	29.99
GAS MOL WT		28.8
GAS TEMPERATURE	DEG F	160.0
GAS VISCOSITY	POISE	0.00016
GAS DENSITY	G/CC	0.00100

STATE	WT OF MATERIAL	DPC	MG/ACF	WT PCNT	CUM WT PCNT
CYCLONE	23.500		11.20	55.23	100.00
1	7.775	1.25	3.71	18.27	44.77
2	2.106	0.72	1.00	4.95	26.49
3	3.104	0.47	1.48	7.30	21.54
4	3.342	0.22	1.59	7.85	14.25
5	2.720	0.12	1.30	6.39	6.39
FILTER	0.000		0.00	0.00	0.00

CASCADE IMPACTOR PARTICLE SIZE DISTRIBUTION FOR RUN 3C

INPUT VARIABLE	UNITS	INPUT DATA
SAMPLING TIME	MIN	30.0
PRESSURE DROP	CM HG	4.27
STATIC PRESSURE	CM H2O	-9.40
PARTICLE DENSITY	G/CC	9.53
BAROMETRIC PRESSURE	CM HG	76.17
GAS MOL WT		28.8
GAS TEMPERATURE	DEG C	71.1
GAS VISCOSITY	POISE	0.00018
GAS DENSITY	G/CC	0.00100

STAGE	WT OF MATERIAL	DPC	MG/ACM	WT PCNT	CUM WT PCNT
CYCLONE	23.500		0.32	55.23	100.00
1	7.775	1.25	0.10	18.27	44.77
2	2.106	0.72	0.03	4.95	26.49
3	3.104	0.47	0.04	7.30	21.54
4	3.342	0.22	0.05	7.85	14.25
5	2.720	0.12	0.04	6.39	6.39
FILTER	0.000		0.00	0.00	0.00

CONTENTS

I. PROCESS DESCRIPTION AND OPERATION

1. GENERAL DESCRIPTION OF LEAD-ACID BATTERY MANUFACTURING

Battery manufacturing begins with two unrelated operations; grid casting and paste mixing. The grids are generally cast from lead hardened by the addition of between 5 and 12 percent antimony.

Casting techniques for grids vary with the alloy used, the type of molds, and mold preparation before casting. Grid casting machines can have melting pots attached directly to them or have a central pot furnace from which the molten lead is either pumped or fed by gravity. Lead alloy ingots are melted in these gas-fired lead pots at approximately 700°F. The produced grids are sent to the grid pasting operation.

The paste making operation, a batch-type process, takes place in either a muller, Day, or dough-type mixer. From 600 to 3,000 pounds of lead oxide is added to the mixer, water and sulfuric acid is then added, and the mixture is blended to form a stiff paste. Because of the exothermic conditions, mixers are usually water-jacketed and air-cooled to prevent excessive temperature build-up which causes the paste to become stiff and difficult to apply to the grids. Varying amounts of expander and other constituents are added

depending on whether a positive or negative paste batch is desired. The time of the mixing cycle is dependent on the type of mixer utilized. Mixing cycles range from 15 minutes up to an hour in duration.

Pasting machines force the lead sulfate paste into the interstices of the grid structure at production rates exceeding 200 plates per minute (the grids are called plates after the paste has been applied). The freshly pasted plates are transported by a horizontal chain through a temperature controlled, heated tunnel about 20 feet long, where the surface water is removed. This allows the plates to be stacked without sticking together. It is important that only the surface water be removed since the plates depend on moisture to produce the chemical reaction within the paste while curing --- very much similar to a cementitious process. The plates are cured for about 72 hours to increase plate strength.

Following the curing stage, the plates are normally sent to the three-process operation. This operation encompasses stacking, burning, and assembly. First, the plates are stacked in an alternating positive and negative block formation. Insulators are sandwiched between each plate to insulate the oppositely charged plates while permitting free ionic flow. These dividers are made from materials such as

wood, treated paper, plastics, or rubber. While machines have been designed which can stack the plates and separators automatically, hand stacking is not uncommon, even in some relatively large plants.

Leads (pronounced leeds) are welded to the tabs of each positive plate and each negative plate, fastening the assembly (element) together. This is the burning operation. An alternative to the welding or burning process is the "cast strap" process. In the latter, molten lead is poured around and between the plate tabs, thus forming the connection. Then a positive and negative terminal is welded to the element. The completed elements can go to either the wet or dry battery lines.

In the wet battery line, elements are placed within cases made of durable plastic or hard rubber. Covers equipped with openings and lead inserts are aligned so that the terminals project out of the inserts. The covers are sealed to the cases and the batteries are filled with dilute sulfuric acid and made ready for formation.

The only difference between the wet battery and the dry battery process is that, for dry batteries, the elements are formed prior to being placed in a sealed case. The dry batteries are shipped without acid. Sulfuric acid is then added to the battery at the point of use. This gives the

battery an indefinite shelf-life.

Formation is a chemical process wherein the inactive lead oxide-sulfate paste is converted into an active electrode. Formation is essentially an oxidation-reduction reaction wherein the positive plates are oxidized from lead oxide to lead peroxide, and the negative plates are reduced from lead oxide to metallic lead. This is accomplished by placing the unformed plates in a dilute sulfuric acid solution and connecting the positive plates to the positive pole of a dc source and the negative plates to the negative pole of the dc source.

When manufacturing wet lead-acid batteries it is common practice to place the cells into the battery cases, place the lid on the battery, and add sulfuric acid. The plates are then formed within the battery case itself. After formation, the spent acid is dumped from the batteries, new acid added, and a boost charge given to the battery. The unit is then ready for use and only requires decoration and manufacturer's markings.

The plates used in dry batteries are formed in one of several ways. Some plates are individually formed in tanks of sulfuric acid and then assembled. However, most dry batteries are made by assembling the plates into the elements beforehand. The completed elements are then formed in one

of two manners. First, the elements themselves can be placed into large tanks of sulfuric acid, electrically connected, and formed. Some companies place the assembled elements directly into the battery case. Thereafter, the formed elements are removed, the acid dumped, and the cases and elements rinsed and dried, reassembled and shipped dry.

Formation takes anywhere from one to four days. Most plants use a 36-to-48-hour forming cycle. The charging rate is high during the first 24 to 36 hours, then lower during the remaining 12 hours. The ampere rates are dependent upon the battery size. Figure 8 shows a typical battery plant flow diagram.

2. DESCRIPTION OF ESB's MISSISSAUGA PLANT

ESB's Mississauga plant has a normal operating output of 3500 batteries per day with a maximum of 4500 batteries per day.

The major operations employed are lead oxide production, grid casting, paste mixing, battery assembly (the three-process operation), and formation. Figure 9 illustrates the general flow of material through ESB's Mississauga plant.

The plant receives pure lead from an outside source and manufactures lead oxide by the ball mill process. There are two lead oxide production lines at the plant which operate 5 days per week, 24 hours per day. A typical feed rate for

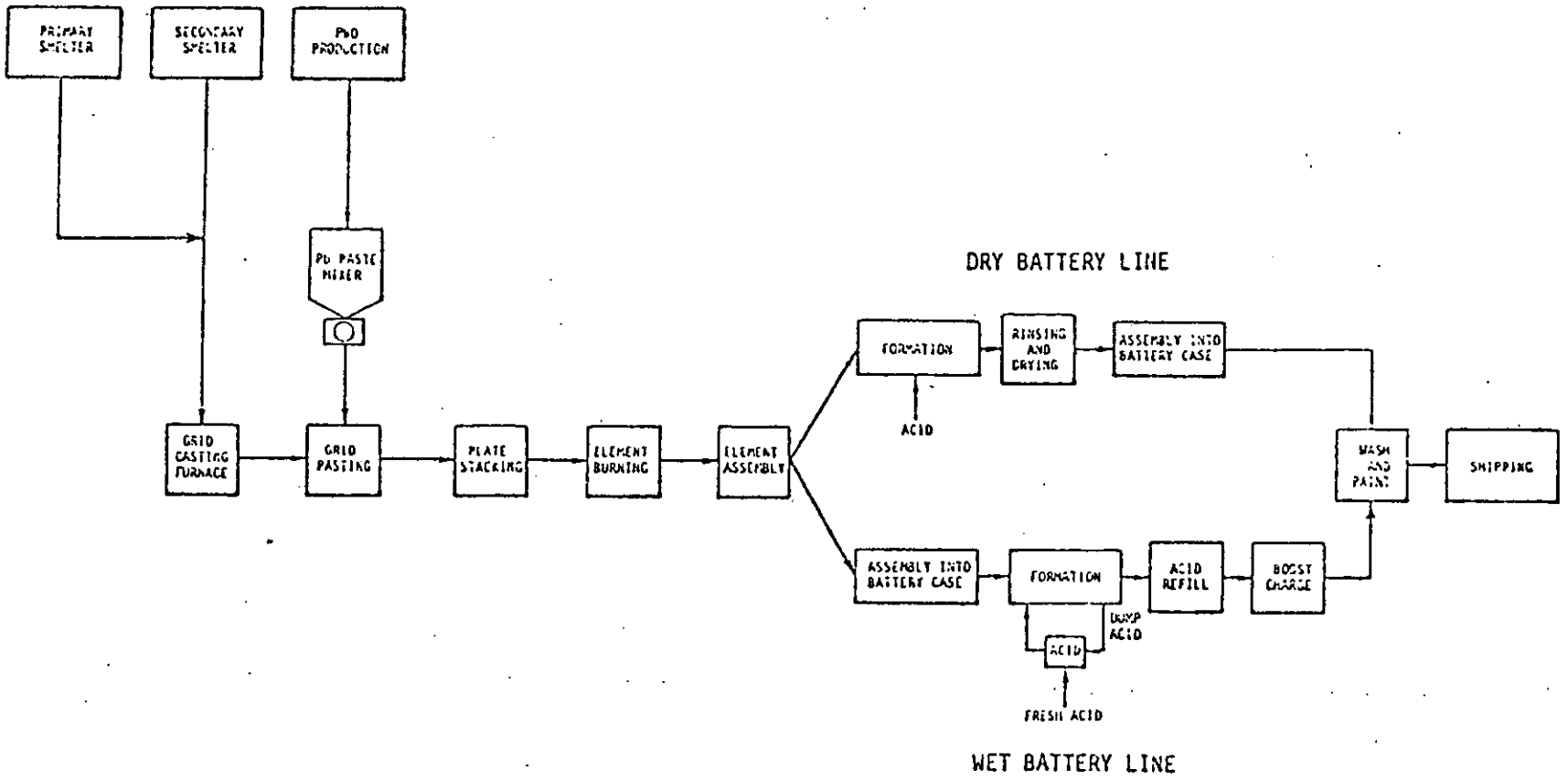

Figure 8. Process flow diagram - lead-acid battery manufacture.

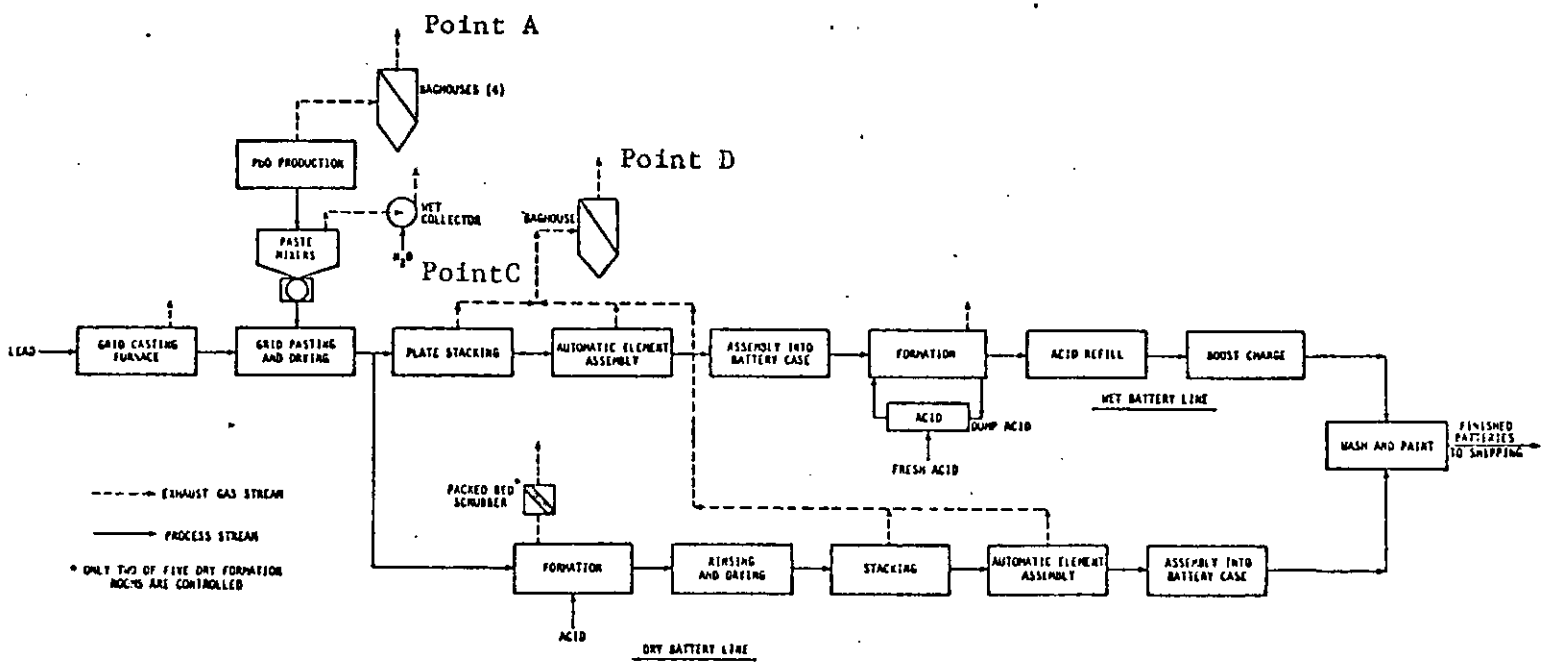

Figure 9. Production flow diagram, ESB Canada Ltd.

each PbO production line is 15 one-hundred pound lead pigs per hour. These pigs are fed into a Harding rotary mill which tumbles the pigs and forms lead oxide. After screening with a vibrating rotary screen, milling with a Raymond impact mill, and collecting the product with a cyclone, the PbO product consists of approximately 70 percent lead oxide and 30 percent lead. Each lead oxide production line has two baghouses which act mainly as control devices but also collect the product which is not retained by the cyclone separator. Water sprays maintain rotary mill temperatures around 410°F.

Production line 1 is controlled by two NORBLO two-compartment baghouses which each have air-to-cloth ratios of 2:1 with both compartments on line. One baghouse controls the screens which follow the rotary mill and the other controls the product recovery cyclone separator and the barrel filling station. The felted dacron bags are cleaned in each compartment every 15 minutes by shaking. During shaking the baghouses have a air-to-cloth ratio of 4:1. These units were rebagged in April 1976.

Lead oxide production line 2 is controlled by two Mikro-pulsaire baghouses (model no. 64S-6-POTR), each having air-to-cloth ratios of 4:1. One baghouse controls the screens following the rotary mill and the other controls the product recovery cyclone separator, a vibrating screen

following the cyclone, and the barrel filling station. The felted dacron bags are continuously pulse jet cleaned. These baghouses were rebagged in July, 1976.

All four baghouse exhausts are combined and released to the atmosphere through a 50 foot stack equipped with an opacity meter. If a control system should fail, the increased opacity of the exhaust activates an audible alarm. The meter recorder is normally set at 15 percent even though actual opacity is zero. Plant personnel report that this facilitates observance of meter fluctuations.

The grid casting facility consists of four machines. The cast grids are taken to the grid pasting machine where both positive and negative pastes are applied to the grids. After pasting, these plates are dried, slit, stacked, and formed.

The paste is produced by mixing dry lead oxide powder, water, and sulfuric acid in two 2000 lb/hr paste mixers. Each mixer is controlled by a separate low energy impingement-type wet collector designed for a 8-10 inch W.G. pressure drop at 2000 acfm.

The plates used in the dry battery production line are formed in vats of sulfuric acid. After charging, or forming, the plates are rinsed, slit, and stored.

A lug breaking step is required for dry formed plates. These lugs are merely used to support the plates in the

formation process. Once the plates are formed, these lugs are no longer needed so they are manually removed with a hammer at four lug breaking stations. These stations are ducted to the same baghouse which controls the three-process operation.

Plates for both wet and dry batteries are processed similarly in the three-process operations. The plates and separators are automatically or manually stacked in the proper sequence. ESB's Mississauga facility has four hand stacking stations and two automated stations, (a Reed stacker, and a Winkel stacker). Leads (pronounced leeds) and posts are cast on some of these stacks of plates to form elements. Two automatic element assembly units (cast-on-strap machines) are used. The balance of the stacks of plates are processed on a proprietary system called STAR. Here the stacks are inserted into specially constructed battery cases and the leads and posts are connected to the plate stacks at a burning station.

Lug breaking, hand and automatic stacking, automatic element assembly and STAR are controlled by a Mikro-pulsaire (Model No. 11F 26410) baghouse. Ducts from each of these processes join into a 30 inch duct and the baghouse exhaust exits via a 48 foot stack that is 30 inches in diameter. The baghouse is bagged with felted bags and is rated at 20,000 acfm with an air-to-cloth ratio of 6.5:1. The felted bags are continuously cleaned by pulse jet.

Following the three-process operation, batteries from the dry battery line are washed, decorated and sent to shipping while the batteries from the wet production line are sent to be formed. These batteries are filled with dilute sulfuric acid and formation is initiated. After the batteries are formed, the acid is replaced with fresh acid. The wet formed batteries are then given a boost charge, washed, decorated, and sent to shipping.

3. NORMAL PROCESS OPERATION

Source tests were performed on August 17 through 19, 1976. The three-process facility and lead oxide production facility were tested. Test sites included inlet and outlet of the baghouse controlling the three-process operation and the common outlet of the four baghouses controlling lead oxide production. These test sites are shown schematically in Figure 10.

a. Three Process Operation

Both dry and wet battery plates are processed at the three-process facility. Production requirements determine the amount of each that need to be stacked, made into elements, and inserted into battery cases. Normally 3500 batteries per day are produced but up to 4500 batteries per day can be assembled. This is equivalent to a normal and maximum daily element production of 21,000 and 27,000 elements, respectively.

There are four stations in this area for lug breaking

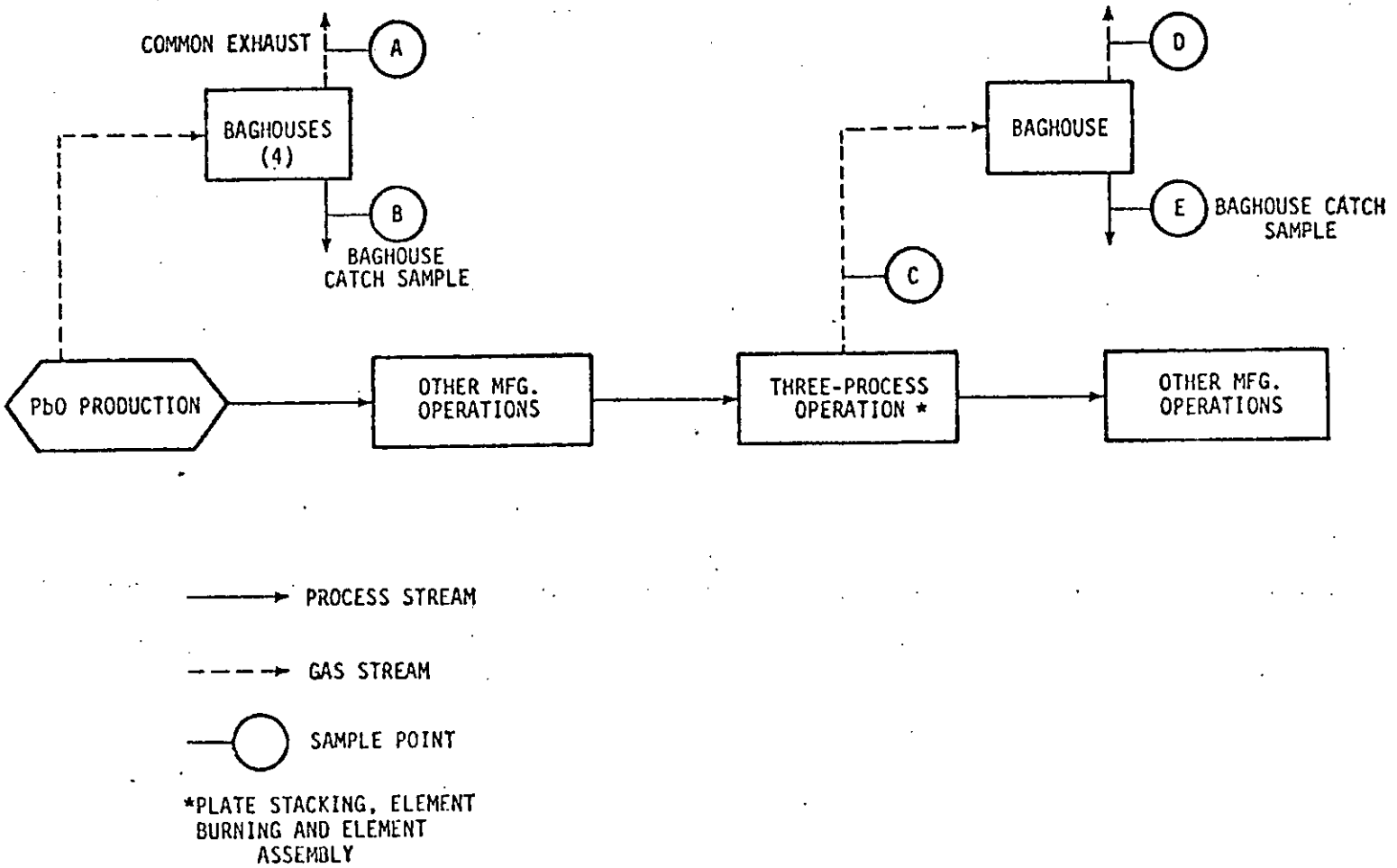

Figure 10. Source test locations for ESB Canada, Ltd., Mississauga, Canada

and four for hand stacking. However, only two hand stacking stations are normally used when the Reed (automatic) stacker is operating and lug breaking is performed only on an as-needed basis. Dampers isolate the work stations from the baghouse when they are not in use.

A Winkel stacker and a Reed stacker automatically stack about 2 plates per second during normal operation. These stackers operate at a constant rate with brief periods of downtime caused by jams (2 or 3 minutes) or changeover (5 to 10 minutes). Stacker changeover requires adjustment for plate size and for the required number of plates per element. Both stacking machines are operated independently. The stacks of plates are stored on skids until they are needed for the automatic element assembly units or STAR.

The operators of the two automatic element assembly lines work on a quota system. When they are ahead of their quota, the operation is shut down while the operators take a break of 10 to 20 minutes duration. Since the lines are completely independent, one crew can take an unscheduled break while the other crew continues work.

STAR is a proprietary battery assembly line at the plant that was partially operational as of September 1, 1976. The portions presently controlled by the three-process baghouse are two stations for inserting element assemblies into the battery case, and the element burning process.

b. Lead Oxide Production

The lead oxide production operation at ESB Mississauga is started up each Monday morning and shut down at the end of the afternoon shift on Friday. Lead pigs are fed to each lead oxide mill at a normal rate of 1500 pounds per hour (one 100-pound pig every four minutes). These pigs are tumbled in a rotary mill. The attrition and heat caused by the interraction of the lead pigs causes the formation of lead oxide particles. This reaction is performed at a temperature of approximately 400°F. The heat is controlled by water sprays which automatically operate when the rotary mill exceeds 410°F. The lead oxide product can be pneumatically conveyed to storage bins or barreled for shipment to other ESB facilities. The product is not weighed until it is used from the bins or it is shipped. This could be several days after production.

4. PROCESS OPERATION DURING SOURCE TESTS

a. Elements Assembly

All the source tests for the Mississauga plant's element assembly operations were done on August 18, 1976. Plate stacking was performed by the Reed and Winkel automatic stackers and at two of the four hand stacking stations. Enough plates were stacked during the shift to form 1845 twelve volt batteries. Battery assembly for the test shift was 1468 batteries which consisted of 926 and 542 twelve volt

batteries assembled on both automatic element assembly units and the STAR unit respectively. One or two lug breaking stations were operating at various times during the source tests. Records are not kept of the process throughput. A plant representative stated that the production data for August 18, 1976 indicates normal operation. While these figures as reported are after rejects, the same representative states that the rejects would amount to the equivalent of one or two batteries during one shift's production. Table 10 shows the time interval when each test was conducted.

Lead throughput during the tests ranged from 1160 lb/hr at the manual stacking stations to 2020 lb/hr at the Winkel stacker. The two cast-on-strap machines and the STAR unit had lead throughputs of 2080 and 1800 lb/hr respectively. These figures are based on estimates that plates are made up of 60 percent lead oxide paste and 40 percent lead grid. The paste contains approximately 70 percent lead oxide and 30 percent metallic lead. The process parameters during the source tests are summarized in Table 10. Since these figures have been prorated over seven hours of operation, and the tests performed the same day, the lead throughputs for each test are identical.

b. Lead Oxide Production

The source tests for the lead oxide production operation were done on August 19, 1976. During each test, one-hundred

Table 10. TIME INTERVALS FOR

THREE PROCESS OPERATION TESTS (August 18, 1976)

Test Number[a]	Time Interval (24-hour clock)
1C	0710-0834
1C1,1D2	0710-0821
2C	0950-1108
2D1,2D2	0950-1105
3C[b]	1140-1339
3D1,3D2	1238-1346

[a] Refer to Figure 10 for test site locations C and D.

[b] The source test was discontinued from 1155 to 1240 because process stopped for lunch break.

Table 11. LEAD THROUGHPUT RATE FOR ELEMENT ASSEMBLY SOURCE TESTS

Process	Lead Throughput, lb/hr[a]
Hand stacking	1160
Reed stacking	1800
Winkel stacking	2020
Stacking Total	4980
Automatic element assembly	2080
STAR	1800
Element assembly Total	3880

[a] Lead throughput was prorated from shift production records. A seven hour day was used to account for lunch, cleanup time, and breaks.

pound lead pigs were automatically fed to each ball mill at the rate of 15 pigs per hour (1500 lb/hr). The PbO product was pneumatically conveyed to bulk storage rather than barreled. No malfunctions occurred during the tests. Process parameters during the source tests are shown in Table .

The opacity meter was set at 22.5 percent as a zero point during the tests. This remained constant throughout. Baghouse pressure drops were within the normal range supplied by the plant prior to the source tests.

Table 12. TIME INTERVAL FOR EACH LEAD OXIDE MILL SOURCE TEST[a] (August 19, 1976)

Test Number[b]	Time Interval (24-hour clock)
4A1, 4A2	1050 - 1159
5A1, 5A2	1246 - 1355
6A1, 6A2	1446 - 1559

[a] Refer to Figure 10 for test site location.

[b] The lead throughput rate for each test was 1500 lb/hr.

METHOD 22--DETERMINATION OF LEAD EMISSIONS FROM THE MANUFACTURING OF LEAD BATTERIES

1. Principle, Applicability and Range

1.1 Principle. Particulate and gaseous lead emissions are isokinetically sampled from the source. The collected samples are digested in acid solution and analyzed by arsenic absorption spectrophotometry.

1.2 Applicability. This method is applicable for the determination of lead emissions from the manufacuring of lead batteries.

1.3 Range. The upper limit can be considerably extended by dilution. For a minimum analysis accuracy of $\pm$ 10 percent, a minimum lead mass of 50 µg should be collected in even sample fraction.

2. Apparatus

2.1 Sampling train. A schematic of the sampling train used in this method is shown in Figure 22-1. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD-0581; for changes from the APTD-0581 document and for allowable modifications to Figure 22-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

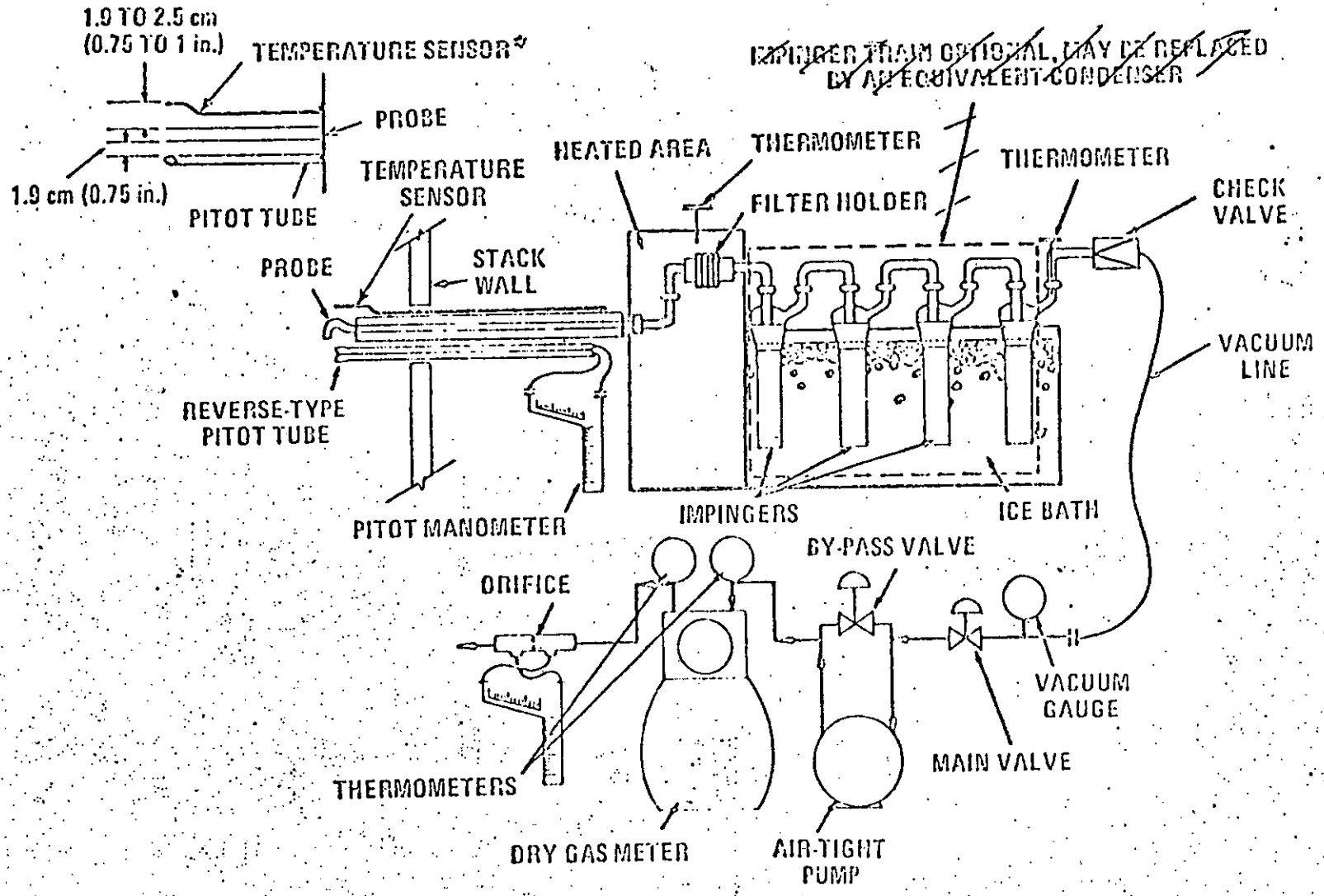

Figure 11. Particulate Sampling Train

*If difficulty is expected in inserting the temperature sensor-pitot tube-probe assembly into the stack due to spacing requirements, the temperature sensor may be located between the probe and pitot tube so that the tip of the temperature sensor is no closer than 5cm (2 in.) from the tip of the pitot tube.

Note: To be modified to show filter located between THIRD and FOURTH impingers

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Fig 11

2.1.1 Probe nozzle--Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be $\leq$ 30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise approved by the Administrator. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used subject to approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (1/8 in.) up to 1.27 cm (1/2 in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles, in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration secton.

2.1.2 Probe liner--Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of no greater than 120 $\pm$ 14° (248 $\pm$ 25°F) or no greater than such other temperature as specified by an applicable subpart of the standards. Since the actual temperature at the outlet of the probe is not monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 or calibrated according to the procedure outlined in APTD-0576 will be considered as acceptable.

When length limitations, i.e., greater than about 2.5 m (8.2 ft), are encountered, stainless steel (316) or *Incoloy 825*[1] (both of seamless tubing), or other materials as approved by the Administrator, may be used.

[1]Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.3 Pitot tube--Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, but not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the largest sized nozzle shall be used to set the free space.

The pitot tube must also meet the criteria specified in Method 2 and be calibrated according to the procedure in the calibration section of that method.

2.1.4 Differential pressure gauge--Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value or $\pm$ 0.013 mm (0.0005 in.), whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.1.5 Filter holder--Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then the filter holder may be stainless steel.

The holder design shall provide a positive seal against leakage from the outside or around the filter.

2.1.6 Impingers--Four Greenburg-Smith impingers connected in series with glass ball joint fittings. Impingers one, three, and four are modified by replacing the tip with a 1/2-inch ID glass tube extending to 1/2 inch from the flask bottom.

2.1.7 Metering system--Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter with 2 percent accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications in section 2 of this method are met. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

2.1.8 Barometer--Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.9 Gas density determination equipment--Temperature and pressure gauges and gas analyzer as described in Methods 2 and 3.

2.2 Sample recovery.

2.2.1 Probe liner and probe nozzle brushes--Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, nylon, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Glass wash bottles--One.

2.2.3 Glass sample storage containers--Chemically resistant, borosilicate glass bottles, for 0.1 N HNO_3 impinger and probe solutions and washes, 500 ml or 1,000 ml. Screw cap closures shall be teflon rubber-backed liners or of such construction so as to be leak free and prevent chemical attack from the 0.1 N HNO_3. (Narrow mouth glass bottles have been found to be less prone to leakage.) Other types of containers must be approved by the Administrator.

2.2.4 Petri dishes--For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5. Graduated cylinder and/or balance--To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdiyisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances are suitable for use here and in section 2.3.4.

2.2.6 Plastic storage containers--Air tight containers to store silica gel.

2.2.7 Funnel and rubber policeman--To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.3 Analysis.

2.3.1 Atomic absorption spectrophotometer with lead hollow cathode lamp and burner for air/acetylene flame.

2.3.2 Steam bath.

2.3.3 Hot plate.

2.3.4 Reflux condensers--24/40 $ to fit Erlenmeyer flasks.

2.3.5 Erlenmeyer flasks--125 ml 24/40 $.

2.3.6 Membrane filters--Millipore* SCWPO 4700 or equivalent.

2.3.7 Filtering apparatus--Millipore filtering unit, consisting of one of the assemblies shown in Figure 22-1.

2.3.8 Graduated cylinders--50 ml, 100 ml.

2.3.9 Volumetric flasks--100 ml.

3. Reagents

3.1 Sampling.

3.1.1 Filters--High purity glass fiber filters, without organic binder exhibiting at least 99.95 percent efficiency ($\leq$ 0.05 percent penetration) on 0.3 micron diotyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program is sufficient for this purpose. Filters shall be Gelman Spectro Grade, or equivalent, with lot Assay for Pb.

3.1.2 Silica gel--Indicating type, 6-16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received.

3.1.3 0.1N HNO_3--Prepared from reagent grade HNO_3 and distilled water. It may be desirable to run blanks prior to field use to eliminate a high blank on test samples. Prepare by diluting 6.5 ml of concentrated nitric acid (69 percent) to 1 liter with distilled water.

3.1.4 Crushed ice.

3.1.5 Stopcock grease--Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

3.2 Pretest preparation.

3.2.1 6N HNO_3--Prepared from reagent grade HNO_3 and distilled water. Prepare by diluting 390 ml of concentrated nitric acid (69 percent) to 1 liter with distilled water.

3.3 Sample recovery.

3.2.1 0.1N HNO_3--Prepared from reagent grade HNO_3 and distilled water (same solution as 3.1.3).

3.4 Analysis.

3.4.1 Deionized, distilled water.

3.4.2 Nitric acid, redistilled ACS reagent grade, concentrated.

3.4.3 30 percent HNO_3, v/v--Dilute 300 ml of redistilled concentrated nitric acid to 1 liter with deionized distilled water.

3.4.4 Stock lead standard, 100 µg Pb/ml--Dissolve 0.1598 g of reagent grade Pb $(NO_3)_2$ in about 700 ml of distilled water, add 10 ml redistilled concentrated HNO_3, and dilute to 1000 ml.

3.4.5 Lead standards.

3.4.5.1 Solution sample standards--Pipet aliquots of 1.0, 5.0, 10.0 and 20.0 ml of the 100 µg/ml stock lead standard into 100 ml volumetric flasks. Add 30 ml redistilled concentrated HNO_3 and dilute to volume with deionized distilled water. These working standards contain 1.0, 5.0, 10.0, and 20.0 µg Pb/ml, respectively. Additional standards at other concentratios should be prepared as needed. Use 30 percent HNO_3, v/v (Reagent 3.4.3) as the reagent blank.

3.4.5.2 Filter sample standards--Pipet aliquots of 1.0, 5.0, 10.0 and 20.0 ml of the 100 µg/ml stock lead standard into 125 ml Erlenmeyer flasks. Place a glass fiber filter (Section 3.1.1) cut into strips in each flask. Use filters from the same lot as those used for sampling. Add 30 ml redistilled concentrated nitric acid. Reflux for two hours and cool to room temperature. Rinse the condenser column with a small amount of deionized distilled water and remove the flask. Filter each standard through a millipore filter, rinse the filter and the remaining glass fiber mass with several small portions of deionized distilled water, and combine with the filtrate. Dilute each standard to 100 ml, using a 100 ml graduated cylinder.

3.4.6 Air--of a quality suitable for atomic absorption analysis.

3.4.7 Acetylene--of a quality suitable for atomic absorption analysis.

4. Procedure

4.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

4.1.1 Pretest preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576. In addition to those equipment cleaning procedures, rinse all sample exposed surfaces with 6N HNO_3 followed by distilled water.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks.

Label the shipping containers (glass or plastic petri dishes) and keep each filter in its respective container at all times except during sampling and weighing.

4.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10 percent, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 minutes or some greater time interval as specified by the Administrator and the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that 1/2 or an integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of 0.1N HNO_3 in each of the first two impingers, leave the third impinger empty, and place approximately 200-300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record weight of the silica gel and container to the nearest 0.5 g. Place the container in a clean place for later use in the sample recovery.

Using a tweezer or clean disposable surgical gloves, place the labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A O-ring when stack temperatures are less than 260° C (500° F) or an asbestos string gasket when temperatures are higher. The Viton A O-ring and asbestos string gasket are installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from both the pitot tube and probe nozzle to avoid interference with the gas flow.

Set up the train as in Figure 5-1, using, if necessary, a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. With approval from the Administrator a glass cyclone may be used between the probe and filter holder.

Place crushed ice around the impingers.

4.1.4 Leak check procedure--After the sampling train has been assembled, turn on and set the filter and probe heating system to the power required to reach temperatures of no greater than 10°C (18°F) in excess of the stack gas dewpoint, or such other other temperature as specified by an applicable subpart of the standards for the leak check. (If water condensation is not a problem the probe and/or filter heating system need not be used.) Allow time for the temperature to stabilize. If a Viton A O-ring or other leak free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum. (Note: A lower vacuum may be used provided that it is not exceeded during the test.) If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak check the train as above by first plugging the inlet to the glassware that connects the probe to the first impinger. Then connect the probe to the train and leak check at about 25 mm Hg (1 in. Hg) vacuum. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m^3/min. (0.02 cfm), whichever is less, is unacceptable in either case.

The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. Do not reverse direction of by-pass valve. This will cause impinger solution back up into the probe. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as shown below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the impinger solution from being forced backward into the probe.

Leak checks shall be conducted as described whenever the train is disengaged, e.g., for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce the lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes at the highest vacuum reading drawn during that sampling traverse.

4.1.5 Sample train operation--During the sampling run, isokinetic sampling rate to within 10 percent, or as specified by the Administrator, of true isokinetic and the temperature around the

filter of no greater than 120 ± 14° C (248 ± 25° F), or as specified by an applicable subpart of the standards, shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer.

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle can, verify that the filter and probe are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85 ± 0.02 coefficient and when sampling in air or a stack gas with equivalent density (molecular weight equal to 29 ± 4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomograph unless appropriate steps (see Reference 7.7) are taken to compensate for the deviations.

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

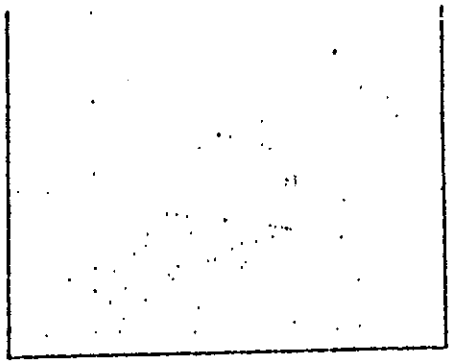
SAMPLE BOX NO. _____

METER BOX NO. _____

METER $\Delta H_@$ _____

C FACTOR _____

PITOT TUBE COEFFICIENT, C_p _____



SCHEMATIC OF STACK CROSS SECTION

BAROMETRIC PRESSURE _____

ASSUMED MOISTURE, % _____

PROBE LENGTH, m (ft) _____

NOZZLE IDENTIFICATION NO. _____

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____

PROBE HEATER SETTING _____

LEAK RATE, m^3/min (cfm) _____

PROBE LINER MATERIAL _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE mm Hg (in Hg)	STACK TEMPERATURE (T_S) °C (°F)	VELOCITY HEAD (ΔP_S), mm(in.)H_2O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H_2O (in. H_2O)	GAS SAMPLE VOLUME m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		FILTER HOLDER TEMPERATURE, °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPIN. °C (°F)
							INLET °C (°F)	OUTLET °C (°F)		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

22-2

Figure 12. Field data.

When the stack is under significant negative stack pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid 0.1N HNO_3 backing into the probe. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes, to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper temperature and add more ice and, if necessary, salt to maintain a temperature of less than 20° C (68° F) at the silica gel impinger outlet to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The results shall be the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check at a vacuum equal to or greater than the maximum reached during sampling. Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining lead. Do not tightly cap off the probe tip while the sampling train is cooling down as whis would create a vacuum in the probe, thus drawing solution from the impingers into the probe.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present by maintaining the probe in an essentially level position. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. After wiping off the silicone

grease, cap off the filter holder outlet and impinger inlet. Either ground glass stoppers or plastic caps or serum caps may be used to close these openings.

Transfer the probe and impinger-filter assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the 0.1N HNO_3 used for sampling and cleanup as a blank. Place about 200 ml of this 0.1N HNO_3 taken directly from the bottle being used into a glass sample container labeled "0.1N HNO_3 blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the sample side is inside the fold. Quantitatively remove any visible sample matter and/or filter which adheres to the filter holder gasket by carefully using a dry nylon bristle brush and/or a sharp-edged blade and place into this container. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1N HNO_3 and placing the wash into a glass container in the following manner:

Carefully remove the probe nozzle and clean the inside surface by rinsing with 0.1N HNO_3 from a wash bottle and brushing with a nylon bristle brush. Brush until the 0.1N HNO_3 rinse shows no visible particles, after which make a final rinse of the inside surface with 0.1N HNO_3.

Brush and rinse with 0.1N HNO_3 the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with 0.1N HNO_3 by tilting the probe and squirting 0.1N HNO_3 into its upper end, while rotating the probe so that all inside surfaces will be rinsed with 0.1N HNO_3. Let the 0.1N HNO_3 drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washes to the container. Follow the 0.1N HNO_3 rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1N HNO_3 into the upper end as the probe brush is being pushed with a twisting action through the probe, hold a sample container underneath the lower end of the probe, and catch any 0.1N HNO_3 and sample matter which is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 9.1N HNO_3 or remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1N HNO_3 and quantitatively collect these washings in the sample container. After the brushing make a final 0.1N HNO_3 rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize losing the sample. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a nylon bristle brush and rinsing with 0.1N HNO_3. Rinse each surface three times or more if needed to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1N HNO_3 washings and sample matter are collected in the sample container, tighten the lid on the sample container so that 0.1N HNO_3 will not leak out when it is shipped to the laboratory. Measure and record the total quantity of 0.1N HNO_3 used for rinsing. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label container to clearly identify its contents.

Container No. 3. Note color of indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure under analysis.

Container No. 4. Due to the large quantity involved, the impinger solutions are placed in a separate container. However, they may be combined with the contents of container No. 2 at the time of analysis in order to reduce the number of analyses required. Each of the first three impingers and their connecting glassware is cleaned in the same manner using the following procedures:

(1) Impinger ball joints are wiped free of silicone grease and capped.

(2) Impingers are rotated and agitated using the impinger contents as the first rinse solution.

(3) The impinger parts (inner and outer tubes) must not be separated while transferring their contents to a measuring container. The outlet ball joint cap should be removed and the contents drained through this opening.

(4) The liquid in the first three impingers is measured either to within $\pm$ 1 ml by using a 500-ml graduated container or to within $\pm$ 0.5 g by using a balance. The volume or weight of liquid present, along with a notation of any color or film observed in the impinger catch, is recorded in the log. This amount of liquid (less the initial charge) is added to the moisture data recorded from the silica gel. The measured impinger contents are poured into the No. 4 container.

(5) Approximately 50 ml of 0.1N HNO_3 is poured into each of the three impingers and again agitated. The 0.1N HNO_3 is then drained through the outlet ball joints into the No. 4 sample container as before.

This operation is repeated a second time after which the impingers are inspected for any abnormal conditions.

(6) The ball joints of the glassware connecting the impingers, and the back half of the filter holder are wiped free of silicone grease and rinsed twice with 0.1N HNO_3. This rinse is placed in the impinger contents container. (Note: Do not rinse or brush the glass-fritted support.)

(7) Measure and record the total amount of 0.1N HNO_3 used for rinsing.

4.3 Analysis.

4.3.1 Weigh the silica gel to the nearest gram.

4.3.2 Lead sample preparation.

4.3.2.1 Filter--Cut the filter into strips and transfer the strips and all loose particulate matter to a 125 ml Ş Erlenmeyer flask. Rinse the petri dish with 15 ml of distilled water to assure a quantitative transfer and add to the flask. Add 15 ml redistilled concentrated nitric acid. Reflux for two hours and cool to room temperature. Rinse the condenser column with a small amount of deionized distilled water and remove the flask. Filter the sample through the millipore filter, rinse the filter and the remaining glass fiber mass with several small portions of deionized distilled water, and combine with the filtrate. Dilute to 50 ml, using a 50 ml graduated cylinder.

4.3.2.2 Impinger samples--Evaporate the liquid sample to approximately 15 ml, transfer to an Erlenmeyer flask, and add 15 ml of redistilled concentrated nitric acid. Reflux for two hours, rinse the

condenser column, cool to room temperature, and dilute to 50 ml with deionized distilled water.

4.3.2.3 Probe wash--Treat in the same manner as directed in Section 4.3.2.2. As an option, this solution can be combined and treated with the impinger solution.

4.3.2.4 Filter blank--Determine a filter blank using one filter from each lot of filters used in the sampling train. Cut the filter into strips and treat in the same manner as directed in Section 4.3.2.1.

4.3.2.5 Instrument preparation--Turn on the power, set the wavelength, slit width, and lamp current as instructed by the manufacturer's manual for the particular atomic absorption spectrophotometer. Adjust the burner and flame characteristics as necessary.

4.3.2.6 Lead determination--After the absorbance values have been obtained for the standard curves (Section 5), determine the absorbance of the filter blank and each sample against the reagent blank. Determine the lead concentration in the filter blank and in each sample from the standard curve. If the sample concentration falls above the limits of the curve, make an appropriate dilution with 30 percent v/v HNO_3 such that the final concentration falls within the range of the curve.

5. Calibration and Standards

Maintain a laboratory log of all calibrations.

5.1 Calibration. Determine the absorbance of the standards against the reagent blank (Section 3.4.5). These absorbances should be checked frequently during the analysis to assure that no drift has occurred. Prepare standard curves for both the solution sample standards and the filter

sample standards of absorbance versus concentration. (Note: For instruments equipped with direct concentration readout devices preparation of a standard curve will not be necessary.) In all cases, the manufacturer's instruction manual should be consulted for proper calibration and operational procedures.

6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Amount of lead collected:

$M_n = M_a \times A \times F_d$ where

M_n = Total µg of lead collected in a fraction

M_a = µg/ml of lead in the aliquot as read from the standard curve

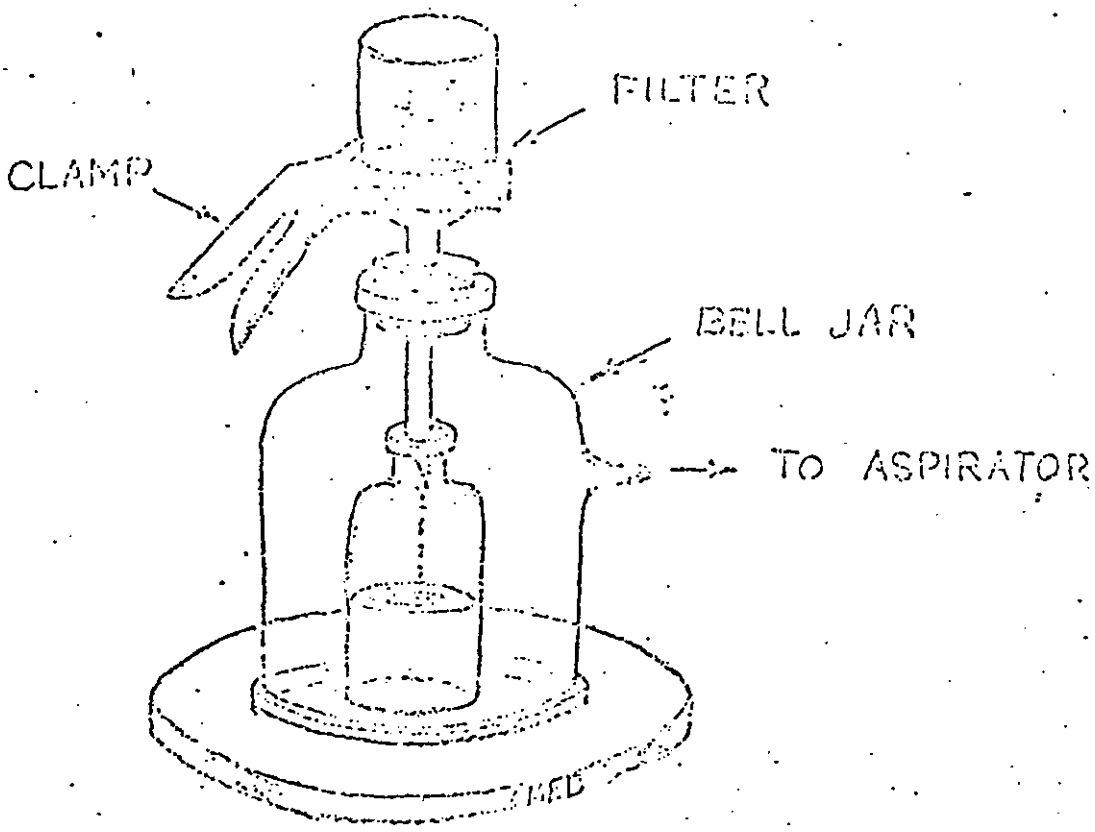
A = 50 ml/aliquot = sample volume

F_d = dilution factor = 1 if the sample has not been diluted

$M_t = M_n$ (filter) + M_n (probe wash) + M_n (impingers) - M_n (filter blank), where

M_t = total lead collected

6.2 Refer to EPA Method 5 for other necessary calculations.



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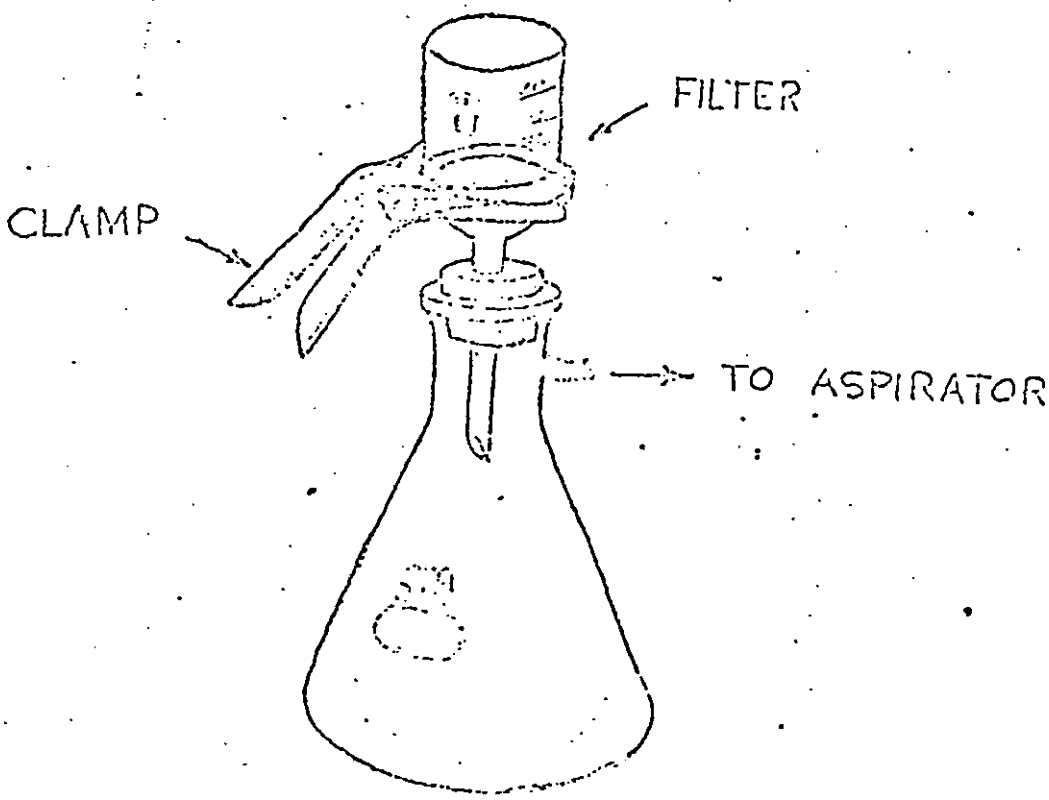

Figure 13. FILTERING APPARATUS