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COMPARATIVE TESTING OF EPA METHODS 5 AND 17 AT NONMETALLIC MINERAL PLANTS

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations and to evaluate the means of monitoring compliance with regulations and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina has responsibility for: assessment of environmental monitoring technology and systems; implementation of agency-wide quality assurance programs for air pollution measurement systems; and supplying technical support to other groups in the Agency including the Office of Air, Noise and Radiation, the Office of Toxic Substances and the Office of Enforcement.

This study was conducted to determine if in-stack filters are suitable substitutes for EPA Reference Method 5 when they are used to sample dry, ambient temperature processes.

ABSTRACT

Comparative tests between EPA Reference Method 5 and commercially-available in-stack filters (EPA Reference Method 17) showed that in-stack filters can be acceptable substitutes for the Reference Method at nonmetallic mineral plants. These comparative tests were conducted in the exhaust stacks from a clay calciner, a feldspar grinding mill and a limestone crusher. Particulate concentrations encountered ranged from 30 to more than 300 mg/m^3. Laboratory studies using feldspar dust gave results identical to the field study results. Insignificant amounts of material were found downstream of the in-stack filters.

CONTENTS

Foreword iii
Abstract iv
Figures vi
Tables vi

1. Introduction 1
2. Conclusions 3
3. Experimental 5
   Laboratory studies 5
   Field Studies 5
4. Results 9
   First field test - feldspar plant ball mill stack 10
   Second field test - clay plant calciner stack 12
   Third field test - limestone plant secondary crusher stack 15
   Fourth field test - feldspar plant ball mill stack 15

References 19
Appendices

A. A field evaluation of EPA Method 17 in a kraft pulp mill . . . 20
B. A comparative study of Method 17 with Method 5 at a grain elevator 45

FIGURES

Number		Page
1	Four-train sampling arrangement showing train position	6

TABLES

1	Results of Laboratory Filter Evaluation	11
2	Results at Feldspar Plant	13
3	Results at Clay Calciner	14
4	Results at Limestone Crushing Plant	16
5	Results at Feldspar Plants	18

SECTION 1

INTRODUCTION

Method 5 was the first EPA Reference Method for measuring particulate emissions from stacks (1). In Method 5, the sampled gas passes through a heatable glass-lined probe and then through a heatable glass-fiber filter located outside the stack. It then passes into a series of Greenburg-Smith impingers where moisture and other condensible material is collected. These impingers protect the pump and volume meter and also allow the percent moisture in the sampled stack gas to be determined.

Subsequently, EPA promulgated a second Reference Method for particulate measurement, EPA Reference Method 17 (2) in which the out-of-stack (Method 5) filter is replaced with an in-stack filter located immediately after the sampling nozzle. This method serves as an alternate method for Method 5 at kraft pulp mills, provided 9 mg/m^3 is added to the measured particulate concentration to correct for condensible material that can pass through the in-stack filter but would be stopped by an out-of-stack Method 5 filter. This correction factor was derived from comparative testing conducted at kraft pulp mills by the National Council for Air and Stream Improvement (NCASI) and was confirmed in a subsequent EPA study (Appendix A).

Testers prefer Method 17 to Method 5 primarily because it is more convenient to use. For example, a filter heater box is not required. Also, sample recovery is simpler and possibly more quantitative because the particulate catch is concentrated in the nozzle and filter and not dispersed throughout a glass sampling probe. These features make the in-stack filter attractive for sampling the dry, ambient temperature, baghouse-equipped stacks associated with plants that process dry, water-insoluble, feed materials such as grains and minerals. At these sources, if penetration of the in-stack filter is

insignificant, a sampling train composed only of a nozzle, an in-stack filter and a control console is adequate.

Reported below are the results of comparative tests between in-stack and out-of-stack filters conducted in the laboratory and at three nonmetallic mineral plants that processed feldspar, clay and limestone. The particulate concentration encountered at these plants ranged from 35 to 360 mg/m^3 and represented the range in mineral hardness that would be encountered in sampling the different kinds of plants that comprise the nonmetallic mineral processing industry. Appendix A and Appendix B describe similar studies done at a kraft pulp mill and a grain elevator, respectively.

SECTION 2

CONCLUSIONS

If in-stack filters are properly seated and filter integrity is maintained during sampling, very little material gets past them when they are used to sample the dry, ambient-temperature stacks encountered in the grain and mineral processing industries. This conclusion is drawn from the results of this study and a similar study conducted at a grain elevator (Appendix B). However, significant amounts of material can get past the in-stack filters when they are used to sample elevated temperature stacks that can contain condensible materials such as those encountered in kraft pulp mills (Appendix A).

On the average at the nonmetallic mineral plants the amount of material getting past the in-stack filters evaluated was only 1.3 mg/m^3. This value is statistically similar to the 3.7 mg/m^3 obtained at grain elevators but statistically different from the 9 mg/m^3 obtained at a kraft pulp mill.

In the first and third field tests, the in-stack filter train collected significantly more material than the Method 5 train. There is no clear explanation for this. Possibly it resulted from stratification of the particulate in the stack. Alternately, it could have been caused by non-quantitative recovery of material from the Method 5 probe, but this seems unlikely for two reasons. First, the probes were visually inspected after cleaning to make sure that all particulate was recovered. Second, the within-train precision of the Method 5 train was not significantly different from that of the in-stack filter trains. If sample recovery was a problem we would expect both the within train variation to be larger for the Method 5 train and some of the Method 5 trains to yield results significantly larger than the corresponding in-stack filter trains. Neither event occurred.

All in-stack filter holders evaluated to date could be improved. The BGI 19 x 90 mm thimble holder clamp screw is easily lost and it is extremely difficult to align the clamp screw with the threaded hole. Threading both holes rather than just one, would prevent dropping the screw during filter assembly. Alternately, a piece of copper wire can be slipped through the two holes and then wire twisted to pull the clamp together. (Private communication from Richard H. Russell, Standard Oil of Ohio, Cleveland, OH.) The larger thimble holder (30 x 100 mm) is easier to assemble, but the variability in filter diameter still makes it difficult to get a leak-free seal.

The glass-fiber filter tends to stick to the gasket and filter support screen of the Gelman 47 mm diameter filter holder and this makes it difficult to completely recover the filter after use. (The Misco Corporation 47 mm diameter filter also suffers from these problems -- Appendix A.)

The NuTech Corporation 51 x 178 mm rectangular filter is cumbersome to assemble and much heavier than the other filter holders. It is difficult to make leak-free and also difficult to remove the filter after use. The portion of the filter in contact with the viton gasket adhers strongly to the gasket and the portion of the filter supported by the sintered metal support plate can break free when the filter assembly is opened. The balance of the filter must then be scraped from the gasket.

To prevent leaks when using these in-stack filters at stack temperatures above 175°C, it is necessary to bring the filters to stack temperature and then tighten them further before use.

SECTION 3

EXPERIMENTAL

LABORATORY STUDIES

Samples of feldspar dust that had been sieved through a 200-mesh screen, were placed in the sampling nozzle and pulled into the train at an air sampling rate of 22 lpm for 20 to 30 minutes. The in-stack filters evaluated were followed by a glass-lined probe, an 82-mm diameter glass-fiber filter contained in a Method 5 filter holder and two Greenburg-Smith impingers. The out-of-stack filters evaluated were preceded by a glass-lined probe and followed by two Greenburg-Smith impingers. All impingers contained 100 ml of distilled, deionized water. All out-of-stack filters were supported on 200 mesh screens.

At the conclusion of sampling each portion of the sampling train was cleaned separately to determine the distribution of the material in the train. Acetone was used as the wash reagent.

The two commercially-available in-stack glass-fiber filters evaluated extensively were the BGI TA-3, 19 x 90 mm thimble filter and the Gelman 47 mm diameter filter. The 82 mm diameter, glass fiber filter was the only out-of-stack filter evaluated. Less extensive studies were done using the NuTech Corporation rectangular 51 x 178 mm glass-fiber filter and the Gelman 47 mm diameter, 0.8 and 5.0 micron membrane filter.

FIELD STUDIES

The field tests employed single-point, isokinetic sampling with the four-train sampling assembly shown in Figure 1. Prior to field use this assembly was studied in a wind tunnel to confirm that the pitot tube measurement was not affected by the four sampling nozzles.

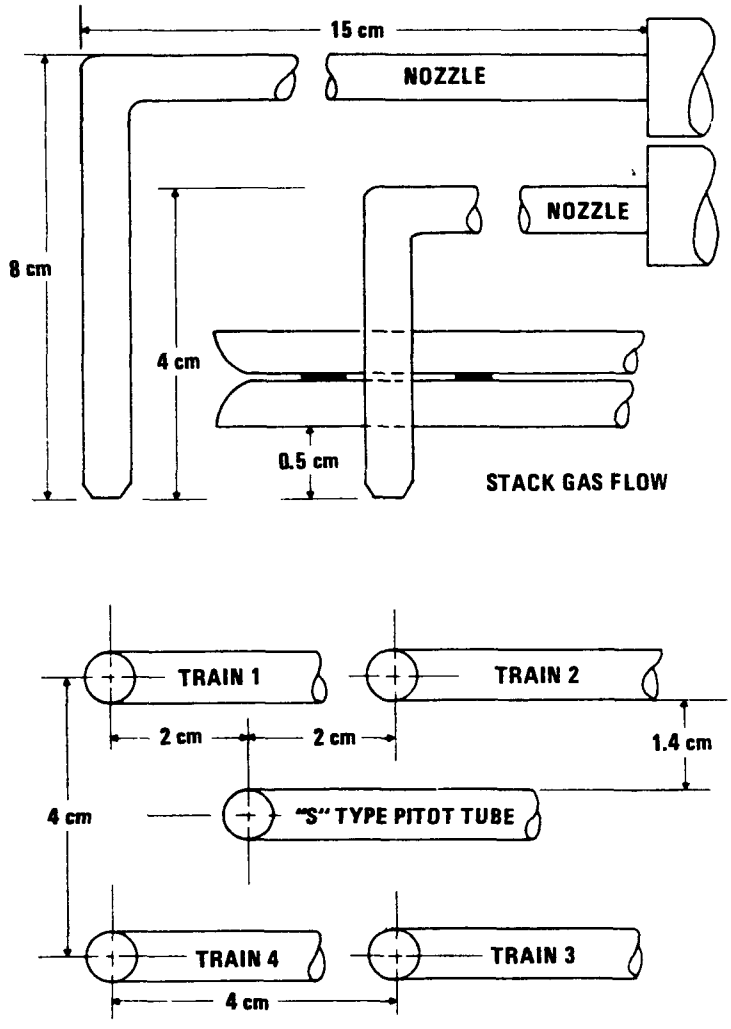

Figure 1. Four-train sampling arrangement showing train position.

Because this sampling assembly allows four trains to simultaneously sample at essentially the same point in the stack, it reduces the affect of spatial and temporal variations in the velocity and particulate profiles on the sampling results (3). It also permits a statistically significant number of samples to be taken in a short amount of time. Further, if two of the four trains are identical, the within-train precision can be determined at the same time the relative performance of two different trains is being compared.

Stack testing was conducted using BGI and Gelman in-stack filters at a feldspar grinding mill, a clay calciner and a limestone crusher. All stacks were at ambient temperature, contained less than two percent moisture, and controlled their emissions using a baghouse. Respective particulate concentrations encountered were: 70-160 mg/m^3, 35-60 mg/m^3, and 30-360 mg/m^3.

At a minimum, each in-stack filter was backed-up by a glass-lined probe and an out-of-stack, 82 mm diameter glass-fiber filter. In one test this back-up filter was followed by two Greenburg-Smith impingers containing 100 ml of distilled, deionized water. At the feldspar plant the out-of-stack filter train, which served as the control, contained two Greenburg-Smith impingers in back of the filter. At the other two plants there was no back-up system behind the out-of-stack filter.

Prior to use, each probe, filter holder and back-up system was subjected to a sample recovery wash with acetone. Less than 1 mg was found in these pre-test washes.

To reduce both the time spent on sample recovery and the effect of sample recovery errors on the results, material in a back-up system was not recovered until at least two and sometimes three sampling runs had been completed (acetone was the only reagent used for sample recovery of all sample-exposed surfaces). After recovery, the weight of material found in the back-up system was distributed equally among the two or three samples it represented. Of course, waiting several runs before recovering the back-up system meant that a broken or misaligned primary filter could invalidate the back-up system results for several samples. Also, the back-up system results could be adversely affected if the

penetration of the filter was a function of the particulate catch and there was a sudden, dramatic change in the stack concentration between runs.

To further reduce total field time, to utilize field personnel more effectively, to minimize the effect of daily variations in the particulate concentration on sampling results, and to simplify sample identification, three, four-train assemblies were used at each plant. While one assembly was sampling, the second was being recovered and the third was being reassembled for use in the next run. All trains were leak-checked through the nozzle before and after use and no leaks in excess of 0.0008 m^3/min were found.

SECTION 4

RESULTS

LABORATORY TESTS

The initial experiments determined the overall suitability of various types of commercially-available in-stack filters for field use and familiarized field personnel with the techniques required to quantitatively recover mineral dust from these filter holders. The following in-stack filter holders were evaluated in these initial experiments. Gelman holder with 47 mm diameter, 5.0 and 0.8 micron membrane and glass-fiber filters; BGI Corporation TA-3 with 19 x 90 mm glass-fiber thimble; and NuTech Corporation rectangular, flat filter holder with 51 x 178 mm glass-fiber filter.

In the preliminary tests recovery efficiencies on 20 to 100 mg quantities of feldspar dust ranged from 80 to 98% with no correlation between recovery efficiency and original weight of dust. This "lost" material was not found in the back-up system, which indicated that significant quantities of dust were not penetrating the filters. Careful study of these results indicated that extreme care was necessary in assembling and disassembling the filter holders to ensure that some of the filter was not lost. The NuTech Corporation filter and the Gelman glass-fiber filter tended to stick to the gasket and filter support screen, respectively. The BGI thimble clamp tended to cut the thimble and at times the thimble lip would tear slightly when the thimble was slipped over the shaft.

With experience the filter handling problems of the BGI and Gelman holders became manageable, but those of the NuTech did not, so the latter was not evaluated further in field or laboratory tests.

Extensive laboratory experiments on in-stack filters were done only with the BGI and Gelman filter holders equipped with glass-fiber filters. The

out-of-stack filter (Method 5) was also evaluated in these experiments to serve as a control.

Results for all three filters were the same, i.e., consistent filter penetration was not found and recovery efficiency did not correlate with sample weight (Table 1). Generally, the amount of material recovered in the front half differed no more than ± 3 mg from the amount of material originally placed in the train. Where penetration did occur, it could reflect only filter slippage and not penetration.

FIRST FIELD TEST - FELDSPAR PLANT BALL MILL STACK

This plant had been tested four years earlier by an EPA contractor and found to have a low particulate loading (10-15 mg/m^3) and a fairly uniform distribution of particulate across the 45 cm stack. Based on this data, the comparative testing of the in-stack and Method 5 filters was done utilizing three separate sampling assemblies that contained two identical trains and two non-identical trains. Assembly A consisted of two Method 5 trains, one Gelman train and one BGI train; Assembly B consisted of two BGI trains, one Gelman train and one Method 5 train; and Assembly C consisted of two Gelman trains, one BGI train and one Method 5 train. The BGI trains consisted of a nozzle, a TA-3 filter holder with 19 x 90 mm glass-fiber filters and were followed by a 50 cm glass-lined probe and an 82 mm diameter Method 5 glass-fiber filter. The Gelman trains consisted of a nozzle, a Gelman filter holder with 5.0 micron, 47 mm diameter membrane filters, and a back-up system identical to that of the BGI train. The Method 5 train which consisted of a nozzle, 55-65 cm glass-lined probe and a filter holder with a 82-mm diameter glass-fiber filter did not have a back-up system.

Unfortunately, the particulate concentration encountered was apparently stratified and about five times larger than anticipated because several of the baghouse bags were broken — a fact not detected until after the test series was completed. Because of the heavy particulate loading, some of the membrane filters cracked (one actually ruptured) during use. Poor quality control during manufacture caused several of the BGI thimbles to fit loosely

TABLE 1. RESULTS OF LABORATORY FILTER EVALUATION

	Sample wt. (mg)	Total % recovered	% in front section	% in back-up section
Method 5 Filter Followed by Two Impingers				
	26	111	92	8.0
	28	106	100	0.0
	32	108	100	0.0
	32	119	100	0.0
	95	103	99.6	0.4
	100	92	96	4.0
	106	99	100	0.0
	109	98	100	0.0
	AVERAGE:	104		
Gelman 47 mm Filter Followed by Method 5 Filter and One Impinger				
	22	103	100	0.0
	28	83	100	0.0
	36	106	100	0.0
	98	99	100	0.0
	112	97	100	0.0
	AVERAGE:	98		
BGI 19 x 90 Thimble Followed by Method 5 Filter and One Impinger				
	25	142	100	0.0
	32	102	100	0.0
	104	100	100	0.0
	110	103	99	1.0
	AVERAGE:	112		

over the thimble holder shaft and this made it difficult to attach the thimble securely to the shaft with the rubber bands being used. (The standard clamp supplied with the BGI thimble would not slide over these thimbles easily and it was difficult to align the clamp screw with the threaded hole under field conditions, so they were not used.) These facts essentially invalidated the use of the back-half catch, although the general conclusion was drawn that where filter integrity was apparently maintained, little material was found behind the in-stack filter. Surprisingly, the Method 5 results were generally lower than the Method 17 results (Table 2). Possibly this resulted from stratification in the particulate concentration caused by the broken bags. Poor sample recovery from the Method 5 probe may also be the cause (the gritty material is difficult to remove by washing) but this seems unlikely because the probes were visually inspected after sample recovery to make certain they were clean.

SECOND FIELD TEST - CLAY PLANT CALCINER STACK

The second test, conducted in a 100-cm diameter stack, used sampling trains identical to those used in the first test, except that Gelman glass-fiber filters were substituted for the membrane filters and wire was used to hold the BGI thimbles in place. The statistical design was modified to better tolerate particulate stratification and sudden shifts in the particulate concentration, but neither event occurred and only one sample was of questionable validity (Table 3).

Penetration of the in-stack filters was fairly low and consistent. For example the average penetration of the Gelman trains was 0.7 and 3.2 mg/m^3 for an overall average of 2.0 mg/m^3 and average penetration of the BGI trains was 1.3, 4.0, 2.2, and 3.0 mg/m^3 for an overall average of 2.6 mg/m^3. Interestingly, if 2.0 mg/m^3 is added to the average Gelman filter train catch (43.0 mg/m^3), the total (45.0 mg/m^3) compares extremely well with the average catch for the corresponding Method 5 train (47.0 mg/m^3). Similarly, the average total catch for the BGI train and its back-up system (47.4 mg/m^3) compares well with the corresponding Method 5 train (47.9 mg/m^3).

TABLE 2. RESULTS AT FELDSPAR PLANT

Sampling assembly	Train position[a]	Type train	Particulate conc. measured (mg/m^3) First run	Second run	Third run	Mg in back-up system	Average back-up catch in mg/m^3/train
A	1	Method 5	51.2	45.8	46.5	-	-
	2	Method 5	52.6	54.0	65.7	-	-
	3	BGI	59.5	b	73.4	5.7	1.7
	4	Gelman	62.1	75.3	90.3	11.7	2.1
B	1	Gelman	58.0	c	59.7	-	-
	2	BGI	78.8	80.5	41.5	30.3	6.0
	3	BGI	81.9	86.6	117.8	9.3	1.9
	4	Method 5	47.8	48.9	44.4	-	-
C	1	BGI	73.2	66.8	41.6	33.9	7.0
	2	Gelman	78.6	55.3	82.6	41.7	9.3
	3	Method 5	49.8	46.3	50.3	-	-
	4	Gelman	70.7	69.9	59.5	4.8	1.0

[a]See Figure 1 for explanation of train position.

[b]Nozzle rinse spilled.

[c]Membrane filter broke during test.

TABLE 3. RESULTS AT CLAY CALCINER

Sampling assembly	Train position[a]	Type train	Particulate conc. measured (mg/m^3) First run	Second run	Third run	Mg in back-up system	Average back-up catch in mg/m^3/train
A	1	BGI	44.7	39.0	-	4.4	1.3
	2	Method 5	51.1	45.4	-	-	-
	3	BGI	56.2	38.7	-	11.3	4.0
	4	Method 5	66.3	38.9	-	-	-
B	1	Method 5	40.3	39.8	-	-	-
	2	BGI	47.1	38.5	-	7.7	2.2
	3	Method 5	54.4	47.1	-	-	-
	4	BGI	49.4	44.7	-	9.9	3.0
C	1	Gelman	37.0	51.9	39.3	3.1	0.7
	2	Method 5	41.4	54.1	-[b]	-	-
	3	Gelman	37.1	50.8	41.7	11.9	3.2
	4	Method 5	44.6	54.4	40.4	-	-

[a]See Figure 1 for explanation of train position.

[b]Obvious contamination of nozzle rinse observed.

THIRD FIELD TEST - LIMESTONE PLANT SECONDARY CRUSHER STACK

Testing was done in a 66 x 81 cm rectangular duct using sampling trains and sampling assemblies identical to those used in the second field test. Based on a previous test at the plant, a particulate concentration of about 10 mg/m^3 was expected, but when sampling was initiated the loading was three times higher. The plant broke down repeatedly during the test and after the third run was completed the opacity of the emissions increased dramatically indicating baghouse malfunction. Testing scheduled for that day was completed and no further testing was attempted. Test results are presented in Table 4.

As in the first field test (feldspar plant), the in-stack filter trains collected more particulate than the Method 5 trains. However, as in the second field test, the average penetration for the in-stack filter trains was small and consistent, i.e., 1.6 mg/m^3 and 1.9 mg/m^3 for the Gelman and BGI trains, respectively.

FOURTH FIELD TEST - FELDSPAR PLANT BALL MILL STACK

Testing was repeated at this plant nine months after the first field test. Just before testing was initiated the baghouse was inspected to be certain that all bags were properly positioned and were not leaking. Each train contained a back-up system identical to that used in the laboratory study, i.e., all Method 5 filters were supported on screens and were followed by two Greenburg-Smith impingers that each contained 100 ml of distilled, deionized water. BGI 30 x 100 mm thimbles were substituted for the 19 x 90 mm thimbles used in the other tests. Their larger size made it possible to securely attach the thimble to the holder shaft using the screw clamp supplied.

The test was designed such that day to day variations in the particulate concentration and stratification could be statistically removed from the test results. During testing the baghouse again malfunctioned, but this time the test design tolerated it. Back-up samples were recovered after the second run.

TABLE 4. RESULTS AT LIMESTONE CRUSHING PLANT

Sampling assembly	Train position[a]	Type train	Particulate conc. measured (mg/m^3)			Mg in back-up system	Average back-up catch in mg/m^3/train
			First run	Second run	Third run		
A	1	Method 5	38.9	59.9	211.3	-	-
	2	Gelman	31.8	74.3	248.0	6.0	1.3
	3	Method 5	38.2	62.0	221.8	-	-
	4	Gelman	44.8	71.5	242.1	10.2	1.9
B	1	BGI	43.6	111.8	366.5	9.3	2.2
	2	Method 5	27.9	70.4	260.3	-	-
	3	BGI	49.6	111.2	325.5	8.1	1.5
	4	Method 5	37.0	99.3	288.4	-	-

[a]See Figure 1 for explanation of train position.

Analogous to the laboratory tests with feldspar dust, no significant penetration of the in-stack filters was found. Penetration of the Method 5 filter trains was found in three instances but this likely resulted from the filter slipping out of position and not an actual penetration. (Filter slippage occurred because the screen filter supports used did not contain a deep enough recessed area to accommodate the glass fiber filter. These screens were made by hand and were used in place of the standard fritted-glass filter supports used in the other field tests to ensure that any material penetrating the Method 5 filter would not be trapped in the filter support itself.)

Statistical analysis of the results showed that the concentration level increased over the four days of testing, but no correlation between concentration and between-train (Gelman versus Method 5, BGI versus Method 5) precision or concentration and within-train (Method 5 versus Method 5, BGI versus BGI, Gelman versus Gelman) precision was found. Further, no significant difference was detected between Method 5 and Gelman or Method 5 and BGI, which indicates that the two in-stack filters would give similar results if compared directly (Table 5). (Some small amount of stratification was observed, based on a position by position comparison of the sampling results for the assembly that contained four Method 5 trains, and this difference in positions was handled in the statistical analysis.)

TABLE 5. RESULTS AT FELDSPAR PLANTS

Sampling assembly	Train position[a]	Type train	Particulate conc. measured (mg/m^3) First run	Particulate conc. measured (mg/m^3) Second run	Mg in back-up per run	Average back-up catch in mg/m^3/train
A	1	Method 5	72.2	78.5	0.2	0.1
	2	BGI	88.6	89.0	0.0	0.0
	3	BGI	94.2	85.8	0.4	0.1
	4	Method 5	74.4[b]	74.1	2.5	0.8
B	1	Method 5	87.3	90.5	0.3	0.1
	2	Method 5	80.5	72.9	0.3	0.1
	3	Method 5	85.7	77.9	0.0	0.0
	4	Method 5	81.7	72.3	0.0	0.0
C	1	Method 5	83.7	83.9	0.0	0.0
	2	Gelman	74.4	95.6	0.0	0.0
	3	Gelman	83.5	79.2	1.2	0.8
	4	Method 5	84.3	95.2	0.0	0.0
A'	1	Method 5	85.9[c]	131	6.8	6.6
	2	Method 5	87.3	129	0.0	0.0
	3	Method 5	92.8	120	0.2	0.2
	4	Method 5	82.6	124	0.0	0.0
B'	1	Method 5	120	126	0.0	0.0
	2	BGI	118	124	0.0	0.0
	3	BGI	123	105	0.0	0.0
	4	Method 5	113	113	0.0	0.0
C'	1	Method 5	131	175	0.0	0.0
	2	Gelman	128	170[c]	7.8	4.6
	3	Method 5	122	139	0.0	0.0
	4	Gelman	114	153	0.0	0.0
A"	1	Method 5	127	135[c]	8.0	7.2
	2	Method 5	122	150	0.0	0.0
	3	Method 5	142	136	0.0	0.0
	4	Method 5	106	131	0.4	0.3

[a]See Figure 1 for explanation of train position.

[b]Filter cracked during testing.

[c]Filter definitely slipped out of position during testing.

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APPENDIX A

A FIELD EVALUATION OF EPA METHOD 17 IN A KRAFT PULP MILL[a]

I. INTRODUCTION

The proposed standard for particulate matter emissions from Kraft pulp mill recovery furnaces is 100 milligrams/dry standard cubic meter (mg/dscm) as set forth in the Federal Register, Part II, Volume 41, Number 187, Friday, September 24, 1976. The specified test method for particulate determination is EPA Method 5. EPA Method 17 may be used as an alternative method provided that a constant value of 9 mg/dscm is added to the Method 17 results and provided that the stack temperature is no greater than 205 °C. Water is used as a cleanup solvent instead of acetone in the sample recovery in both methods.

A field evaluation of EPA Method 17 at a Kraft pulp mill recovery furnace to include a comparison with EPA Method 5 was therefore considered appropriate. This report presents the results obtained and conclusions drawn from statistical analysis of the test data.

[a]Report prepared by Dr. H. F. Hamil and Mr. R. E. Thomas of Southwest Research Institute, San Antonio, TX, under EPA Contract 68-02-2489 and under the direction of Mr. Michael C. Osborne, EPA, Research Triangle Park, NC.

II. TEST DESCRIPTION

The field evaluation of Method 17 specified in the work assignment has a two-fold purpose: a field comparison of EPA Methods 17 and 5 by simultaneous sampling of the same source, and an evaluation of various commercially available Method 17 filter holders by simultaneous sampling of the same source.

The test plan was provided by the work assignment monitor and is shown in Figure 1. During the first week, six runs were to be accomplished with two Method 5 and two Method 17 trains sampling simultaneously on each run. The Method 17 filters were to be backed up by a heated Method 5 filter and glass-lined probe to collect any particulate which might pass through the in-stack filter. Nutech filter holders were to be used in the first week's test as representative Method 17 filter assemblies. The relative position of the trains in the sampling assembly were to be rotated after each run to avoid any bias due to sample nozzle location.

During the second week, eight runs were to be accomplished with one Method 5 train and three Method 17 trains sampling simultaneously on each run. The Method 17 trains

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1. All test runs will be conducted with the configuration shown in Figure 2.

2. Runs 1-6 Characteristics

   a. Trains 1 and 3 will be standard Method 5 trains with heated filter boxes.

   b. Trains 2 and 4 will be Method 17 trains with heated backup Method 5 filters.

   c. The relative position of trains 1,2,3, and 4 within the sampling arrangement will be rotated clockwise after each run.

   d. An additional front-half cleanup will be required following the regular cleanup of runs 1,2, and 6.

   e. Saving and determining the back-half catch will also be required in runs 1, 2, and 6.

   f. The Nutech filter holders should be used to represent Method 17 unless it is observed during pitot calibration that these holders cause significant turbulence that changes the pitot tube coefficient. If such a disturbance is observed, the Misco filter holders should be used in place of the Nutech filter holders.

   g. All filter holders and train components should be thoroughly leak checked before sending them into the field.

(continued)

Figure 1. Test plan for Kraft mill recovery furnace.

3. Runs 7-14 Characteristics

a. Trains 1,2,3, and 4 should use Misco, BGI, Nutech and Method 5 filter holders, respectively.

b. Trains 1,2, and 3 should be operated with filters in-stack as characterized by Method 17.

c. Train 4 should be operated as a standard Method 5 train with the filter holders out-of-stack in a heated compartment.

d. The relative position of trains 1,2,3, and 4 within the sampling arrangement will be rotated clockwise after each run.

e. It will not be necessary to do any additional front-half cleanups or to save the back-half catch.

Figure 1. (continued)

were to employ one each Nutech, Misco, and BGI filter holder. No backup Method 5 filters were to be used in the Method 17 trains during the second week of testing. The relative position of the trains within the sampling bundle were to be rotated after each run as in the first week of testing.

The use of a four-train sampling system was required to accomplish the test plan. To assure that no bias due to spatial variation in particulate loading was introduced into the sampling, the probe bundle was assembled so the sample nozzles were situated at the corners of a square with sides of 6 cm. A single pitot tube was centrally located. The pitot tube-sample nozzle spacings are shown schematically in Figure 2. A view of the assembled probe bundle with a Method 5 probe and the three Method 17 probes is shown in Figure 3.

A common thermostatically-controlled oven was fabricated to house the four Method 5 filter holders required in the first week of testing. A view of the filter holder arrangements is shown in Figure 4. The filter holder oven was mounted on a stand in such a manner that four standard impinger trains with ice baths could be accommodated in an assembly mounted on a common base.

This configuration allowed the entire sampling system to be assembled and leak checked. The entire unit could then be moved to place the sample nozzles at the desired sample point in the stack. Single point sampling without traversing was employed throughout the test. The assembled sampling system is shown in Figure 5.

To avoid differences in filter media, all Method 5, Nutech Method 17, and Misco Method 17 filters were cut from Gelman type A-E glass fiber high-volume filters. BGI glass fiber filters were used with the BGI filter holders.

The site selected for the field evaluation was the recovery furnace of a Kraft pulp mill owned and operated by the Arkansas Kraft Corporation, Morrilton, Arkansas. The

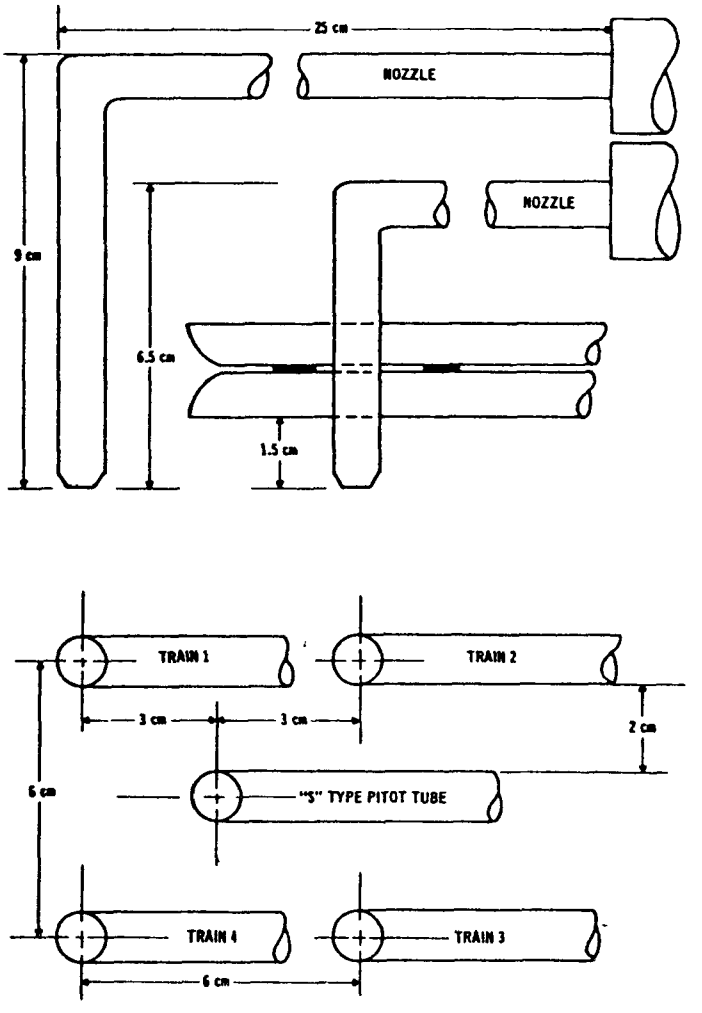


Figure 2. Single-pitot sampling arrangement.
Top: Side view.
Bottom: Upstream view.

recovery furnace at this site utilizes an electrostatic precipitator (ESP) for control of particulate emissions. The effluent from the ESP is fed to two 68-inch I.D. stacks on the roof of the ESP. The sampling platforms are located on the stacks 18 feet above the roof. Figures 6 and 7 show front and top views of the sample site, respectively. A preliminary site visit was made, and arrangements for installation of an 8-inch diameter sampling port on the east stack to accommodate the four-probe sample bundle were made. Data on particulate loading, stack gas velocity and temperature, and stack gas moisture content were provided by plant personnel from their previous sampling. Based upon these data and an inspection of the facilities available, the site was considered acceptable for the proposed test.

The field test was performed during the time period 23 August 1977 through 1 September 1977. A total of sixteen runs was made, as compared to the planned fourteen runs. Eight runs were made during each of the two weeks of the test. On the first two runs

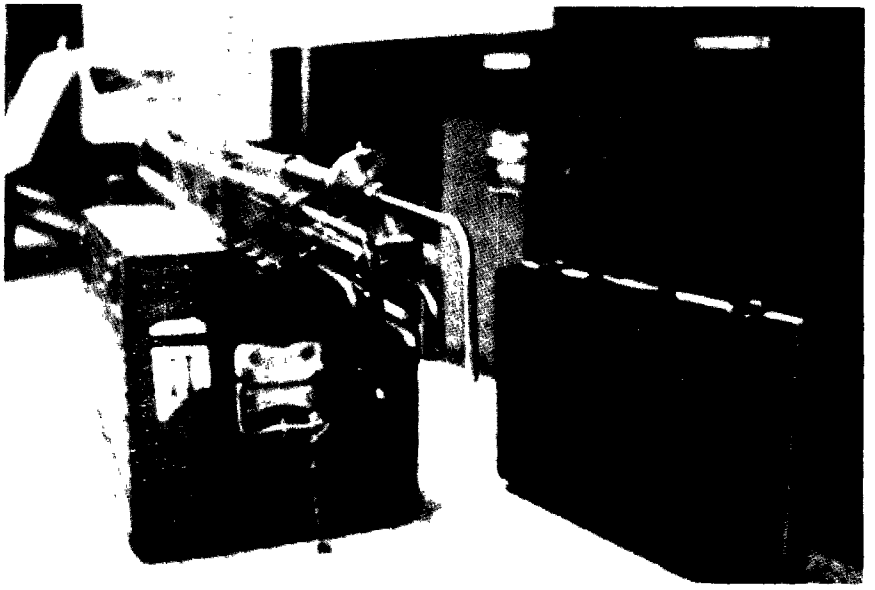


Figure 3. Four probe sample bundle.

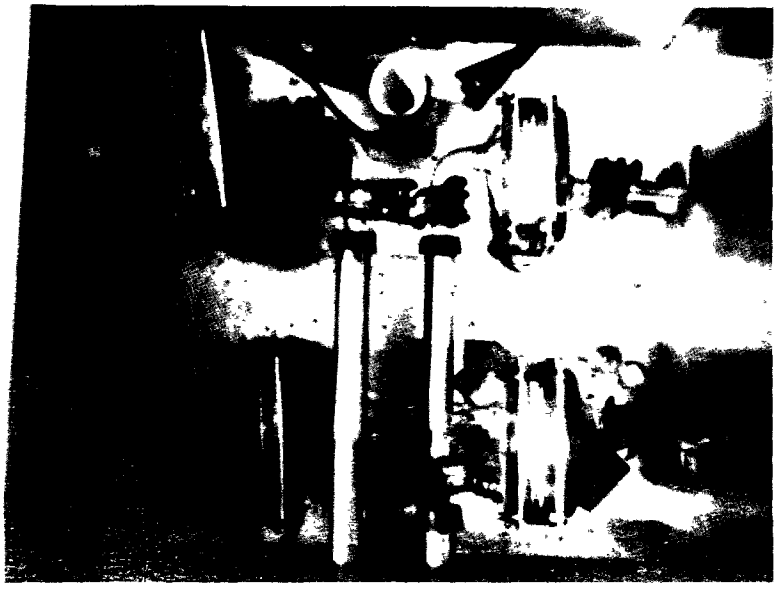
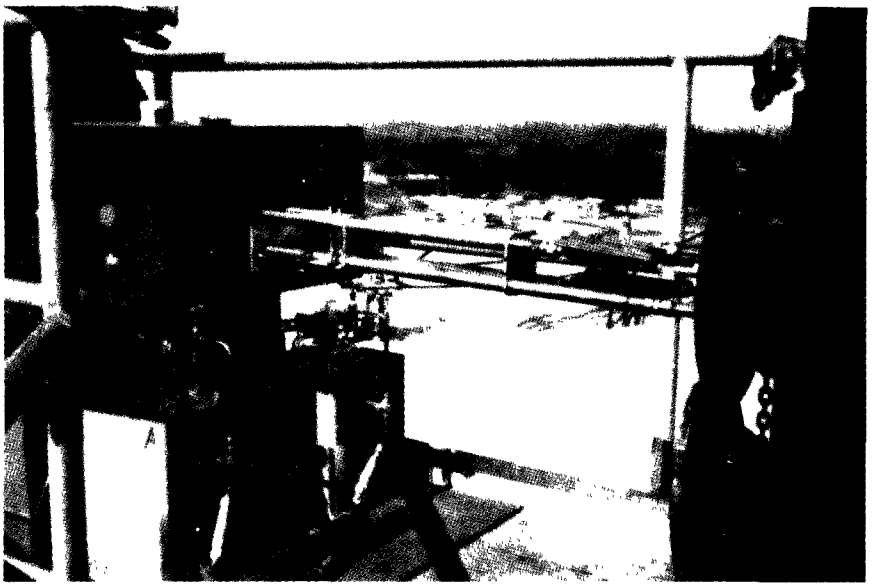
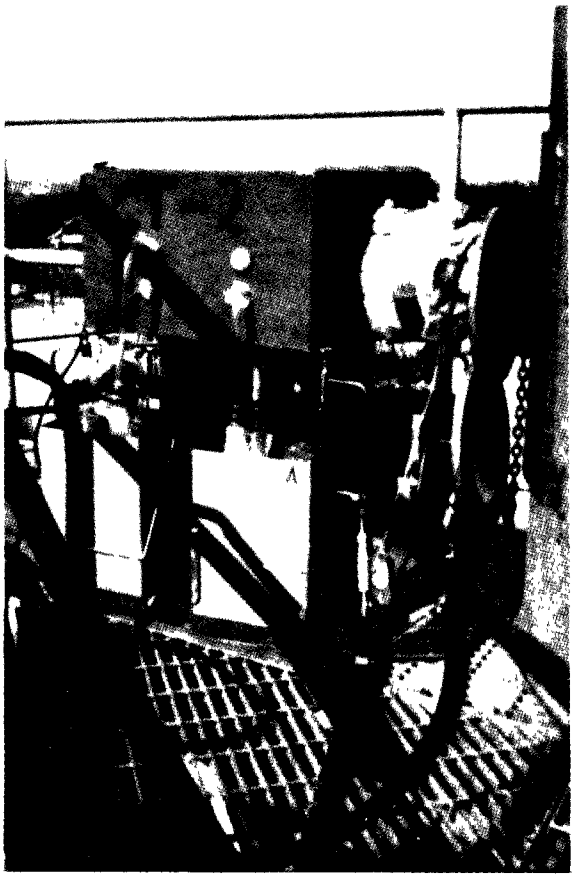


Figure 4. Filter holder oven



Ready for leak check.



During sampling.

Figure 5. Sampling system.

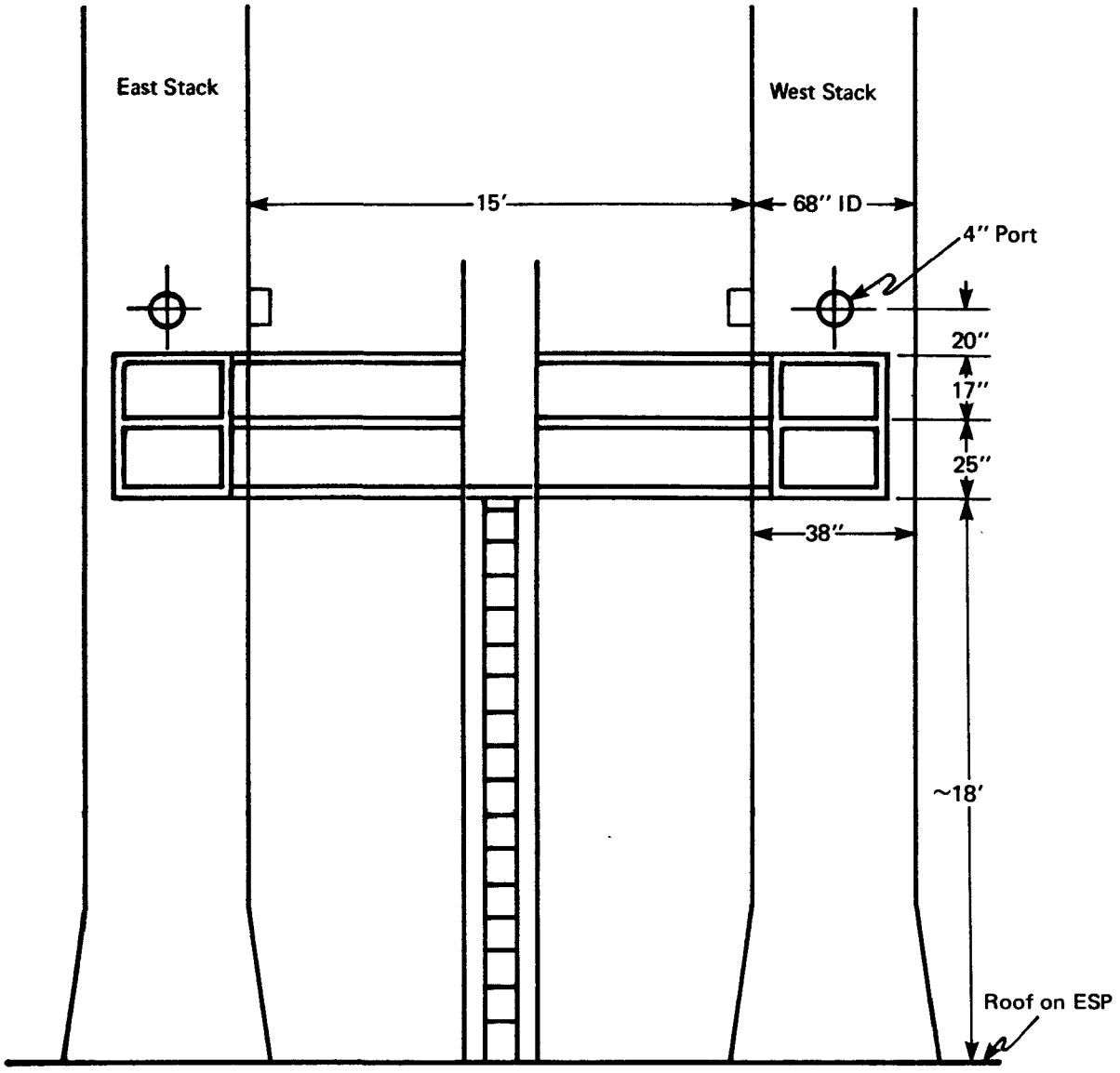


Figure 6. Arkansas Kraft Corporation precipitator exits—front view.

of week one, problems were encountered with leaks in the Method 5 filter holders on trains C and D, which were positioned nearest to the oven heating element. Presumably uneven heating caused the metal clamps on the Method 5 filter holders to loosen. Repositioning of the filter holders as far from the heating element as possible in conjunction with checking the tightness of the filter holder clamps after oven heatup corrected the problem on subsequent runs. Even though leaks occurred in only one of the four trains in each of the first two runs, the data from these runs are considered invalid for the purpose of the test and are not included in this report. However, two additional runs were made the first week, giving the total of six valid runs required by the test plan. A summary of the run parameters for the fourteen valid runs is presented in Appendix A.

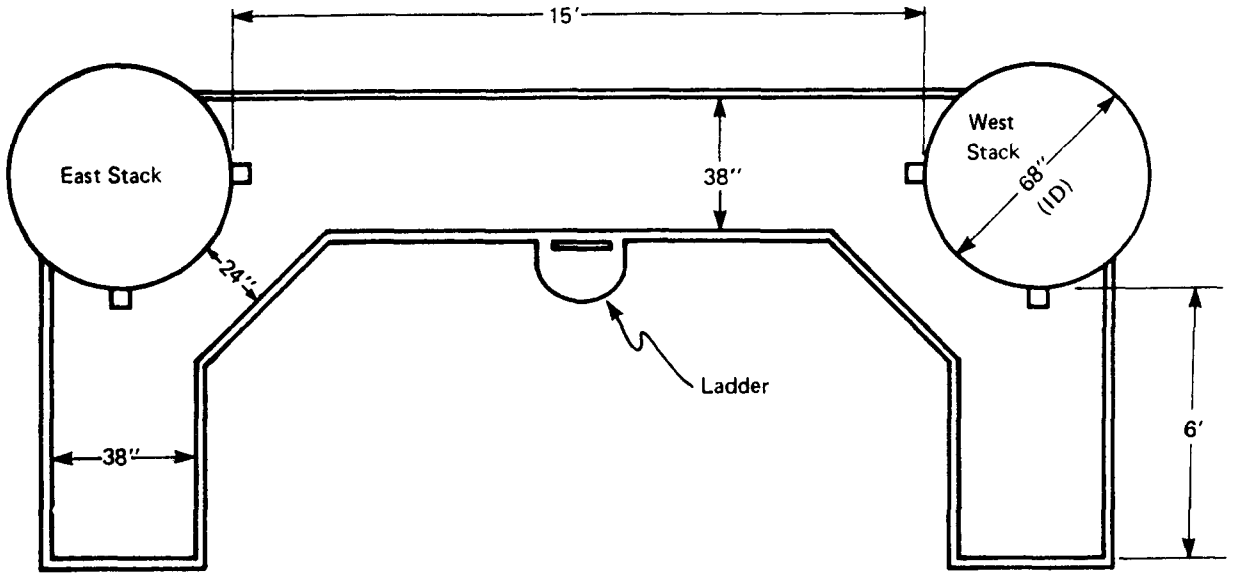


Figure 7. Arkansas Kraft Corporation precipitator exits—top view.

The particulate loadings for each train within each run are presented in Table I for the first week's test and in Table II for the second week's test. The particulate loading C_S for the Method 17 determinations was calculated three ways for the first week of testing (Table I). The C_S values presented under the heading "Method 17" were calculated using the actual particulate catch in the Method 17 filter and filter holder front half. The C_S values presented under the heading "Method 17 Adjusted" were calculated as just described above but with a constant correction of 9 mg/dscm added according to the instructions in the Federal Register pertaining to use of Method 17 in Kraft pulp mills. The C_S values presented under the heading "Method 17 Plus Backup" were calculated using the particulate catch from the Method 17 filter and filter holder front half, the backup probe, and the backup Method 5 filter and filter holder front half.

The particulate loading C_S for the Method 17 determinations for the second week of testing is presented in Table II. Also presented in Table II are the differences, in mg/m^3, between the Method 5 train and the individual Method 17 trains.

The actual weight of particulate caught and the location within the sample train are given in Table III for week one and Table IV for week two. Also included in Table III is the back-half catch for each train obtained by workup of the impinger catch. These back-half catch values are substantial but probably do not reflect filter inefficiency. The residue which was obtained upon evaporating the impinger water to dryness was not a particulate solid but was an oily semiliquid material. It probably consists of materials volatile at the filter oven temperature which are condensed in the impingers.

TABLE I. PARTICULATE LOADING, C_s
mg/dscm

WEEK 1

Run Number	Method 17	Method 17[a] Adjusted	Method 17[b] Plus Backup	Method 5
3A	18.8	27.8	34.4	
B				35.4
C	20.7	29.7	34.9	
D				34.4
4A				49.2
B	40.3	49.3	53.5	
C				57.4
D	46.3	55.3	55.6	
5A	46.4	55.4	60.7	
B				58.2
C	47.0	56.0	62.5	
D				52.9
6A				51.6
B	44.4	53.4	54.4	
C				56.4
D	37.0	46.0	46.7	
7A	60.9	69.9	70.4	
B				71.5
C	53.5	62.5	62.3	
D				70.1
8A				63.1
B	61.2	70.2	73.7	
C				73.5
D	54.1	63.1	63.5	
Mean	44.22	53.22	56.05	56.15

a. Actual Method 17 C_s value plus 9 mg/dscm per Kraft pulp mill regulations.
b. Actual Method 17 C_s value plus the material collected in the glass-lined probe and the back-up Method 5 filter and filter holder.

TABLE II. PARTICULATE LOADING, C_s
mg/dscm (Sampling Train)

WEEK 2

Run No.	Method 5	Actual Method 17[a] Nutech	Misco	BGI	Differences (Method 5-Method 17) Nutech	Misco	BGI
9	53.0(B)	35.2(A)	44.7(C)	45.4(D)	17.8	8.3[b]	7.6
10	33.1(C)	20.3(D)	28.0(B)	29.1(A)	12.8	5.1	4.0
11	68.9(B)	51.8(A)	54.3(C)	25.7(D)	17.1	14.6	43.2[c]
12	68.6(A)	50.0(D)	49.4(B)	54.1(C)	18.6	19.2	14.5
13	39.8(B)	35.0(A)	30.0(C)	29.8(D)	4.8	9.8	10.0
14	31.3(A)	27.1(D)	29.7(B)	26.6(C)	4.2	1.6	4.7
15	162.5(B)	137.7(A)	164.9(C)	138.3(D)	24.8	—2.4	24.2
16	162.7(C)	196.7(D)	155.8(B)	127.5(A)	—34.0	6.9	35.2
				Mean	8.3	7.9	17.9

a. Does not include the 9 mg/dscm conversion factor. In the statistical analysis, 9 mg/dscm was added to these values per Kraft pulp mill regulations.
b. Leak rate at conclusion of sampling four times the allowance leak rate.
c. Large particulate deposit observed in probe indicating leakage around filter. Results not deleted from analysis.

TABLE III. PARTICULATE CATCH, mg

WEEK 1

Run	Train	Method 5 Filter	Method 17 Filter	Method 17 Nozzle and Filter Holder	Method 5 Probe and Front of Filter Holder	Impinger Train and Back[a] of Filter Holder
3	A	12.8	17.5	2.8	4.1	46.5
	B	29.1	---	---	9.8	40.0
	C	13.0	14.1	8.3	2.5	43.1
	D	31.5	---	---	6.0	32.4
4	A	52.8	---	---	6.9	30.8
	B	10.1	47.3	1.0	5.7	36.9
	C	59.3	---	---	9.9	30.3
	D	6.8	50.5	5.0	4.4	34.8
5	A	8.3	46.9	9.1	8.4	34.6
	B	55.5	---	---	15.7	34.2
	C	11.2	52.0	3.6	7.1	34.2
	D	52.9	---	---	9.7	34.0
6	A	53.2	---	---	8.1	61.6
	B	7.1	46.1	6.1	4.7	68.3
	C	58.6	---	---	8.0	60.4
	D	7.2	41.0	2.9	4.4	69.9
7	A	9.6	78.3	3.8	3.2	42.3
	B	79.2	---	---	17.0	35.4
	C	6.3	60.9	8.3	5.1	43.4
	D	75.2	---	---	13.4	38.4
8	A	71.3	---	---	10.0	100.9
	B	11.4	74.7	3.3	4.5	106.0
	C	84.4	---	---	9.0	100.8
	D	6.1	65.0	2.6	5.7	106.1

a. Oily residue obtained upon evaporation of water.

TABLE IV. PARTICULATE CATCH, mg

WEEK 2

Run	Train	Method 5 Filter	Method 17 Filter	Nozzle, Probe, M-5 Front Half	Nozzle, M-17 Filter Holder	Method 17 Filter Holder Type
9	A	---	43.4	---	3.2	Nutech
	B	59.2	---	8.5	---	---
	C	---	53.0	---	5.0	Misco
	D	---	54.5	---	4.3	BGI
10	A	---	30.2	---	3.9	BGI
	B	---	29.2	---	3.3	Misco
	C	29.9	---	9.1	---	---
	D	---	18.2	---	5.8	Nutech
11	A	---	68.1	---	1.6	Nutech
	B	68.9	---	22.4	---	---
	C	---	63.5	---	9.0	Misco
	D	---	28.0	---	6.8	BGI
12	A	72.4	---	18.6	---	---
	B	---	61.8	---	2.4	Misco
	C	---	66.8	---	4.3	BGI
	D	---	62.3	---	3.2	Nutech
13	A	---	38.5	---	3.8	Nutech
	B	43.5	---	6.0	---	---
	C	---	28.9	---	8.6	Misco
	D	---	33.3	---	3.3	BGI
14	A	29.0	---	8.1	---	---
	B	---	28.9	---	6.4	Misco
	C	---	26.5	---	5.2	BGI
	D	---	26.7	---	5.2	Nutech
15	A	---	146.9	---	4.0	Nutech
	B	171.8	---	10.9	---	---
	C	---	167.5	---	7.2	Misco
	D	---	144.9	---	5.9	BGI
16	A	---	113.7	---	8.7	BGI
	B	---	102.4	---	48.5	Misco
	C	142.3	---	17.1	---	---
	D	---	181.4	---	8.2	Nutech

The particulate material recovered in the three special cleanup procedures is tabulated in Table V. As can be seen from the values presented, the residual particulate left in a sampling train after a properly conducted sample recovery is quite small and should not affect the accuracy of a determination.

Since the second week of the test was intended to compare the performance of various commercially available Method 17 filter holders, a discussion of their relative merits will be presented here. The statistical evaluation of their performance with regard to particulate determination is included in the next section.

TABLE V. PARTICULATE RECOVERED POSTCLEANUP/CLEANUP
mg

	1A	1B	1C	1D	2A	2B	2C	2D	8A	8B	8C	8D
Nozzle, probe, M-5 filter front-half		1.1		4.4	2.8		0.4		1.9		2.1	
Nozzle, M-17 filter front	1.7		1.4			1.3		2.0		1.9		3.0
M-17 back, probe, M-5 front	1.9		1.4			2.3		1.5		3.1		1.3

Three Method 17 filter holders were evaluated: Nutech, Misco and BGI. The Nutech filter holder was quite heavy and somewhat cumbersome to assemble. The substitution of hex-head bolts for the slotted-head bolts used in assembling the filter holder permitted the use of a wrench instead of a screwdriver. This substitution speeded up the assembly and provided a means of more even tightening of the assembly to prevent leaks. Sample recovery with the Nutech is relatively straightforward, but care must be exercised in disassembly for sample recovery, since that portion of the filter in contact with the gasket adheres to the gasket quite strongly. The portion of the filter supported by the sintered metal support plate breaks free, and the balance of the filter must be scraped from the gasket with a spatula. With careful recovery techniques, this filter holder appears to give good results.

The Misco filter holder is more compact than the Nutech and is easier to assemble. However, two shortcomings were reported by the field crew. Due to the relatively small filtration area available with the Misco, the pressure drop across the filter increases more rapidly during a run than with the other two filter holders. During the two final runs of the test, the ESP was operating in an upset condition. The particulate loading was high (ca. 125-200 mg/dscm), and the Misco filters blinded off to such an extent that isokinetic sampling could not be maintained for one hour. Both these runs were terminated at the point that isokinesis could not be maintained. The second problem involves the Teflon gaskets used in the Misco filter holder. The three wing nuts used to secure the assembly can be tightened finger-tight before a run, and the gaskets will provide a leak-free seal. However, due to the high coefficient of thermal expansion and plastic flow characteristics of Teflon, the gaskets deform upon heating to about 180 °C (350 °F). As a result of this deformation, the gaskets tend to leak upon cooling. More important than these leaks on cooling is the fact that the filter is subjected to fairly high pressure by the gasket due to the expansion of the gasket on heating. The glass fiber filter material is pressed into the surface of the gasket and is extremely difficult to remove during sample recovery. Care must be exercised to avoid scraping Teflon off the face of the gasket while trying to remove the filter material with a spatula. This problem made sample recovery from the Misco filter holder more difficult than for either of the other two filter holders evaluated. Use of alternative gasket materials should be considered when using the Misco unit at elevated temperatures.

The BGI thimble filter holder was the lightest, easiest to clean, and easiest to assemble of the three units evaluated. Since it is only slightly larger in diameter than a standard Method 5 probe, it is easy to insert and withdraw through a standard four-inch sample port. The major problem encountered with the BGI involves the thimble filter rather than the filter holder itself. The inside diameters of the lot of thimble filters used were not uniform. As a result, the fit of the thimble onto the thimble mounting tube of the filter holder ranged from snug to loose. The possibility of particulate matter bypassing the filter exists for those thimbles which do not fit tightly. During the portion of the test involved with filter holder evaluation, no backup filters were used on the Method 17 trains. However, during sample recovery, the glass probe liners used behind the Method 17 filters were examined visually for particulate matter. At the end of Run 11, Train D, equipped with the BGI thimble, was observed to have considerable particulate material in the probe liner. The calculated Method 17 C_S value for this run was approximately 50 percent of the C_S values for the other two Method 17 determinations and the Method 5 determination in Run 11. Either some modification of the filter holder clamp or closer quality control during filter manufacture to assure a leak-free fit of the thimble into the holder should be considered.

III. RESULTS

The data from the test runs were submitted to statistical evaluation. For Method 17, 9 mg/dscm were added to the observed particulate loading as specified in the Test Methods and Procedures section of the Federal Register pertaining to Kraft mills to obtain the adjusted particulate concentration. The results from each week of the test are treated separately below.

A. WEEK 1

The particulate loadings from Runs 3 through 8 were used to evaluate Method 17 (adjusted) and Method 17 plus back-half catch in comparison with Method 5 for particulate determination. The first two runs were not considered representative due to the problems encountered and are not included in these analyses. The two evaluations were treated separately, and the results are summarized below.

1. Method 5 versus Method 17 (Adjusted)

The sampling trains, A, B, C, and D, retained their integrity throughout the test. The only change was made at the probe, with either only a nozzle or a combination of nozzle and Method 17 filter attached. As such, the possibility exists that differences might exist between trains which could influence the results. This possibility of train-to-train differences is investigated using an analysis of variance (ANOVA) with factors of runs and trains. The run-by-train interaction is used as the error term for this ANOVA in the absence of replicate samples. The results are summarized in Table VI.

The between-trains factor is determined not to be significant at the 5-percent level, and the conclusion is that there is no need to account for the sample train in future analyses. In addition to this, a Friedman test,* a nonparametric equivalent to the two-way ANOVA, was run on these data to confirm the result. The test statistic was calculated at 7.04 with 3 degrees of freedom, which is not significant when compared to a table of the chi-squared distribution at a significance level of 5 percent.

Since the train factor was not significant, the mean square for error from the ANOVA was used as an estimate of sampling variability and the differences between the Method 5 and Method 17 results investigated. A t-test was used for this comparison, using the average

*Snedecor, G.W. and Cochran, W.G., *Statistical Methods*, 6th Edition Iowa State University Press, Ames., 1967.

TABLE VI. SUMMARY OF ANALYSES COMPARING METHOD 17 (ADJUSTED) AND METHOD 5

ANOVA Table

Source	df	SS	MS	F
Between Runs	5	3553.97	710.79	---
Between Trains	3	52.78	17.59	1.08*
Run x Train (error)	15	244.50	16.30	
Total	23	3851.25		

$\hat{\sigma}^2 = MS_{error} = 16.30$

Friedman Test

	Train			
	A	B	C	D
Rank sum	9.5	20	18	12.5

Test Statistic = 7.04, 3 df*

Means (mg/dscm)

Method 5	Method 17 (adjusted)
56.15	53.22

t statistic = 1.78, 15 df*

*Not significant at 5-percent level.

$F_{0.05}(3,15) = 3.29$, $X^2_{0.05}(3) = 7.81$, $t_{0.05}(15) = 2.13$

values for the 12 determinations by each method. A t-statistic is calculated according to the formula

$$t_c = \frac{\bar{x}_5 - \bar{x}_{17}}{\sqrt{\frac{2\hat{\sigma}^2}{12}}}$$

where $\bar{x}_5$ — average Method 5 results
$\bar{x}_{17}$ — average Method 17 results
$\hat{\sigma}^2$ — mean square for error from the ANOVA.

The calculated t was 1.78, which is not significant at the 5-percent level. The conclusion is, then, that the two methods gave equivalent results with the correction factor of 9 mg/dscm added to the result obtained from the particulate catch.

To estimate the variability associated with each method, a percent coefficient of variation was calculated for each run using the ratio of the standard deviation between the two results to the average value. The individual values obtained and the averages are shown in

Table VII. The average for Method 5 was 6.34 percent, while for Method 17, it was 7.99 percent.

TABLE VII. COMPARATIVE COEFFICIENTS OF VARIATION, METHOD 17 (ADJUSTED) VERSUS METHOD 5 (Percent)

Run	Method 17 (Adjusted)	Method 5
3	12.97	2.00
4	8.07	10.93
5	0.76	6.66
6	10.60	6.25
7	7.96	1.41
8	7.57	10.79
Avg	7.99	6.34

2. Method 5 versus Method 17 plus Back-Half Catch

The particulate loadings for Method 17 were added to the particulate catch from the probe wash and Method 5 backup filter to determine total particulate matter for each run. The concentrations calculated in this manner were analyzed in the same manner as the Method 17 data, and the results are presented below.

The ANOVA for investigation of a train effect is shown in Table VIII. As before, the F-ratio for between-train variability is insignificant, and no allowance for train differences is indicated. The Friedman test gave a test statistic of 6.40, which also is not significant, and it is concluded that no bias existed among the four trains.

The mean for the twelve Method 17 determinations with the back-half workup included was 56.05, compared to the Method 5 mean of 56.15 mg/dscm. The resultant t-statistic was calculated to be 0.05 and is clearly nonsignificant.

The percent coefficients of variation for these runs are shown in Table IX, along with their average value. The mean of 5.97 percent compares favorably to the Method 5 value of 6.34 percent.

B. WEEK 2

Using the eight runs from the second week of testing, the three types of Method 17 filters were compared both against each other and against the Method 5 data. The first analysis, as with the previous data, was to determine if any train-to-train bias existed which could influence the test results.

TABLE VIII. SUMMARY OF ANALYSES COMPARING METHOD 17 PLUS BACKUP CATCH AND METHOD 5

ANOVA Table

Source	df	SS	MS	F
Between Runs	5	3145.92	629.18	---
Between Trains	3	73.27	24.42	1.60*
Run x Train (error)	15	229.33	15.29	
Total	23	3448.52		

$\hat{\sigma}^2 = MS_{error} = 15.29$

Friedman Test

	Train			
	A	B	C	D
Rank Sum	11	19	19	11

Test Statistic = 6.40, 3 df*

Means (mg/dscm)

Method 5	Method 17 + back-half
56.15	56.05

t-statistic = 0.05, 15 df*

*Not significant at 5-percent level.

$F_{0.05}(3,15) = 3.29$, $X^2_{0.05}(3) = 7.81$, $t_{0.05}(15) = 2.13$

TABLE IX. COMPARATIVE COEFFICIENTS OF VARIATION, METHOD 17 PLUS BACKUP CATCH VERSUS METHOD 5 (Percent)

Run	Method 17 + Back-Half	Method 5
3	1.14	2.00
4	2.75	10.93
5	2.02	6.66
6	10.81	6.25
7	8.61	1.41
8	10.49	10.79
Avg	5.97	6.34

The ANOVA is summarized in Table X. The F-ratio for the between-trains component is less than one and clearly nonsignificant. The Friedman test for these data gave a test statistic of 1.35, which also is not significant. As a result, no train bias can be detected, and no allowance is made in the subsequent analyses for the sampling train used to collect the sample.

TABLE X. SUMMARY OF ANALYSES ON SECOND WEEK'S DATA

ANOVA Table

Source	df	SS	MS	F
Between Runs	7	83,275.08	11,896.44	---
Between Trains	3	239.98	79.99	<1*
Run x Train (error)	21	3,647.56	173.69	
Total	31	87,162.62		

$\hat{\sigma}^2 = MS_{error} - 173.69$

Friedman Test (train bias)

	Train A	B	C	D
Rank Sum	18	23	21	18

Test Statistic = 1.35, 3 df*

Friedman Test (filter type)

	Nutech	Misco	BGI
Rank Sum	16	16	16

Test Statistic = 0.00*

Means (mg/dscm)

Method 5	Nutech	Misco	BGI
77.49	78.23	78.60	68.56

$LSD_{.05} - 13.71$
Ho: $\mu_N + \mu_M + \mu_B - 3\mu_5 = 0$
Test Statistic = -1.57, 21 df*
*Not significant at 5-percent level.
$F_{0.05}(3,21) = 3.07$, $X^2_{0.05}(3) = 7.81$, $t_{0.05}(21) = 2.08$

The first examination of the Method 17 trains was made using the Friedman test. The factors in this analysis were the runs and the type of filter. The values for the three Method 17 trains were ranked across each run and the rank sums computed. The rank sum was calculated to be 16 for each of the three trains, and no difference can be detected.

A parametric approach is also used. A Least Significant Difference (LSD) is calculated according to the formula

$$LSD = t_{.05}(21)\sqrt{\frac{2\hat{\sigma}^2}{n}}$$

where

$\hat{\sigma}^2$ — mean square for error from the ANOVA

and n = 8, the number of runs used to obtain the mean for each type of filter.

Substituting gave an LSD of 13.71 for these data. The averages were 78.23, 78.60, and 68.56 mg/dscm for the Nutech, Misco, and BGI filters, respectively. Since the range of these means, 10.04, was less than the LSD, no difference among the filter types can be detected.

The Method 5 mean for these runs was 77.49 and shows good agreement with both the Nutech and Misco average values. To test for consistency between the two methods, a contrast among the means is tested. The hypothesis is

$$Ho: \mu_N + \mu_M + \mu_B - 3\mu_5 = 0$$

where

μ_N — mean Method 17 result using Nutech filters

μ_M — mean Method 17 result using Misco filters

μ_B — mean Method 17 result using BGI filters

and μ_5 — mean Method 5 result.

The test statistic is calculated as

$$t_c = \frac{\bar{x}_N + \bar{x}_M + \bar{x}_B - 3\bar{x}_5}{\sqrt{\frac{\Sigma c_i^2}{n}\hat{\sigma}^2}}$$

where

$\bar{x}_N \ldots\ldots \bar{x}_5$ — sample means corresponding to the μ's above

c_i — coefficient of the means in the null hypothesis

n — number of runs

and $\hat{\sigma}^2$ — mean square for error from the ANOVA.

Substituting gives a calculated t-statistic of -1.57. This does not exceed the tabled t-value for 21 degrees of freedom at the 5-percent level, and the results may be considered equivalent.

IV. SUMMARY AND CONCLUSIONS

The results of the statistical analysis of data from the first week of testing indicate that Method 5 and Method 17 gave equivalent particulate results at a Kraft pulp mill recovery furnace at the 95-percent confidence level when the correction factor of 9 mg/dscm specified in the Federal Register was added to the Method 17 determination. Additionally, the Method 5 versus Method 17 plus backup filter data indicated that the particulate loadings obtained on all four trains were equivalent throughout the first week of testing. The variability of Method 5 and Method 17 (Adjusted) was good, being 6.34 percent and 7.99 percent, respectively.

The results of the statistical analysis of data from the second week of testing indicate that the particulate loadings determined with all three Method 17 filter assemblies were equivalent at the 95-percent confidence level. The Method 17 (Adjusted) particulate loadings determined during the second week of testing were also equivalent to the Method 5 result at the 95-percent confidence level.

APPENDIX A
RUN PARAMETERS SUMMARY

Appendix A
RUN PARAMETERS

N—Nutech
M—Misco
B—BGI

Run #	ΔP_S	ΔH	T_S	T_M	$V_{M(STD)}$	B_{WO}	% ISOKINETIC	V_S	NOZZLE ID	% CO_2	% O_2	M_S	TRAIN
3 A	0 469	1.582	350	109	38.228	0.291	102.9	46.27	0.300	14.5	3 7	26 83	17N
B	0.469	1.604	350	109	38.783	0.291	99.7	46.27	0.307				5
C	0.469	1.548	350	109	38.304	0.287	97.6	46.27	0.306				17N
D	0.469	1.496	350	107	38.450	0.282	101.0	46 27	0.302				5
4 A	0.592	2.058	350	118	42.874	0.297	102.9	51.75	0.302	14.5	3.5	27.11	5
B	0.592	2.033	350	119	42.281	0.297	98 7	51.75	0.306				17N
C	0.592	1.982	350	120	42.577	0.297	98.8	51.75	0.307				5
D	0 592	1.857	350	116	42.326	0.298	103.0	51.75	0.300				17N
5 A	0.582	1.972	353	110	42.282	0.296	103.0	51.68	0.300	14.8	3.2	26.79	17N
B	0.582	1.972	353	109	43.217	0.292	100.0	51.68	0.307				5
C	0.582	1.877	353	110	41 786	0.294	97.5	51.68	0.306				17N
D	0.582	1.819	353	111	41.773	0.301	101.1	51.68	0.302				5
6 A	0.582	1.973	352	118	41.943	0.295	102.2	51.64	0.302	15.5	2.3	26.86	5
B	0.582	1.973	352	117	41.495	0.300	97.8	51.64	0.306				17N
C	0 582	1.868	352	116	41 710	0.295	97.1	51.64	0.307				5
D	0.582	1.817	352	113	41.944	0.295	102 2	51.64	0.300				17N
7 A	0.738	2.492	369	109	47.605	0.288	102 9	58.56	0.300	14 9	3 1	26 91	17N
B	0.738	2.492	369	107	47.538	0.288	98 1	58.56	0.307				5
C	0 738	2.410	369	107	45.692	0.286	94.6	58.56	0.306				17N
D	0.738	2.257	369	107	44.661	0.289	95 5	58.56	0.302				5

(continued)

Appendix A (continued)
RUN PARAMETERS

N—Nutech
M—Misco
B—BGI

Run #	ΔP_S	ΔH	T_S	T_M	$V_{M(STD)}$	B_{WO}	% ISOKINETIC	V_S	NOZZLE ID	ORSAT (no CO found) % CO_2	% O_2	M_S	TRAIN
8 A	0.669	2.261	360	112	45.518	29.6	102.8	55.56	0.302	15 8	1.9	26.87	5
B	0.669	2.261	360	116	45.019	29.6	98.8	55.56	0.306				17N
C	0.669	2.203	360	118	44.876	29.6	97.8	55.56	0.307				5
D	0.669	2.051	360	117	44.164	29.8	101.2	55.56	0.300				17N
9 A	0.686	2.328	361	107	46.774	28.0	102.9	56.20	0.300	13.5	4.6	26.85	17N
B	0.686	2.328	361	109	45.120	28.7	95 6	56.20	0.307				5
C	0.686	2.252	361	117	45.789	25.6	95.8 (99.4)[1]	56.20	0.305				17M
D	0.686	2.160	361	116	45.731	28.1	99.6	56.20	0.302				17B
10 A	0.551	1.850	353	104	41 431	27.7	100.1	49.93	0.302	14 9	3 0	27 08	17B
B	0.551	1.850	353	108	41.310	26.3	96.0[2]	49.93	0.305				17M
C	0.551	1.817	353	107	41.631	27.7	97.3	49 93	0.307				5
D	0.551	1.751	353	106	41 764	27.7	102.2	49.93	0.300				17N
11 A	0.742	2.501	361	114	47.522	29.8	103.1	58.52	0 300	14.3	3.7	26.79	17N
B	0.742	2.501	361	117	46.816	29.5	96.6	58.52	0.307				5
C	0.742	2.430	361	116	47 129	29.0	97.9	58.52	0.305				17M
D	0.742	2.375	361	112	47.830	28.9	101 2	58.52	0.302				17B
12 A	0.718	2.398	370	112	46.857	29 5	102 3	57.93	0.302	14 3	3 7	26 79	5
B	0 718	2.398	370	118	45.851	29.2	96.4	57.93	0.307				17M
C	0.718	2.318	370	114	46.442	29 1	98.8	57 93	0 305				17B
D	0 718	2.189	370	112	46.230	29.5	102.2	57.93	0.300				17N

(continued)

Appendix A (continued)
RUN PARAMETERS

N—Nutech
M—Misco
B—BGI

Run #	ΔP_S	ΔH	T_S	T_M	$V_{M(STD)}$	B_{WO}	% ISOKINETIC	V_S	NOZZLE ID	ORSAT (no CO found) % CO_2	% O_2	M_S	TRAIN
13 A	0.610	2.003	356	107	42.674	28.5	100 3	52.90	0.300	14.3	4.1	26.90	17N
B	0.610	2.134	356	111	43.922	29.0	100.7	52.90	0.305				5
C	0.610	2.073	356	110	44.193	28.3	98 9	52.90	0.307				17M
D	0.610	1.908	356	111	43.403	28 3	100 5	52.90	0.302				17B
14 A	0.582	1.933	355	116	41 870	29 7	101.2	51 60	0.302	14.3	4.1	26.90	5
B	0.582	2.024	355	118	41.921	30.0	99.7	51.60	0.305				17M
C	0.582	2.012	355	116	42.108	29.7	98.4	51.60	0.307				17B
D	0.582	1.779	355	111	41.576	24.0	94.0	51 60	0.300				17N
15 A	0.692	2.248	370	105	38.701[3a]	0.282	99 7	56.58	0 300	15.2	2.8	27.02	17N
B	0.692	2.430	370	104	39.712	0.280	99 4	56.58	0.307				5
C	0.692	2.298	370	105	37.418	0.253	91.5	56.58	0.305				17M
D	0.692	2.136	370	107	38.488	0 281	99 8	56 58	0.302				17B
16 A	0.688	2.228	367	110	36.456[3b]	0.278	98.6	56.27	0.302	15.2	2.8	27 02	17B
B	0.688	2.418	367	110	36.815	0.276	95 9	56.27	0.307				17M
C	0.688	2.279	367	109	37.144	0.275	98 1	56.27	0.305				5
D	0.688	2.121	367	112	36.715	0.284	101.0	56.27	0 300				17N

1 Leak detected during post-run leak check. Rate was 0.08 cfm (4 × max allowable)
2 Leak detected during post-run leak check. Rate was 0 03 cfm (1 5 × max allowable)
3a Misco filter blinded off at 50 min 39 sec
3b Misco filter blinded off at 45 min

Δp, ΔH – in H_2O
T_s, T_m – °F
V_m – ft^3

V_s – fps

APPENDIX B

A COMPARATIVE STUDY OF METHOD 17 WITH METHOD 5 AT A GRAIN ELEVATOR[a]

H. F. Hamil

I. INTRODUCTION

Prior to proposing new source performance standards for grain elevators, the Emissions Standards and Engineering Division of the Office of Air Quality Planning and Standards obtained source emission data on approximately twelve grain elevators. This work was performed to characterize the emissions from grain elevators and to determine an emission rate based upon best available control technology. Most, if not all, of these source tests were conducted using EPA Method 5. Based upon the results of this testing, a particulate emission standard of 0.023 grams per dry standard cubic meter was set. This level of particulate emissions was achievable by use of baghouse collectors.

When the particulate emission standard for grain elevators was proposed in the Federal Register, the reference method listed for determination of particulate emissions was not Method 5, but Method 17, an in-stack filtration method.

The Quality Assurance Branch, Environmental Monitoring and Support Laboratory (QAB/EMSL) initiated a comparative study of EPA Methods 5 and 17 in a grain elevator to determine their relative performance in collecting samples simultaneously. This study was conducted at a baghouse collector-equipped grain elevator. This facility operated with particulate emissions which were near the proposed standard. The results of the QAB comparative testing indicated that Method 5 collected about 20 percent more particulates than did Method 17 when equivalent volumes of stack gas were sampled. It should be noted that this relatively large percentage difference amounted to an average difference of only 6 milligrams between the two methods. Such small variations in mass of particulate collected were relatively unimportant in the past for sources with higher standards. With the lower proposed standards, these differences should be resolved, since a review of the data collected by the Emission Measurement Branch for the purpose of proposing the grain elevator standard indicated that the average particulate catch by Method 5 in these tests was 50 milligrams. Additional comparative testing at a grain elevator was considered warranted.

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[a]Report prepared by Dr. H. F. Hamil and Mr. R. E. Thomas of Southwest Research Institute, San Antonio, TX, under EPA Contract 68-02-2489 and under the direction of Mr. Michael C. Osborne, EPA, Research Triangle Park, NC.

II. TEST PLAN AND SITE

A. Test Plan

Week 1. Sampling was accomplished with a four-probe/one-pitot tube assembly. The sample nozzles were at the corners of a square array 6 centimeters on a side. The one pitot tube was centrally located between the four sample nozzles. Two Method 5 trains were operated diagonally opposite two Method 17 trains, as shown in Figure 1. Glass-lined probes with new glass liners were used on all four trains, with Method 5 filters installed as backups on the Method 17 probe-filter assemblies to collect any particulate not collected by the Method 17 filter. BGI thimbles and holders were used as representative Method 17 filters.

A complete cleanup of all trains was performed prior to the first run. Upon completion of the sample recovery, including retention of the impinger catch and train back-half cleanup, an additional complete train cleanup was performed to insure no particulate remained in the sample trains. In this fashion, a complete train cleanup, separate from sample recovery, preceded each sampling run, and an additional train cleanup was performed after the last run. The wash solutions from these additional cleanups were worked up to determine residual particulate matter in the trains after normal sample recovery. Six runs were made during week 1 by this sampling procedure.

Week 2. The test plan for the second week incorporated one major modification. This modification consisted of moving the Method 17 filter out-of-stack to the same location in the trains as a Method 5 filter in order to evaluate the performance of both Method 5 and 17 filters under equivalent conditions (Figure 2). No backup filters were used. All other aspects of the tests were identical to the first week's tests described above. Five runs were made the second week.

During both weeks of the test, the positions of the sampling probes were rotated clockwise in order to avoid any spatial bias.

B. Test Site

The site selected for this test was a grain elevator operated by the Cargill Company at Reserve, Louisiana. This facility was equipped with baghouse collectors and operated at very low particulate emission levels. Pretest sampling to evaluate the site indicated that emissions were in the range of 1.27×10^{-2} to 1.71×10^{-2} grams per dry standard cubic meter, as determined by use of EPA Method 5. The baghouse collector selected for the test was located on the conveyer used in unloading grain barges. A stack extension was installed on the baghouse outlet to bring the exhaust gas to ground level. This extension fed into

a test section from which the samples were taken. A sketch of the site complete with stack extension is shown in Figure 3.

III. FIELD TESTING

Pretest preparations required by the methods were carried out and included all specified equipment calibrations. The pitot tube calibration was conducted with the full four-probe assembly in order to assure that no bias to the velocity head measurements by the four probes would be introduced. The test was conducted the first two weeks of May 1977, commencing on May 2 and being completed on May 13. The test plan originally called for five runs each week. The initial run on May 2 was terminated prematurely by a plant shutdown caused by equipment malfunction. An additional five valid runs were conducted the first week, and five valid runs the second week. Since the particulate loading at the site was quite low, it was considered necessary to sample for three hours in order to obtain ca 50 milligrams of particulate. The first run was considered invalid due to a run time of about 1.5 hours. Other criteria for validity were passage of initial and final leak checks and isokinetic sampling within allowable limits of variation. During the course of the two-week test period, the facility was unloading wheat, corn, and soybeans, which resulted in considerable variation in the particulate loading at the baghouse outlet. Specific attention by test personnel to plant operation was required, since the unloading operations were intermittent, even though the conveyor ran continuously. To assure that sufficient particulate matter was collected, sampling was conducted only when the conveyor was loaded and was interrupted when the conveyor was running empty.

Only one problem of significance occurred with the test equipment. One control console developed a leak in the pump oiler. This console was replaced with a backup unit. During the sample workup in the laboratory, two other problems more serious in nature were encountered.

The first involved the Method 17 filters (BGI thimbles). Prior to the test, the filters were desiccated to constant weight according to the method (less than 0.5 mg change on two subsequent weighings). Since the particulate matter is collected on the outside of the BGI filter, some means of protecting the used filter to prevent loss of particulate during desiccation and weighing is required. Each thimble was placed in a glassine envelope for this purpose. The thimble and envelope was desiccated to constant weight as a unit. Some difficulties were encountered in the pretest drying to constant weight. The filter-envelope combination appeared to pick up moisture fairly rapidly on the balance pan, even though the weighings were conducted in a constant temperature-constant humidity room. However, after several

days the filters were brought to constant weight. When the filters used in field sampling were desiccated to constant weight, some were found to weigh less than the pretest tare weights. Only one unused Method 17 filter was available. This filter was desiccated to constant weight over a period of ten days and was found to weigh 37.2 mg (0.847 wt %) less than the original tare weight. It appears that although in the pretest weighing of the Method 17 filters constant weight was achieved, the filters were not completely dry. Subsequent investigations showed that the problem was not due to the filter thimbles but due to the glassine envelopes. These envelopes, intended for photographic negative storage, have incorporated in them a certain amount of a desiccant and will dry to a constant weight (less than 0.5 mg change on two subsequent weights) while still retaining considerable moisture. Continued desiccation will eventually remove this water.

Based upon the weight change of the unused Method 17 filter, a correction factor (0.9916 x original tare weight) was used to correct the tare weights of the filters. It is realized that this is not a particularly satisfactory solution to the problem, but it is the only means available to try to salvage the data. The Method 17 particulate catch weights are uncertain, and quantitative comparisons of particulate loadings by the two methods are not possible, as will be discussed in the next section.

The second problem concerned the amount of material which passed through the filtration system and was collected in the back-half (impinger section) of both the Method 5 and Method 17 determinations. The particulate which passed the filter was determined by placing the impinger water in a sample bottle along with an acetone wash of the filter holder back-half, the fritted glass filter support, the impingers and connecting glassware. Particulate mass was determined by evaporation of this combined acetone-water mixture to dryness. After evaporation to dryness, small pieces of glass from the fritted glass filter support were observed in most of the samples. As a result, the impinger residue weights are somewhat inflated.

IV. RESULTS

The run parameters for the eleven runs are presented in Table I. The first run is of questionable value due to the short sampling period. For Runs 2 through 11, the Method 17 runs are designated with an asterisk. Runs 2 through 6 were made the first week. In Table I, two particulate loadings (Cs) were calculated for the Method 17 runs; under the column headed Cs is the value calculated on the total particulate catch (Method 17 filter assembly plus backup filter assembly), while the column headed Method 17 Cs is calculated using the particulate catch from the Method 17 filter assembly. The Cs values for Method 5 are calculated in the normal manner. The within-run agreement between the

Method 5 determinations is quite good over the range of particulate loadings studied (9-45 mg/m^3). Unfortunately, the Method 17 results cannot be compared quantitatively to the Method 5 results due to the uncertainty in the Method 17 results introduced by the problem with the thimble tare weights. However, a comparison of the two particulate loadings calculated for each Method 17 run provides some useful information. The difference in these two Cs values gives a measure of the amount of particulate which passed through the Method 17 filter and was collected in the backup Method 5 probe, filter and filter holder. An examination of the data revealed that a fairly constant amount of particulate was collected in the backup train over the range of particulate loading studied. The mean value for particulate collected in the backup train was 3.73 mg/m^3 with a standard deviation of 0.56 mg/m^3. The lowest value was 2.52 mg/m^3 and the highest value was 4.17 mg/m^3. Since the amount of particulate remained relatively constant over the range of particulate loading studied, the percent error introduced in a Method 17 determination would be greatest for low particulate loading and decrease as the loading increased. This apparent increase in filtration efficiency with increased particulate loading may reflect the effect of a filter cake buildup on the filter surface which results in more efficient particulate retention as the run progresses.

The results of the second week's runs to assess the relative performance of Method 5 and Method 17 filters located in the same position in the train (out-of-stack) are inconclusive due to the uncertainty in the Method 17 results.

The results of the impinger train workup to determine the filtration efficiency of the entire filter assembly are inconclusive due to contamination of the back-half catch with glass particles from the Method 5 filter supports.

The additional cleanup procedures before the first run, between each pair of runs and after the last run were performed to assess the amount of particulate which remained in the train after a normal sample recovery. A total of 48 individual train cleanups were performed during the course of the two-week test. The mean value of particulate recovered from the sample nozzle, probe and filter holder on these cleanups was 4.01 mg with a standard deviation of 1.9 mg, indicating that very little material is left in the sample train if good sample recovery procedures are followed.

The weights of particulate caught in each run and the location of the catch in the train components are presented in Table II. The values for impinger train residue for each run are presented in Table III, and the values for particulate recovered in the additional train cleanup procedures are presented in Table IV.

V. CONCLUSIONS

Despite the fact that a quantitative comparison of Methods 5 and 17 could not be made, the data from the first week of the test indicate that a significant amount of particulate (3.75 mg/m^3) passes through the Method 17 filter. The amount of particulate passing the filter was relatively constant for particulate loadings in the range of 10-45 mg/m^3. No information on the amount of particulate passing the filter at higher loadings is available. It is possible that the amount of particulate passing the filter may be site specific in that it could be affected not only by particulate loading but also by the physical and chemical properties of the particulate (size distribution, composition, etc.).

The data from the second week of the test did not allow an assessment of the relative filtration efficiency of the two filter assemblies operated in the same mode (out-of-stack). However, review of the impinger train residue values for Runs 7 through 11 does not show gross discrepancies in the Method 17 versus Method 5 results. It appears possible that the Method 5 probe acts as a precollector and has an effect on filter efficiency.

These conclusions are necessarily speculative due to the uncertainty in the Method 17 data. Further investigations are needed to confirm or refute these tentative findings.

One observation of importance in the use of the BGI thimble filters should be noted. The thimbles were seated in the filter holder merely by friction fit. No securing device was used to hold the filters in place. There was sufficient variation in the inside diameter of the thimbles in the lot used that the fit of the filter in the filter holder varied from snug to loose. There is firm evidence on one run (7C) that considerable particulate bypassed the filter and was collected in the impinger train, which was very milky in appearance at the end of the run. In this run, 103 mg of particulate was collected by the probe and filter and 48.2 mg by the impingers, compared to 174.6 mg by the probe and filter and only 8.1 mg by the impingers on Run 7A, the other Method 17 determination in that run. Some means of assuring that particulate blow-by does not occur should be incorporated in the use of BGI filters.

Table I
RUN PARAMETERS

Run No.	ΔP(avg)	ΔH(avg)	T_s(avg)	T_m(avg)	V_m(std)	V_s	θ	B_{ws}	$P_{bar}=P_s$	Nozzle No.	I	C Factor	C_s	Method 17 C_s
1A	0.131	2.514	540.4	540.6	82.101	20.46	95	0,0246	30.06	2	105.1	1.16	27.78	
1B	0.131	2.513	540.4	560.0	78.285	20.46	95	0.0257	30.06	3	100.7	1.15	19.76	
1C	0.129	2.410	540.6	561.3	79.663	20.31	95	0.0247	30.06	1	104.6	1.13	14.27	
1D	0.129	2.236	540.6	561.5	78.999	20.31	95	0.0262	30.06	4	104.4	1.05	18.24	
								0.0253						
2A	0.144	2.738	539.8	539.0	159.563	21.44	180	0.0216	30.16	1	103.9	1.15	9.34	
2B*	0.144	2.731	539.8	562.0	153.387	21.44	180	0.0217	30.16	2	98.2	1.14	11.79	7.83
2C	0.145	2.604	540.2	563.2	154.363	21.49	180	0.0215	30.16	3	100.9	1.10	11.48	
2D*	0.145	2.471	540.2	564.2	155.300	21.49	180	0.0210	30.16	4	101.4	1.04	6.16	3.64
								0.0214						
3A*	0.146	2.754	548.0	542.1	159.668	21.72	180	0.0210	30.16	1	104.1	1.15	--	--
3B	0.146	2.831	548.0	573.4	153.562	21.72	180	0.0210	30.16	2	98.4	1.19	9.96	
3C*	0.146	2.822	548.0	572.5	159.528	21.72	180	0.0210	30.16	3	104.6	1.18	6.75	2.94
3D	0.146	2.579	548.0	573.0	157.025	21.72	180	0.0210	30.16	4	103.0	1.07	9.51	
								0.0210						
4A	0.142	2.685	550.3	547.2	156.754	21.49	180	0.0178	30.08	1	103.7	1.14	19.01	
4B*	0.142	2.807	550.3	574.0	153.268	21.49	180	0.0156	30.08	2	99.5	1.20	14.37	10.41
4C	0.142	2.671	550.3	571.7	156.626	21.49	180	0.0170	30.08	3	102.8	1.20	18.08	
4D*	0.142	2.500	550.3	574.6	155.316	21.49	180	0.0162	30.08	4	103.8	1.10	16.46	12.71
								0.0167						
5A*	0.142	2.767	549.5	547.8	158.263	21.48	180	0.0196	30.08	1	104.8	1.19	13.86	10.8
5B	0.142	2.854	549.5	576.2	154.107	21.48	180	0.0200	30.08	2	100.4	1.24	13.01	
5C*	0.142	2.721	549.5	572.6	157.230	21.48	180	0.0187	30.08	3	104.6	1.18	9.32	5.05
5D	0.142	2.537	549.5	575.2	155.693	21.48	180	0.0186	30.08	4	103.6	1.12	12.72	
								0.0192						
6A	0.120	2.275	557.1	547.5	143.868	19.92	180	0.0177	29.94	1	104.4	1.17	35.12	
6B*	0.120	2.436	557.1	574.5	145.323	19.92	180	0.0171	29.94	2	103.6	1.24	30.20	26.75
6C	0.120	2.190	557.1	575.7	140.871	19.92	180	0.0162	29.94	3	102.6	1.18	34.54	
6D*	0.120	2.042	557.1	576.5	139.586	19.92	180	0.0170	29.94	4	101.8	1.09	35.95	31.77
								0.0170						

Table I (Cont'd)
RUN PARAMETERS

Run No.	ΔP(avg)	ΔH(avg)	T_s(Avg)	T_m(avg)	V_m(std)	V_s	θ	B_{ws}	$P_{bar}=P_s$	Nozzle No.	I	C Factor	C_s	Method 17 C_s
7A*	0.114	2.133	553.9	549.2	140.158	19.36	180	0.0170	29.94	1	104.0	1.17	43.99	
7B	0.114	2.274	553.9	570.3	142.054	19.36	180	0.0177	29.94	2	103.7	1.24	43.67	
7C*	0.114	2.174	553.9	576.2	140.565	19.36	180	0.0173	29.94	3	104.9	1.18	25.87	
7D	0.114	1.988	553.9	577.0	138.829	19.36	180	0.0175	29.94	4	103.6	1.09	45.15	
								0.0174						
8A	0.117	2.144	540.2	533.1	141.611	19.38	180	0.0135	29.92	1	102.0	1.14	36.53	
8B*	0.117	2.229	540.2	551.9	142.662	19.38	180	0.0113	29.92	2	100.9	1.18	22.70	
8C	0.117	2.218	540.2	548.9	145.379	19.38	180	0.0112	29.92	3	105.1	1.18	34.20	
8D*	0.117	2.085	540.2	554.4	143.292	19.38	180	0.0133	29.92	4	103.8	1.11	27.60	
								0.0123						
9A*	0.124	2.318	547.9	542.4	147.253	20.09	180	0.0131	29.92	1	103.8	1.14	26.38	
9B	0.124	2.359	547.9	566.8	144.221	20.09	180	0.0133	29.92	2	100.0	1.15	26.93	
9C*	0.124	2.292	547.9	562.9	145.973	20.09	180	0.0122	29.92	3	103.4	1.13	18.43	
9D	0.124	2.122	547.9	568.5	145.232	10.09	180	0.0123	29.92	4	103.1	1.05	27.01	
								0.0127						
10A	0.126	2.432	540.9	537.9	150.958	20.04	180	0.0201	30.16	1	105.2	1.17	24.86	
10B*	0.126	2.445	540.9	558.0	148.174	20.04	180	0.0192	30.16	2	101.4	1.19	18.66	
10C	0.126	2.401	540.9	555.2	150.401	20.04	180	0.0196	30.16	3	105.3	1.14	25.78	
10D*	0.126	2.198	540.9	560.8	148.635	20.04	180	0.0191	30.16	4	104.1	1.06	16.65	
								0.0195						
11A*	0.115	2.166	547.0	544.1	141.755	19.24	180	0.0175	30.20	1	103.6	1.17	33.20	
11B	0.115	2.196	547.0	566.7	140.134	19.24	180	0.0178	30.20	2	100.8	1.19	42.73	
11C*	0.115	2.082	547.0	561.5	140.659	19.24	180	0.0164	30.20	3	103.3	1.14	37.83	
11D	0.115	1.980	547.0	566.3	140.628	19.24	180	0.0180	30.20	4	103.4	1.06	43.54	
								0.0174						

* Method 17 runs.

$C_p = 0.84$ $A_{N1} = 6.874 \times 10^{-4} ft^2$ $A_{N2} = 6.99 \times 10^{-4} ft^2$ $A_{N3} = 6.835 \times 10^{-4} ft^2$ $A_{N4} = 6.835 \times 10^{-4} ft^2$

$\Delta P_u \Delta H$ in H_2O T_s, T_m ^oR V_s fps V_m ft^3 θ min P_{bar} in Hg C_s mg/m^3 A_n ft^2

Table II
PARTICULATE CATCH, mg

Run	Train	Method 5 Filter	Method 17 Filter	Method 17 Nozzle and Filter Holder	Method 5 Probe & Front of Filter Holder	Impinger Train and Back of Filter Holde
Results of First Week's Test						
2	A	26.9	---	---	15.3	32.8
	B	2.8	20.3	13.7	14.4	24.6
	C	29.3	---	---	10.9	17.3
	D	1.0	5.7	10.3	10.1	17.1
3	A	1.9	-8.2	7.4	17.4	44.1
	B	23.2	---	---	20.1	40.2
	C	-0.2	6.5	6.8	17.4	20.3
	D	22.2	---	---	20.1	18.4
4	A	52.3	---	---	32.1	22.3
	B	-0.3	26.9	18.9	17.4	19.1
	C	49.0	---	---	31.2	16.4
	C	2.5	42.9	13.0	14.0	13.5
5	A	0.5	33.2	15.2	13.2	19.5
	B	34.3	---	---	22.5	16.6
	C	-1.3	6.6	15.9	19.0	19.8
	D	35.4	---	---	20.7	25.5
6	A	105.9	---	---	37.2	14.2
	B	-0.2	89.9	20.2	14.4	6.1
	C	105.6	---	---	32.2	11.5
	D	0.6	102.9	22.7	15.9	12.4

Table II (Cont'd)
PARTICULATE CATCH, mg

Run	Train	Method 5 Filter	Method 17 Filter	Method 17 Nozzle & Probe & Filter Holder	Method 5 Nozzle & Probe & Front of Filter Holder	Method 5 Back of Holder & Impinger Train	Method 17 Impinger Train
Results of Second Week's Test							
7	A	---	129.0	45.6	---	---	8.1
	B	146.2	---	---	29.5	11.1	---
	C	---	72.3	30.7	---	---	48.2
	D	146.2	---	---	31.3	10.7	---
8	A	114.9	---	---	31.6	8.7	---
	B	---	55.0	36.7	---	---	8.5
	C	109.7	---	---	31.1	9.3	---
	D	---	95.0	17.0	---	---	5.1
9	A	---	58.4	27.2	---	---	7.4
	B	82.7	---	---	27.3	10.2	---
	C	---	54.0	22.2	---	---	7.7
	D	79.5	---	---	31.6	8.8	---
10	A	85.8	---	---	20.5	10.0	---
	B	---	62.8	15.5	---	---	4.5
	C	86.2	---	---	23.6	12.8	---
	D	---	53.4	16.7	---	---	6.8
11	A	---	112.0	21.3	---	---	7.6
	B	136.1	---	---	33.5	12.9	---
	C	---	133.7	17.0	---	---	8.8
	D	152.2	---	---	21.2	18.4	---

Table III
IMPINGER TRAIN RESIDUE

1A	1B	1C	1D
16.3*	10.1*	6.8*	10.1*

2A	2B	2C	2D
32.8*	24.6*	17.3*	17.1*

3A	3B	3C	3D
44.1*	40.2*	20.3*	18.4*

4A	4B	4C	4D
22.3*	19.1*	16.4*	13.5*

5A	5B	5C	5D
19.5*	16.6*	19.8*	25.5*

6A	6B	6C	6D
14.2*	6.1*	11.5*	12.4*

7A	7B	7C°	7D
8.1	11.1*	48.2	10.7*

8A	8B	8C	8D
8.7*	8.5	9.3*	5.1

9A	9B	9C	9D
7.4*	10.2	7.7*	8.8

10A	10B	10C	10D
10*	4.5	12.8*	6.8

11A	11B	11C	11D
7.6	12.9*	8.8	18.4*

* NOTE: High values may be a result of washing the glass frit behind the Method 5 filter. In many instances the glass frit was observed to contribute small fragments of glass to the acetone wash.

° NOTE: Run 7C had a visible residue probably due to a poor fitting of the thimble inside the Method 17 thimble holder

____ No Method 5 filter holder in train.

CONCLUSION: Acetone and water residues from the trains without the Method 5 filter holders are less than 10 mg in almost all instances. Therefore, contribution from the glass frit is highly probable.

Table IV

POST CLEANUP - CLEANUP

1A	1B	1C	1D
3.0	3.1	7.4	4.8

2A	2B	2C	2D
0.5	5.0	1.4	1.8

3A	3B	3C	3D
2.1	2.5	4.5	2.2

4A	4B	4C	4D
5.7	6.3	3.1	5.5

5A	5B	5C	5D
5.2	3.0	5.9	4.2

6A	6B	6C	6D
1.9	2.3	6.8	5.9

7A	7B	7C	7D
1.7	3.6	2.4	4.6

8A	8B	8C	8D
3.2	1.8	3.7	3.9

9A	9B	9C	9D
1.8	4.8	1.8	3.1

10A	10B	10C	10D
5.2	2.3	3.9	2.6

11A	11B	11C	11D
4.0	5.2	4.1	5.6

0A	0B	0C	0D
8.6	6.9	5.7	8.0

*POST CLEANUP - CLEANUP - IMPINGER TRAINS
(Behind Methods Filter)

0A	0B	0C	0D
9.1	8.4	2.1	11.1

1A	1B	1C	1D
8.5	5.3	12.3	11.8

* NOTE: High values may be a result of washing the glass frit behind the Method 5 filter. In many instances, the glass frit was observed to contribute small fragments of the frit to the acetone wash.

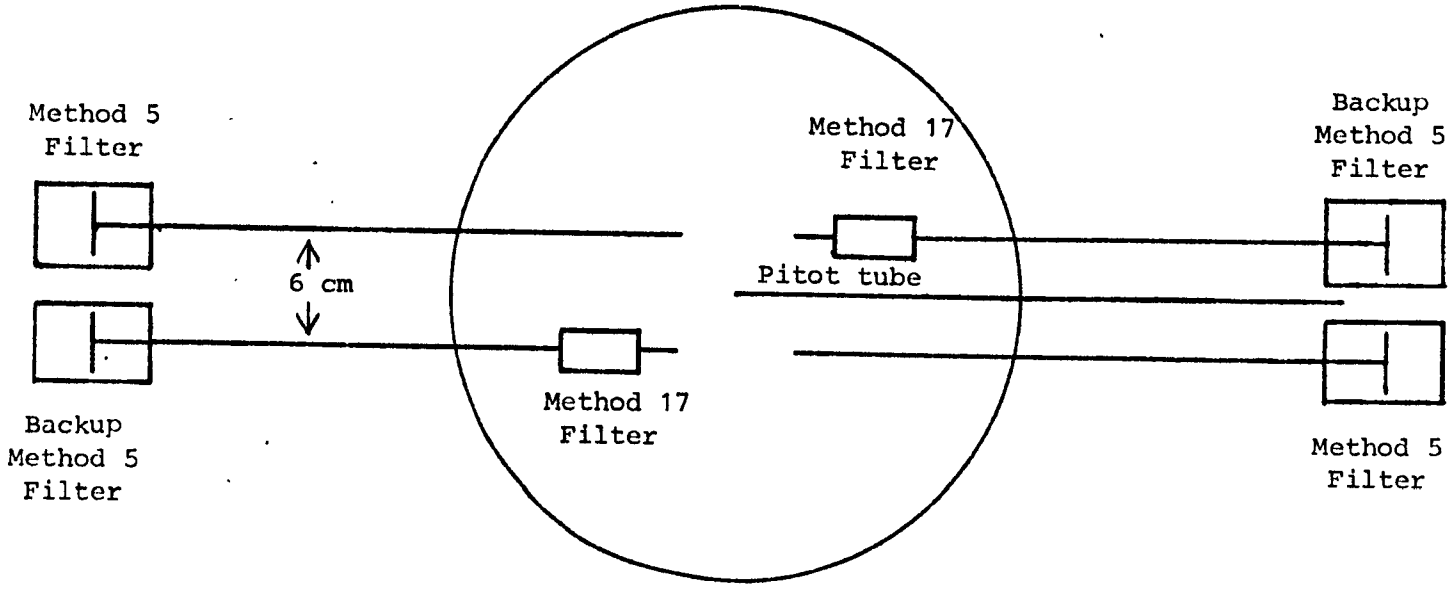

Figure 1. IN-STACK VERSUS OUT-OF-STACK SAMPLING

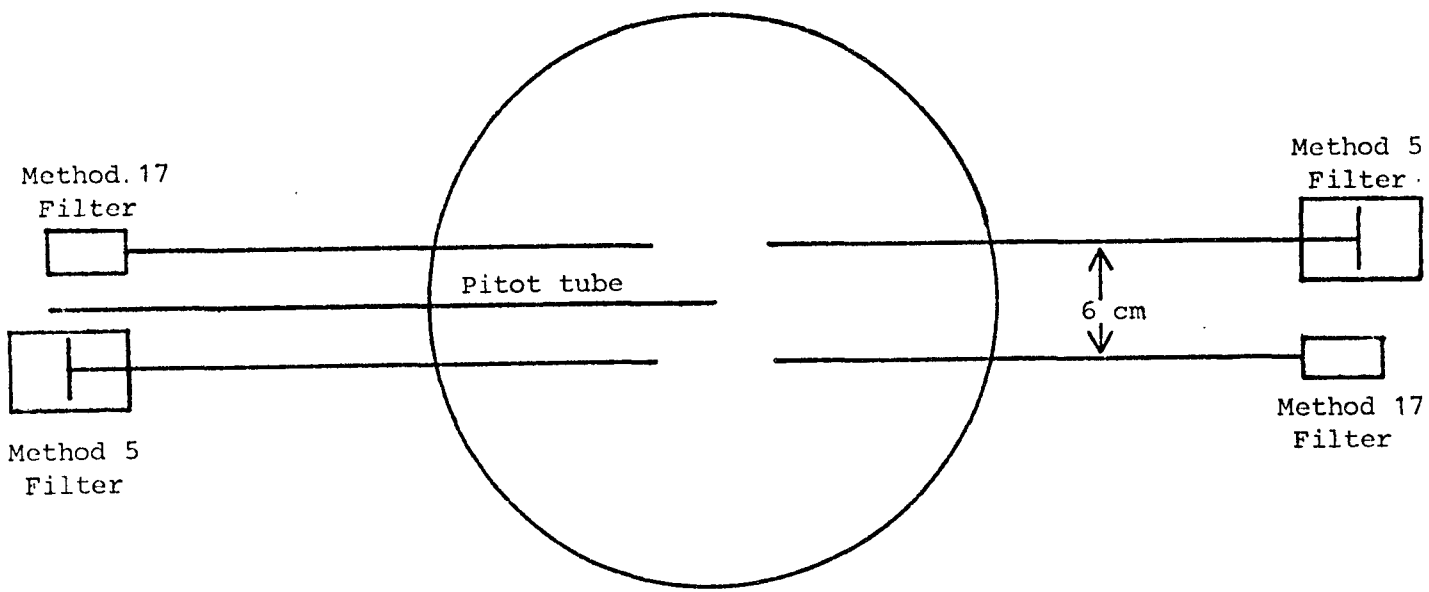

Figure 2. OUT-OF-STACK EQUIPMENT COMPARISON

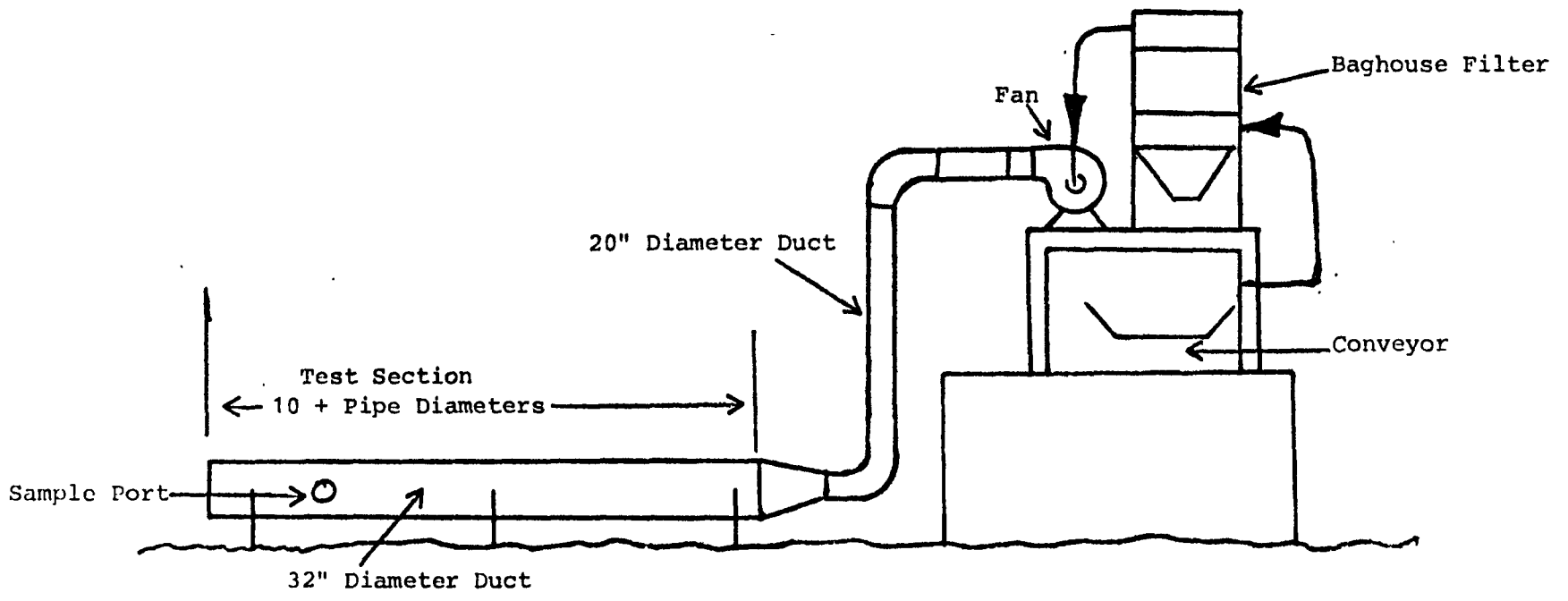

Figure 3. TEST SITE, CARGILL GRAIN ELEVATOR, RESERVE, LOUISIANA

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA 600/4-80-022	2.	3. RECIPIENT'S ACCESSION NO.
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16. ABSTRACT

Comparative tests between EPA Reference Method 5 and commercially-available in-stack filters (EPA Reference Method 17) showed that in-stack filters can be acceptable substitutes for the Reference Method at nonmetallic mineral plants. These comparative tests were conducted in the exhaust stacks from a clay calciner, a feldspar grinding mill and a limestone crusher. Particulate concentrations encountered ranged from 30 to more than 300 mg/m^3. Laboratory studies using feldspar dust gave results identical to the field study results. Insignificant amounts of material were found downstream of the in-stack filters. These results are compared to similar results obtained at a grain elevator and a kraft pulp mill.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Particulate In-stack filter, out-of-stack filter EPA Method 17 EPA Method 5	—	43F 68A

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