



**Interlaboratory Cooperative Study of
the Precision and Accuracy
of the Measurement**

of

**DUSTFALL
using
ASTM Method D 1739**

DS 55-S4



AMERICAN SOCIETY FOR TESTING AND MATERIALS



FINAL REPORT

on

INTERLABORATORY COOPERATIVE STUDY OF THE PRECISION AND ACCURACY OF THE MEASUREMENT OF DUSTFALL USING ASTM METHOD D1739

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INTERLABORATORY COOPERATIVE STUDY OF THE
PRECISION AND ACCURACY OF THE MEASUREMENT OF DUSTFALL
USING ASTM METHOD D 1739

by

J. F. Foster, G. H. Beatty, and J. E. Howes, Jr.

INTRODUCTION

This report represents the results obtained from an experimental study of the variability inherent in measurements of dustfall, using ASTM Method D 1739. (1)* The report also includes a description of the experimental program together with tabulations of the data and the statistical interpretations of the results.

The evaluation of ASTM Method D 1739 was performed as part of the first phase of Project Threshold, a comprehensive program to validate ASTM methods of measuring atmospheric contaminants. In addition, methods for measuring the content of nitrogen dioxide (D 1607), sulfur dioxide (D 2914), lead (D 3112), total sulfation (D 2110), and particulate matter (D 1704) in the atmosphere have also been evaluated during Phase 1.

SUMMARY OF RESULTS

A statistical analysis of 84 dustfall determinations performed in accordance with ASTM Method D 1739 produced the following results:

- The average standard deviation for variations among single dustfall measurements by different laboratories (reproducibility) is 1.46 grams per square meter per month (g/m^2 -month) and is associated with a mean dustfall

* References at end of report.

of $6.45 \text{ g/m}^2\text{-month}$. Measurements at three dustfall rates over the range of 3.28 to $10.47 \text{ g/m}^2\text{-month}$ do not exhibit an apparent relationship between dustfall rate and reproducibility.

- The average standard deviation for variations among repeated dustfall measurements within laboratories (repeatability) is $1.03 \text{ g/m}^2\text{-month}$ and is associated with a mean dustfall of $6.45 \text{ g/m}^2\text{-month}$. Measurements at three dustfall rates of the range of 3.28 to $10.47 \text{ g/m}^2\text{-month}$ did not show an apparent relationship between dustfall and repeatability.
- Known quantities of water soluble and water insoluble particulate material were added to some dustfall samples after their collection and prior to analysis. The average recovery of the dust spikes is 96 percent based on all measurements. The average standard deviation of the spike recovery measurements is 16 percent.
- The average standard deviations of between-and within-laboratory determinations of the water insoluble fraction of the ambient dustfall samples are 1.18 and $0.78 \text{ g/m}^2\text{-month}$, respectively, and are associated with a mean water insoluble dustfall of $3.50 \text{ g/m}^2\text{-month}$.
- The average recovery of water insoluble dustfall spikes by all laboratories and at all sites is 91 percent and the standard deviation of the recovery measurements is 18 percent.
- The standard deviations of between-and within-laboratory determinations of the water soluble fraction of ambient dustfall samples are 1.64 and $0.59 \text{ g/m}^2\text{-month}$, respectively, and are associated with a mean water soluble dustfall of $2.59 \text{ g/m}^2\text{-month}$.

- The average recovery of water soluble dustfall spikes by all laboratories and at all sites is 95 percent and the standard deviation of the recovery measurements is 37 percent.

EXPERIMENTAL PROGRAM

Test Characteristics

The measurement of dustfall is a passive test in which open-top receptacles are exposed on racks at least eight feet above ground level for 30 ± 2 days to collect particulate material which settles into them from the ambient atmosphere. At the end of the exposure period the water soluble, water insoluble, benzene soluble, and combustible and volatile particulate fractions of the collected particulates are determined by gravimetric analysis. The dustfall is calculated from the total quantity of settleable particulates collected and is expressed grams persquare meter-month ($\text{g}/\text{m}^2\text{-month}$).

Test Procedure

Each participating laboratory performed dustfall measurements in accordance with ASTM Method D 1739 as reproduced in the Appendix. Copper sulfate, as prescribed by the Method, was used as an algicide in the Los Angeles tests. The dustfall data were corrected for addition of the copper sulfate. Isopropyl alcohol (approximately 800 ml) was added as an antifreeze in the tests at Bloomington and Manhattan. Copper sulfate was not added in the Bloomington and Manhattan tests.

The participants supplied holders and plastic dustfall jars similar to the No. 190 jars sold by Research Appliance Company. Each laboratory setup their dustfall jars and monitored them during the first five days of the test period. Routine inspections were performed by site or Battelle personnel during the remainder of the exposure. At the end of the test period, the dustfall jars were covered and sealed by Battelle personnel and shipped to the respective laboratories for analysis.

Test Pattern

Tables 1, 2, and 3 show the statistical design for random placement of dustfall containers on the four racks at each of the test sites. Figures 1 and 2 show diagrammatically the placement of containers according to the designs of Tables 1, 2, and 3.

Spiking Procedure

Each laboratory was supplied with spikes containing known quantities of salt, sand, and granular polystyrene which were added to selected samples according to the patterns given in Tables 1, 2, and 3. The spikes were added to the dustfall jars after their exposure and return to the laboratories and prior to the analysis. The equivalent dustfall rate of the spikes ranged from about 2.5 to 40 g/m²-month. Specific data on the composition of the spikes are presented in Tables 7, 8, and 9.

Test Sites

Site No. 1, Los Angeles

The dustfall receptacles at Los Angeles were mounted on four wooden racks placed on a rooftop at the Hancock Foundation building on the campus of the University of Southern California in Los Angeles as shown in Figure 3. The racks and holders extended the top of the jars about six feet above the roof level. The exposure area also included sampling stations for measurement of total sulfation. These sampling stations, which also appear in the photograph were placed so that no direct interaction would be expected between the two tests.

Eight laboratories participated in the Los Angeles tests which were conducted for 30 days over the period of August 15 to September 14, 1971. The mean dustfall at the Los Angeles site was 5.60 g/m²-month.

TABLE 1. STATISTICAL DESIGN OF DUSTFALL TESTS
AT THE LOS ANGELES SITE

Rack	Container Position on Rack ^(a)							
	1	2	3	4	5	6	7	8
A	M ₁	(M ₁)	L ₁	(Q ₁)	Q ₁	(L ₁)	O ₁	(O ₁)
B	(P ₁)	(K ₁)	J ₁	N ₁	(J ₁)	K ₁	P ₁	(N ₁)
C	K ₁	(K ₁)	(O ₁)	(L ₁)	P ₁	O ₁	L ₁	(P ₁)
D	J ₁	(M ₁)	M ₁	(J ₁)	Q ₁	N ₁	N ₁	(Q ₁)

(a) Entries are laboratory code designations. Parentheses indicate samples which were spiked with a known quantity of dust.

TABLE 2. STATISTICAL DESIGN OF DUSTFALL TESTS
AT THE BLOOMINGTON SITE

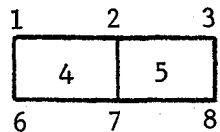
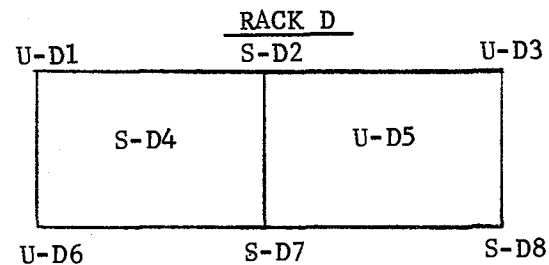
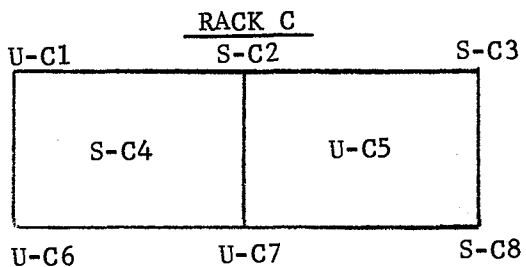
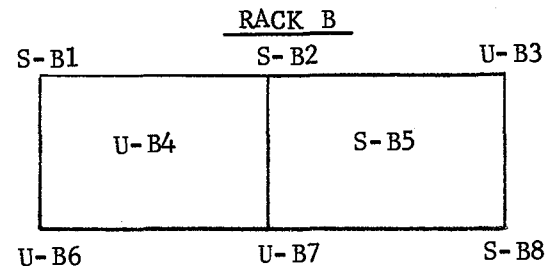
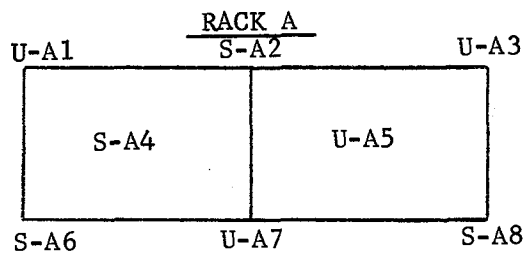
Rack	Container Position on Rack ^(a)							
	1	2	3	4	5	6	7	8
A	(O ₂)	(P ₂)	P ₂	L ₂	(J ₂)	P ₂	--	M ₂
B	(L ₂)	(O ₂)	O ₂	(M ₂)	K ₂	N ₂	N ₂	(K ₂)
C	L ₂	M ₂	J ₂	(L ₂)	K ₂	(N ₂)	--	--
D	J ₂	N ₂	(M ₂)	J ₂	K ₂	(P ₂)	O ₂	--

(a) Entries are laboratory code designations. Parentheses indicate samples which were spiked with a known quantity of dust.

TABLE 3. STATISTICAL DESIGN OF DUSTFALL TESTS
AT THE MANHATTAN SITE

Rack	Container Position on Rack ^(a)							
	1	2	3	4	5	6	7	8
A	(P ₃)	(K ₃)	K ₃	L ₃	(M ₃)	K ₃	--	O ₃
B	(L ₃)	(P ₃)	P ₃	(O ₃)	J ₃	N ₃	(N ₃)	(J ₃)
C	L ₃	O ₃	(M ₃)	(L ₃)	J ₃	(N ₃)	--	--
D	M ₃	N ₃	(O ₃)	M ₃	(J ₃)	(K ₃)	P ₃	--

(a) Entries are laboratory code designations. Parentheses indicate samples which were spiked with a known quantity of dust.



Pattern of the Position of the Containers on each Rack.

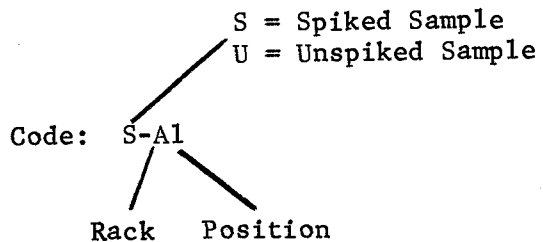
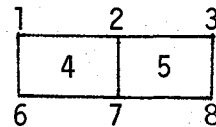
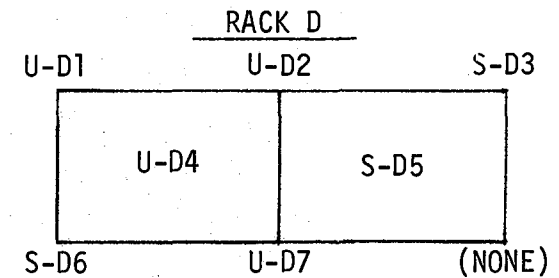
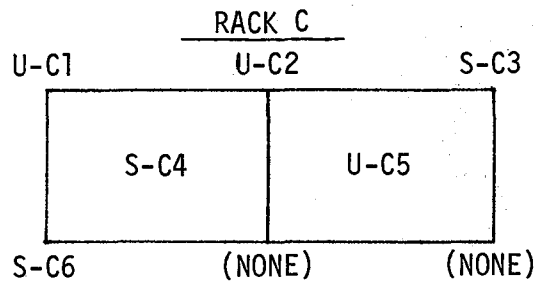
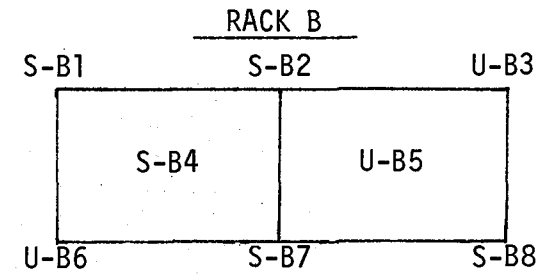
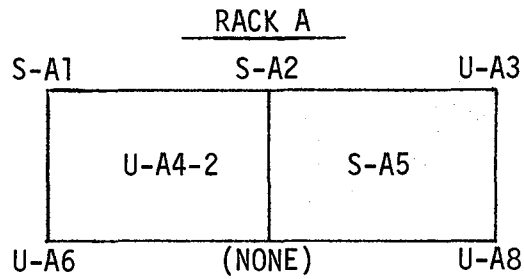


FIGURE 1. DIAGRAM OF PLACEMENT OF THE DUSTFALL CONTAINERS IN THE TEST AT LOS ANGELES



Pattern of the Position of the Container on each Rack

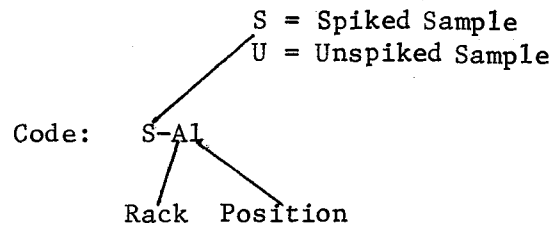


FIGURE 2. DIAGRAM OF PLACEMENT OF THE DUSTFALL CONTAINERS IN THE TESTS AT BLOOMINGTON AND MANHATTAN

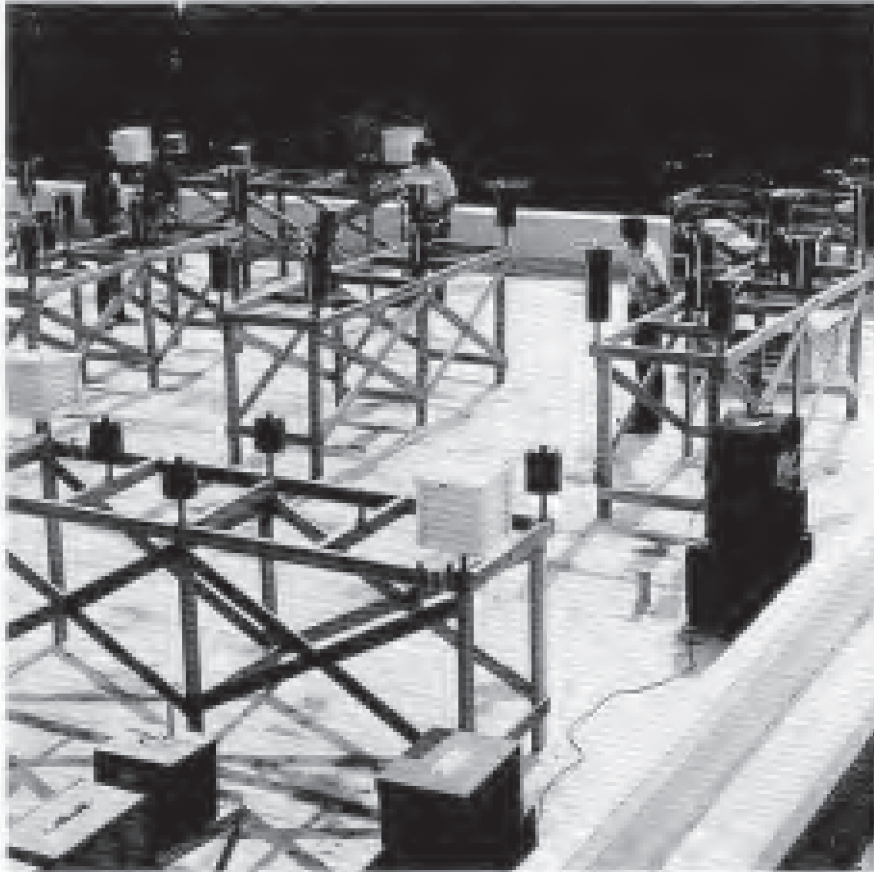


FIGURE 3. ARRAYS OF DUSTFALL COLLECTORS (D 1739) AND TOTAL SULFATION DETECTORS (D 2010) ON ROOF-TOP RACKS AT LOS ANGELES

Site No. 2, Bloomington, Indiana

The dustfall receptacles at Bloomington were mounted on wooden racks placed on the ground, as shown in Figure 3. Each receptacle holder was placed three feet above the top of the rack on an extension rod. This procedure fulfilled the requirement that the open tops of the receptacles be at least eight feet above ground level and four feet above any other surface.

After nine days exposure at Bloomington, some of the racks were tipped over by trespassers. The experiment was then moved to suburban Columbus, Ohio, restarted, and continued from November 4 - December 6, 1971 at Battelle's industrial research location inside the fenced area shown in Figure 4. For simplicity, the results from the restarted tests are designated in this report as the Bloomington test of the Phase 1 program.

Seven laboratories participated in the Bloomington tests. The mean dustfall rate was $3.28 \text{ g/m}^2\text{-month}$.

Site No. 3, Manhattan, New York City

The dustfall receptacles at Site 3 were mounted on racks and exposed on a rooftop of Cooper Union, 51 Astor Place, in lower Manhattan, with an arrangement similar to exposures at the other two sites. The racks and holders extended the top of the dustfall jars about 8 feet above the roof level.

Seven laboratories participated in the Manhattan tests which were conducted during the period of January 9 to February 8, 1972. The mean dustfall at the site was $10.47 \text{ g/m}^2\text{-month}$.

Participating Laboratories

The participating laboratories are listed below in alphabetical order:

George D. Clayton and Associates
Department of Health, State of California

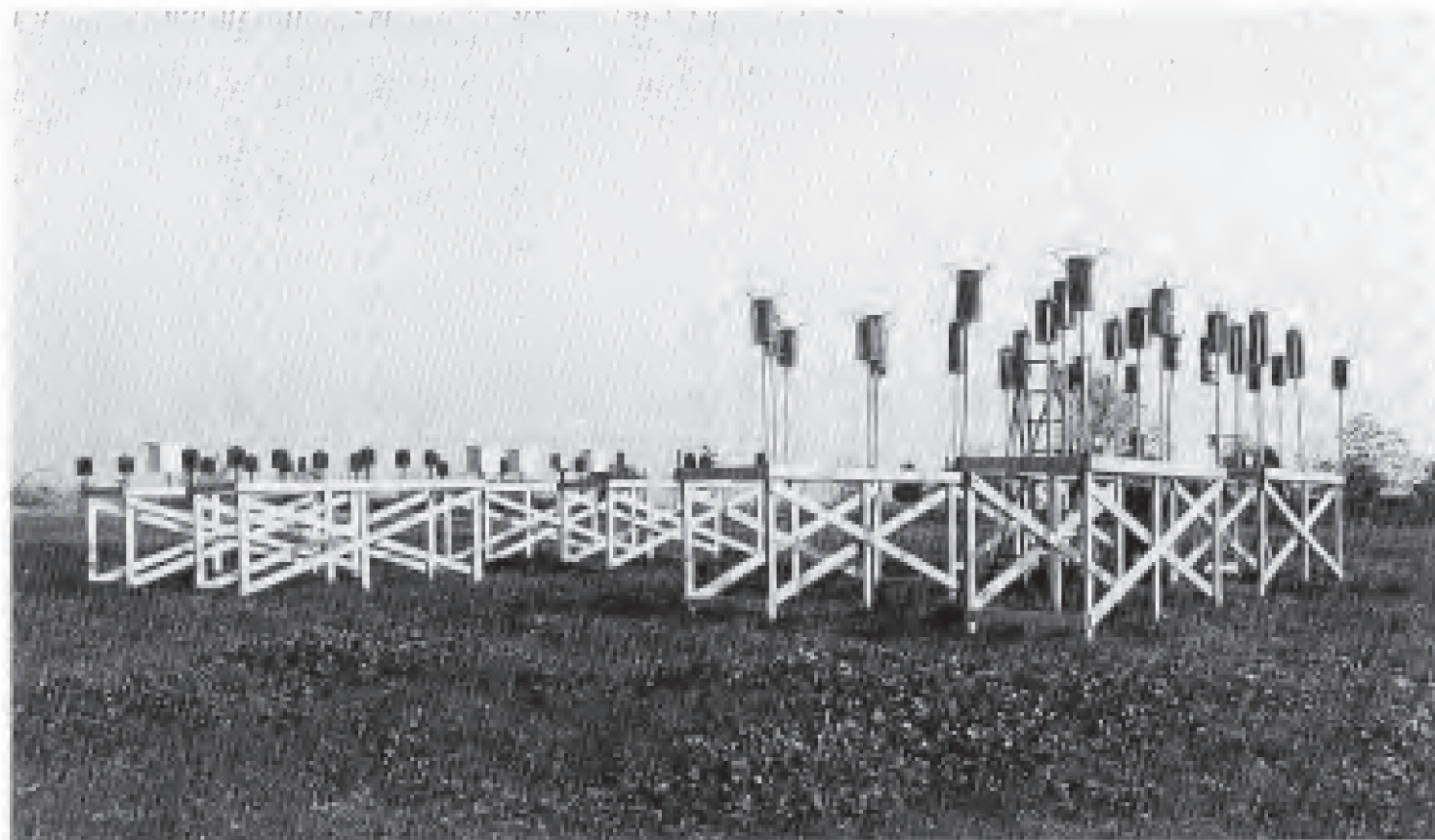


FIGURE 4. THE DUSTFALL (D 1739) AND TOTAL SULFATION RATE (D 2010) TESTS STARTED AT BLOOMINGTON, INDIANA (COMPLETED AT COLUMBUS, OHIO TEST SITE)

Arthur D. Little, Inc.
Midwest Research Institute
Public Service Electric and Gas Company (New Jersey)
Research Triangle Institute
Walden Research Corporation
Western Electric Company

Throughout this report the identity of the participants is concealed by using a set of code letters. Numerical subscripts with the code letters designate the site at which samples were collected. In general, any particular letter designates a different laboratory at each site. Each participant is supplied the key to the code letters for himself in all data tables, but no key for the other participants.

STATISTICAL ANALYSIS OF DUSTFALL MEASUREMENTS

Statistical Measures

The experimental test program was designed and conducted so that statistical analysis of the results would provide measures of the following characteristics of ASTM Method D 1739.

Reproducibility

The participating laboratories performed concurrent measurements of dustfall. Differences among the dustfall rates found in the simultaneous measurements represent a measure of variability between laboratories. The standard deviation of all such measurements by all laboratories is a measure of precision of the Test Method which in this report is called "between-laboratory variability" or "reproducibility" and is represented by the symbol, s_b .

Repeatability

Each laboratory performed duplicate measurements which, ideally, should have produced identical dustfall values. Thus, a difference

between a pair of determinations is a measure of another type of variability. The standard deviation of all such differences by all laboratories is a measure of precision of the Test Method which in this report is called "within-laboratory variability" or "repeatability" and is denoted by the symbol, s_w .

Accuracy

Measurements were performed by each laboratory in which one sample of duplicate ambient dustfall samples was spiked with a known quantity of dust prior to the analysis procedure. The difference between the determinations for such pairs is a measure of the equivalent dustfall of the spike. Differences between the experimentally determined and the "true" equivalent dustfall of the spike is a measure of accuracy of the dustfall recovery and analysis procedure. The data are presented as the percentage of the spike which was recovered in the analysis.

Additional discussions of the preceding statistical measures have been presented by Mandel⁽²⁾ and in ASTM publications.^(3,4)

Experimental Data

Tables 4, 5, and 6 show the results of the dustfall determinations at Los Angeles, Bloomington, and Manhattan, respectively. These sites are designated as Sites 1, 2, and 3 as shown by the subscripts in the laboratory codes in column 1. The laboratories are coded J through P or Q in these tables. The columns headed U contain the dustfall determinations for the unspiked samples and the columns headed S contain dustfall determinations for the spiked samples. The columns headed S-U contain the differences between corresponding spiked and unspiked determinations. The columns headed S' contain the amounts by which the dustfall content of the unspiked (ambient) samples were increased by the addition of known amounts of particulate matter. All concentrations appearing in Tables 3, 4, and 5 are in units of grams per square meter per month ($\text{g/m}^2\text{-month}$).

TABLE 4. DATA FROM DUSTFALL EXPERIMENTS AT LOS ANGELES SITE

Laboratory	Unspiked Samples				Spiked Samples				Estimated Spiking Rate, g/m ² -month (S-U)	Spikes		Recovery, percent of S'	
	Container Location	Exposed Area, m ²	Exposure Time, days	Dustfall, g/m ² -month (U)	Container Location	Exposed Area, m ²	Exposure Time, days	Dustfall, g/m ² -month (S)		Code Number	Weight, g		Rate, g/m ² -month (S')
J ₁	B3	0.018	30	3.80	B5	0.018	30	29.39	25.59	4	0.4634	25.74	99
	D1	0.018	30	4.64	D4	0.018	30	40.55	35.91	12	0.6866	38.14	94
K ₁	B6	0.01824	30	3.19	B2	0.01824	30	(b)	-	5	0.3757	20.60	-
	C1	0.01824	30	2.68	C2	0.01824	30	39.66	36.98	13	0.6557	35.95	103
L ₁	A3	0.01824	30	5.71	A6	0.01824	30	27.17	21.46	1	0.4033	22.11	97
	C7	0.01824	30	4.84	C4	0.01824	30	15.80	10.96	9	0.1776	9.74	113
M ₁	A1	0.01838	30	8.07	A2	0.01838	30	10.62	2.55	8	0.0657	3.57	71
	D3	0.01838	30	8.53	D2	0.01838	30	38.01	29.48	11	0.6125	33.32	88
N ₁	B4	0.01824	30	5.40	B8	0.01824	30	45.50	40.10	3	0.7290	39.97	100
	D6	0.01824	30	(b)	D7	0.01824	30	15.54	-	16	0.1856	10.18	-
O ₁	A7	0.0181	30	6.35	A8	0.0181	30	28.90	22.55	10	0.3871	21.38	105
	C6	0.0181	30	7.62	C3	0.0181	30	23.70	16.08	2	0.3637	20.09	80
P ₁	B7	0.0182	30	5.28	B1	0.0182	30	(b)	-	14	0.5024	27.60	-
	C5	0.0182	30	5.98	C8	0.0182	30	(b)	-	6	0.3235	17.77	-
Q ₁	A5	0.0186	30	6.28	A4	0.0186	30	33.81	27.53	15	0.5154	27.71	99
	D5	0.0186	30	20.38(a)	D8	0.0186	30	21.24	-	7	0.5146	27.67	-

(a) Outlier excluded from analysis on statistical grounds.

(b) Sample lost during analysis.

TABLE 5. DATA FROM DUSTFALL EXPERIMENTS AT BLOOMINGTON SITE

Laboratory	Unspiked Samples				Spiked Samples				Estimated Spiking Rate, g/m ² -month (S-U)	Spikes		Recovery, percent of S'	
	Container Location	Exposed Area, m ²	Exposure Time, days	Dustfall, g/m ² -month (U)	Container Location	Exposed Area, m ²	Exposure Time, days	Dustfall, g/m ² -month (S)		Code Number	Weight, g		Rate, g/m ² -month (S')
J ₂	D1	0.018	32	5.72	A5	0.018	32	11.25	5.53	40	0.0910	4.74	11'
	D4	0.018	32	7.10	C3	0.018	32	11.52	4.42	54	0.0772	4.02	110
K ₂	B5	0.01824	32	1.30	B8	0.01824	32	5.44	4.14	37	0.0796	4.09	101
	C5	0.01824	32	1.43	D5	0.01824	32	3.94	2.51	46	0.0786	4.04	62
L ₂	A4	0.01824	32	3.05	B1	0.01824	32	7.63	4.58	55	0.0565	2.90	158
	C1	0.01824	32	3.23	C4	0.01824	32	8.04	4.81	41	0.0849	4.36	110
M ₂	A8	0.0181	32	2.85	B4	0.0181	32	7.30	4.45	38	0.0793	4.11	108
	C2	0.0181	32	3.21	D3	0.0181	32	3.21(a)	0.0 (a)	47	0.0466	2.41	0(a)
N ₂	B6	0.01824	32	5.90	B7	0.01824	32	1.14(a)	-4.76(a)	48	0.0576	2.96	-161(a)
	D2	0.01824	32	2.82	C6	0.01824	32	6.77	4.23	39	0.0856	4.40	90
O ₂	B3	0.0182	32	1.12	B2	0.0182	32	4.91	3.79	42	0.0717	3.69	103
	D7	0.0182	32	0.96	A1	0.0182	32	4.57	3.61	45	0.0806	4.15	87
P ₂	A3	0.0186	32	4.39	A2	0.0186	32	8.43	4.04	43	0.0752	3.79	106
	A6	0.0186	32	2.81	D6	0.0186	32	8.85	6.04	44	0.0852	4.29	142

(a) Outlying data, excluded from the statistical analysis of the spike recovery.

TABLE 6. DATA FROM DUSTFALL EXPERIMENTS AT MANHATTAN SITE

Laboratory	Unspiked Samples				Spiked Samples				Estimated Spiking Rate, g/m ² -month (S-U)	Spikes		Recovery, percent of S'	
	Container Location	Exposed Area, m ²	Exposure Time, days	Dustfall, g/m ² -month (U)	Container Location	Exposed Area, m ²	Exposure Time, days	Dustfall, g/m ² -month (S)		Code Number	Weight, g		Rate, g/m ² -month (S')
J ₃	A7	0.01824	30	11.29	A8	0.01824	30	20.62	9.33	56	0.1932	10.59	88
	C6	0.01824	30	10.83	C3	0.01824	30	20.92	10.09	69	0.1756	9.63	105
K ₃	B7	0.0186	30	6.81	B1	0.0186	30	16.69	9.88	63	0.1869	10.05	98
	C5	0.0186	30	9.18	C8	0.0186	30	15.83	6.65	62	0.1546	8.31	80
L ₃	A3	0.01824	30	10.37	A6	0.01824	30	18.86	8.49	60	0.1982	10.87	78
	C7	0.01824	30	12.51	C4	0.01824	30	19.59	7.08	65	0.1739	9.53	74
M ₃	B4	0.018	30	9.29	B8	0.018	30	17.92	8.63	66	0.1590	8.83	98
	D6	0.018	30	10.88	D7	0.018	30	21.04	10.16	59	0.1627	9.04	112
N ₃	A5	0.01824	30	11.08	A4	0.01824	30	21.30	10.22	67	0.1757	9.63	106
	D5	0.01824	30	13.01	D8	0.01824	30	23.10	10.09	58	0.1710	9.38	108
O ₃	B6	0.0181	30	10.33	B2	0.0181	30	17.73	7.40	57	0.1949	10.77	69
	C1	0.0181	30	10.99	C2	0.0181	30	19.28	8.29	68	0.1078	5.96	139
P ₃	B3	0.0182	30	11.37	B5	0.0182	30	12.28	0.91	61	0.1803	9.91	9
	D1	0.0182	30	8.61	D4	0.0182	30	12.11	3.50	64	0.1690	9.29	38

One outlying dustfall value in Table 3 and two outlying spike recovery values in Table 4 were excluded from the analysis. The excluded values were reported by Laboratory Q₁, at Los Angeles and Laboratories M₂ and N₂ at Bloomington. On statistical grounds the values can be rejected at the one percent level of significance by applying the t-statistic.(5)

The weight data used to calculate the dustfall rates of the spiked and unspiked samples are presented in Tables 7, 8, and 9 along with data on the composition of the dustfall spikes.

Analysis of Reproducibility

The analysis of between-laboratory variability (reproducibility) was performed using the dustfall measurements for unspiked samples presented in Tables 4, 5, and 6. A summary of the results of the analysis by site is presented in Table 10. The table shows, for each site and for all sites combined, the number (n) of dustfall measurements performed, the mean (m) of the dustfall rate of all laboratories, the number of degrees of freedom (df) associated with the statistical analysis, the reproducibility (s_b), and the coefficient of variation (CV).

TABLE 10. SUMMARY OF BETWEEN-LABORATORY VARIABILITY
(REPRODUCIBILITY) OF DUSTFALL MEASUREMENTS

Site	n	m, g/m ² -month	df	Reproducibility	
				s _b g/m ² -month	CV,%
Los Angeles ^(a)	14	5.60	7	1.69	30
Bloomington	14	3.28	6	1.67	51
Manhattan	14	10.47	6	0.92	9
All Sites ^(a)	42	6.45	19	1.46	23

(a) Determination for Laboratory Q₁ was excluded from the analysis on statistical grounds.

TABLE 7. DUSTFALL WEIGHT DATA - LOS ANGELES SITE

Laboratory	Sample Number	Dustfall Sample Weights, grams			Spike Number	Dustfall Spike Weights, grams ^(c)		
		Water Insoluble	Water Soluble	Benzene Soluble		Water Insoluble	Water Soluble	Benzene Soluble
J ₁	U-B3	0.0474	0.0210	0.0010				
	U-D1	0.0531	0.0304	0.0024				
	S-B5	0.2981	0.2310	0.0333	4	0.2726	0.1908	0.0426
	S-D4	0.4549	0.2750	0.0382	12	0.4317	0.2549	0.0467
K ₁	U-B6	0.0360	0.0221	0.0017				
	U-C1	0.0380	0.0108	0.0016				
	S-B2	(a)	(a)	(a)	5	0.3757	0	0.0519
	S-C2	0.4418	0.2817	0.0442	13	0.4044	0.2513	0.0462
L ₁	U-A3	0.02105	0.08312	<0.00010				
	U-C7	0.01758	0.07076	<0.00010				
	S-A6	0.26078	0.23474	0.03751	1	0.2501	0.1532	0.0423
	S-C4	0.01557	0.27262	<0.00010	9	0	0.1776	0
M ₁	U-A1	0.0308	0.1175	<0.0001				
	U-D3	0.0324	0.1244	<0.0001				
	S-A2	0.0699	0.1254	0.0187	8	0.0657	0	0.0657
	S-D2	0.3335	0.3653	0.0215	11	0.3753	0.2372	0.0461
N ₁	U-B4	0.03717	0.06124	0.00135				
	U-D6	(a)	(a)	(a)				
	S-B8	0.52284	0.30706	0.04150	3	0.4869	0.2421	0.0418
	S-D7	0.06345	0.21992	0.03147	16	0.0324	0.1532	0.0324
O ₁	U-A7	0.035	0.080	0.000				
	U-C6	0.058	0.080	0.000				
	S-A8	0.423	0.100	0.000	10	0.3871	0	0
	S-C3	0.229	0.200	0.000	2	0.2050	0.1587	0.0376
P ₁	U-B7	0.0384	0.0577	0				
	U-C5	0.0254	0.0835	0.0015				
	S-B1	(a)	(a)	(a)	14	0.3245	0.1779	0.0459
	S-C8	(a)	(a)	(a)	6	0.0559	0.2676	0.0559
Q ₁	U-A5	0.0205	0.0963	(b)				
	U-D5	0.2614 (d)	0.1176 (d)	(b)				
	S-A4	0.3722	0.2566	0.0270	15	0.3350	0.1804	0.0237
	S-D8	0.0484	0.3468	(b)	7	0.2973	0.2173	0

(a) Sample lost during analysis.

(b) Less than limit of detection.

(c) Data supplied by National Bureau of Standards.

(d) Outlying data, excluded from statistical analysis.

TABLE 8. DUSTFALL WEIGHT DATA - BLOOMINGTON SITE

Laboratory	Sample Number	Dustfall Sample Weights, grams			Spike Number	Dustfall Spike Weights, grams ^(a)		
		Water Insoluble	Water Soluble	Benzene Soluble		Water Insoluble	Water Soluble	Benzene Soluble
J ₂	U-D1	0.0260	0.0839	0.0012				
	U-D4	0.0271	0.1092	0.0042				
	S-A5	0.0850	0.1310	0.0202	40	0.0552	0.0358	0.0096
	S-C3	0.0634	0.1577	0.0129	54	0.0410	0.0362	0
K ₂	U-B5	0.01018	0.01509	0.00265				
	U-C5	0.01240	0.01535	0.00010				
	S-B8	0.07509	0.03072	0.01163	37	0.0432	0.0364	0.0079
	S-D5	0.04587	0.03072	0.00032	46	0.0387	0.0399	0
L ₂	U-A4	0.0151	0.0442	0.0015				
	U-C1	0.0212	0.0416	0.0017				
	S-B1	0.0795	0.0689	0.0098	55	0.0565	0	0.0121
	S-C4	0.0750	0.0814	0.0178	41	0.0499	0.0350	0.0098
M ₂	U-A8	0.030	0.025	0.002				
	U-C2	0.042(b)	0.020(b)	0.002				
	S-B4	0.086	0.055	0.011	38	0.0448	0.0345	0.0084
	S-D3	0.032(b)	0.030(b)	0.008	47	0.0089	0.0377	0.0089
N ₂	U-B6	0.0823(b)	0.0325	0.0100				
	U-D2	0.0425	0.0123	0.0017				
	S-B7	0.0212(b)	0.0009	0.0010	48	0.0576	0	0.0091
	S-C6	0.0826	0.0492	0.0118	39	0.0510	0.0346	0.0103
O ₂	U-B3	0.0178	0.0040	0.0005				
	U-D7	0.0110	0.0077	0.0008				
	S-B2	0.0454	0.0500	0.0075	42	0.0472	0.0245	0.0120
	S-A1	0.0357	0.0530	0.0060	45	0.0537	0.0269	0.0079
P ₂	U-A3	0.02293	0.06426	0.00147				
	U-A6	0.03433	0.02150	0.00680				
	S-A2	0.06831	0.09895	0.02314	43	0.0460	0.0292	0.0106
	S-D6	0.09478	0.08079	0.02096	44	0.0540	0.0312	0.0119

(a) Data supplied by National Bureau of Standards.

(b) Outlying data, excluded from statistical analysis of spike recovery.

TABLE 9. DUSTALL WEIGHT DATA - MANHATTAN SITE

Laboratory	Sample Number	Dustfall Sample Weights, grams			Spike Number	Dustfall Spike Weights, grams (a)		
		Water Insoluble	Water Soluble	Benzene Soluble		Water Insoluble	Water Soluble	Benzene Soluble
J ₃	U-A7	0.12577	0.08016	0.00575				
	U-C6	0.11976	0.07774	0.00539				
	S-A8	0.23375	0.14229	0.03115	56	0.1309	0.0623	0.0285
	S-C3	0.21616	0.16541	0.0499	69	0.0874	0.0882	0
K ₃	U-B7	0.0504	0.0762	0.0114				
	U-C5	0.0670	0.1037	0.0223				
	S-B1	0.1534	0.1570	0.0620	63	0.1030	0.0839	0.0253
	S-C8	0.1573	0.1372	0.0512	62	0.0861	0.0685	0.0197
L ₃	U-A3	0.1267	0.0625	0.0051				
	U-C7	0.1625	0.0657	0.0073				
	S-A6	0.2280	0.1160	0.0132	60	0.1320	0.0662	0.0196
	S-C4	0.2304	0.1270	0.0114	65	0.1013	0.0726	0.0207
M ₃	U-B4	0.1214	0.0458	0.0020				
	U-D6	0.1513	0.0446	0.0069				
	S-B8	0.2082	0.1144	0.0148	66	0.0939	0.0651	0.0172
	S-D7	0.2574	0.1213	0.0183	59	0.0941	0.0686	0.0195
N ₃	U-A5	0.1331	0.0691	0.0065				
	U-D5	0.1554	0.0819	0.0087				
	S-A4	0.2339	0.1547	0.0330	67	0.1004	0.0753	0.0266
	S-D8	0.2758	0.1455	0.0199	58	0.1111	0.0599	0.0223
O ₃	U-B6	0.121	0.066	0.012				
	U-C1	0.127	0.072	0.004				
	S-B2	0.241	0.080	0.032	57	0.1124	0.0825	0.0340
	S-C2	0.215	0.134	0.038	68	0.1078	0	0.0267
P ₃	U-B3	0.2014	0.0055	0.0832				
	U-D1	0.1502	0.0065	0.0116				
	S-B5	0.2072	0.0163	0.0440	61	0.0909	0.0894	0.0207
	S-D4	0.2039	0.0165	0.0518	64	0.0921	0.0769	0.0197

(a) Data supplied by National Bureau of Standards.

The mean square (MS) of dustfall measurements at each site is obtained by the equation

$$MS = \frac{\sum n_i (\bar{x}_i - \bar{x})^2}{df}, \quad \text{Equation (1)}$$

where, \bar{x}_i denotes the measurement value obtained by the i^{th} laboratory, \bar{x} denotes the arithmetic mean of the measurements obtained by all laboratories at the site, and df is the degrees of freedom. For those laboratories making duplicate measurements, \bar{x}_i is taken to be the average of the two measurements, and the mean is given a weight of 2 so that $n_i = 2$. If a laboratory made a single determination, then \bar{x}_i is equal to that determination and n_i is 1.

The mean square calculated from Equation (1) includes both the within- and between-laboratory variance. The relationship is described by the equation

$$MS = s_w^2 + Ks_b^2, \quad \text{Equation (2)}$$

where, s_w^2 denotes the within-laboratory variance, s_b^2 denotes the between-laboratory variance, and K is the number of replicate measurements by each laboratory.⁽⁶⁾ The between-laboratory variance (s_b^2) is determined by substitution of values for MS , s_w , and K in Equation (2). Duplicate determinations were performed by each laboratory at each site, therefore $K = 2$.

The reproducibility of the Test Method, defined in this report as the standard deviation (s_b) of the dustfall measurements between laboratories, is computed by extracting the square root of the between-laboratory variance. Another measure of reproducibility is provided by the coefficient of variation (CV) computed from the following equation

$$CV = 100 s_b / m.$$

The coefficient of variation, as formulated here, measures reproducibility as a percentage of the mean dustfall rate.

The mean dustfall rates in Table 10 differ from site to site. The observed dustfall rate at Los Angeles is almost double the observed rate at Bloomington, and the rate at Manhattan is almost double the rate at Los Angeles. These differences make it possible to examine the relationship of reproducibility (s_b) to mean dustfall (m). However, when pairs of values for m and s in Table 10, corresponding to the three sampling sites, are plotted in a two-dimensional graph, the resulting configuration can only be explained in terms of random sampling error. Consequently, the average of the data from all sites which is listed in the last line of Table 10 provides the best basis for forecasting the reproducibility of the Test Method. The mean of the dustfall at all sites is $6.45 \text{ g/m}^2\text{-month}$ and the average standard deviation is $1.46 \text{ g/m}^2\text{-month}$. The average coefficient of variation is 23 percent. These measures characterize the reproducibility of the between-laboratory determinations of dustfall over the range of 3.28 to $10.47 \text{ g/m}^2\text{-month}$.

Analysis of Repeatability

The pairs of duplicate unspiked dustfall determinations were used to obtain a measure of within-laboratory variability (repeatability) of the Test Method. Table 11 presents the results of the analysis of repeatability of the data obtained at each test site.

Table 11 shows, for each site and for all sites combined, the number (n) of dustfall measurements performed, the mean (m) of the dustfall rate of all laboratories, the number of degrees of freedom (df) associated with the statistical analysis, the repeatability (s_w), and the coefficient of variation (CV).

TABLE 11. SUMMARY OF WITHIN-LABORATORY VARIABILITY
(REPEATABILITY) OF DUSTFALL MEASUREMENTS

Site	n	m, g/m ² -month	df	Repeatability	
				s _w , g/m ² -month	CV,%
Los Angeles (a)	14	5.60	6	0.58	10
Bloomington	14	3.28	7	1.00	30
Manhattan	14	10.47	7	1.33	13
All Sites (a)	42	6.45	20	1.03	16

(a) Determination for Laboratory Q₁ was excluded from the analysis on statistical grounds.

The mean square or variance of the measurements at each site was determined by the equation

$$MS = \frac{\frac{(x_i - y_i)^2}{2}}{df}$$

where, x_i and y_i denote the pairs of duplicate measurements made by the i^{th} laboratory.

The repeatability of the Test Method is obtained by taking the square root of the mean square of the within-laboratory measurements.

The coefficient of variation, CV, is calculated from the mean sulfation rate, m, and the repeatability, s_w , as follows

$$CV = 100 s_w / m.$$

The repeatability of the Test Method over the dustfall range studied is characterized by the average of measurements at the three test sites. The last line of Table 11 presents the averages for these data which show that the repeatability is 1.03 g/m²-month and the mean dustfall is 6.45 g/m²-month. The average coefficient of variation of the within-laboratory measurements is 16 percent.

Analysis of Accuracy

A measure of the accuracy of the recovery and analytical steps of the dustfall measurement is provided by the results of corresponding spiked and unspiked determinations. The difference (S-U) between the spiked determination (S) and the unspiked determination (U) is a measure of the known quantity of particulate material added prior to the analysis. The spike recoveries are listed by site and laboratory in the last columns of Tables 4, 5, and 6.

Table 12 summarizes the dustfall spike recovery data for each site and for all sites combined. The statistics in this table represent average results for all laboratories. The mean spike recoveries ranged from 86 percent at Manhattan to 108 percent at Bloomington with a pooled average for all sites of 96 percent. The standard deviations of the between- and within-laboratory recovery measurements over all sites are 16 and 20 percent, respectively.

Analysis of the Variability of Water Soluble and Water Insoluble Dustfall Determinations

The total dustfall is derived from the determination of the water soluble and water insoluble components of the collected particulate matter. Tables 7, 8, and 9 give the results of the analyses of the water soluble and insoluble fractions of the unspiked (U) samples collected at the three test sites. Statistical analysis of these data was performed to determine the variability inherent in the determination of the dustfall components.

The results of the analysis of between-and within-laboratory variation of the water soluble and water insoluble dustfall measurements are summarized in Table 13. The table shows for each site and all sites combined the variability of single measurements by different laboratories and of repetitive measurements by the same laboratory expressed both as the standard deviation (σ_b and σ_w) and the coefficient of variation (CV).

The examination of the mean values of water soluble and water insoluble dustfall and the corresponding standard deviations do not yield a discernible relationship between m and either σ_b or σ_w . Therefore, the best estimate of variability is provided by the average of the data from all sites.

TABLE 12. SUMMARY OF TOTAL DUSTFALL SPIKE RECOVERY DATA^(a)

Site	n	M,%	Between-Laboratory Variation			Within-Laboratory Variation		
			df	σ_b	CV,%	df	σ_w	CV,%
Los Angeles (b)	11	96	6	(b)	0	4	12	13
Bloomington (d)	12	108	6	9	8	5	23	21
Manhattan	14	86	6	25	29	7	22	26
All Sites (b) (d)	37	96	18	16	17	16	20	21

- (a) Column headings: n, number of measurements; m, mean spike recovery; df, degrees of freedom; σ_b , between-laboratory standard deviation of recovery; σ_w , within-laboratory standard deviation of recovery; CV, coefficient of variation.
- (b) Outlying data from Laboratory Q₁ were excluded from the analysis on statistical grounds.
- (c) This variation could not be calculated because the averages for different laboratories were more nearly equal than the replicate measurements within laboratories. The model for computing s_b does not hold in this case.
- (d) Outlying data from Laboratories M₂ and N₂ were excluded from the analysis on statistical grounds.

TABLE 13. ANALYSIS OF VARIANCE OF WATER SOLUBLE AND WATER INSOLUBLE DUSTFALL DETERMINATIONS ^(a)

Dustfall Fraction	Site	n	g/m ² -month	Between-Laboratory Variation			Within-Laboratory Variation		
				df	σ_b g/m ² -month	CV,%	df	σ_w g/m ² -month	CV,%
Water Soluble	Los Angeles	14	3.67	7	1.92	52	6	0.52	14
	Bloomington	14	1.83	6	1.48	81	7	0.74	40
	Manhattan	14	3.35	6	1.49	44	7	0.45	13
	All Sites	42	2.95	19	1.64	56	20	0.59	20
Water Insoluble	Los Angeles	14	1.93	7	0.53	27	6	0.44	23
	Bloomington	14	1.45	6	0.78	54	7	0.61	42
	Manhattan	14	7.12	6	1.84	26	7	1.10	15
	All Sites	42	3.50	19	1.18	34	20	0.78	22

(a) Column headings: N, number of samples; m, mean dustfall; df, degrees of freedom; σ_b , standard deviation of between-laboratory measurements; CV, coefficient of variation; σ_w , standard deviation of within-laboratory measurements.

The pooled averages show that for a mean water soluble dustfall of $2.95 \text{ g/m}^2\text{-month}$, the standard deviations of between-and within-laboratory determinations are 1.64 and $0.59 \text{ g/m}^2\text{-month}$, respectively; the respective coefficients of variation are 56 and 20 percent.

The mean water insoluble dustfall is $3.50 \text{ g/m}^2\text{-month}$ and the standard deviations of between-and within-laboratory measurements are 1.8 and $0.78 \text{ g/m}^2\text{-month}$, respectively, and the respective coefficients of variation are 34 and 22 percent.

Tables 7, 8, and 9 also contain data on the determination of the benzene soluble fraction of the water insoluble dustfall. A statistical analysis of these data was not performed. A cursory investigation of the results indicates that measurement of the benzene soluble fraction is not highly reproducible.

Analysis of the Variability of the Recovery
of Water Soluble and Water Insoluble
Dustfall Spike

The spikes which were added to selected dustfall samples contained known quantities of water soluble and water insoluble particulate material. The difference between pairs of spiked and unspiked dustfall samples is an experimentally determined measure of the known quantity of the spike. The ratio of the experimentally determined-to-known dustfall represents the recovery of the spike.

The recovery, in percent, of the water soluble and water insoluble dustfall spikes from samples obtained at Los Angeles, Bloomington, and Manhattan are given in Tables 14, 15, and 16. A summary of the recovery data by site and for all sites is presented in Table 17. Also shown in Table 17 is the between-and within-laboratory variability of the water soluble and water soluble determinations expressed as standard deviations (σ_b and σ_w) and as coefficients of variation (CV). The statistical analysis of the recovery data was performed in the same manner as the analyses of reproducibility and repeatability described in previous sections.

The mean recovery of water soluble dustfall ranged from 76 percent at Manhattan to 112 percent at Bloomington with a pooled mean

TABLE 14. WATER SOLUBLE AND WATER INSOLUBLE
SPIKE RECOVERY DATA FOR LOS ANGELES
DUSTFALL SAMPLES

Laboratory	Spike Number	Spike Recovery, %	
		Water Soluble	Water Insoluble
J ₁	4	110	92
	12	96	93
K ₁	5	(a)	(a)
	13	108	100
L ₁	1	98	96
	9	114	(b)
M ₁	8	(c)	60
	11	102	80
N ₁	3	102	100
	16	(a)	(a)
O ₁	10	(c)	100
	2	76	83
P ₁	14	(a)	(a)
	6	(a)	(a)
Q ₁	15	89	105
	7	105	-72(d)

(a) Sample lost during analysis.

(b) Water insoluble spike not added.

(c) Water soluble spike not added.

(d) Outlying data based on statistical test.

TABLE 15. WATER SOLUBLE AND WATER INSOLUBLE
SPIKE RECOVERY DATA FOR BLOOMINGTON
DUSTFALL SAMPLES

Laboratory	Spike Number	Spike Recovery, %	
		Water Soluble	Water Insoluble
J ₂	40	132	107
	54	134	89
K ₂	37	43	150
	46	39	86
L ₂	55	(a)	114
	41	114	108
M ₂	38	87	125
	47	27	-112 (b)
N ₂	48	(a)	-106 (b)
	39	107	79
O ₂	42	188	58
	45	168	46
P ₂	43	119	99
	44	190	112

(a) Water soluble spike not added.

(b) Outlying data based on statistical test.

TABLE 16. WATER SOLUBLE AND WATER INSOLUBLE
SPIKE RECOVERY DATA FOR MANHATTAN
DUSTFALL SAMPLES

Laboratory	Spike Number	Spike Recovery, %	
		Water Soluble	Water Insoluble
J ₃	56	100	82
	69	99	110
K ₃	63	96	100
	62	50	104
L ₃	60	81	77
	65	84	67
M ₃	66	105	92
	59	112	113
N ₃	67	114	100
	58	106	108
O ₃	57	17	107
	68	(a)	81
P ₃	61	12	6
	64	13	58

(a) Water soluble spike not added.

TABLE 17. ANALYSIS OF VARIANCE OF THE RECOVERY OF WATER SOLUBLE AND WATER INSOLUBLE DUSTFALL SPIKES

Dustfall Fraction	Site	n	m, %	Between-Laboratory Variation			Within-Laboratory Variation		
				df	s_b , %	CV, (%)	df	s_w , %	CV, (%)
Water Soluble	Los Angeles	9	99	6	6	6	2	10	10
	Bloomington	12	112	6	49	44	5	30	27
	Manhattan	13	76	6	38	50	6	14	18
	All Sites	34	95	18	37	39	13	21	22
Water Insoluble	Los Angeles	10	99	6	8	8	3	11	11
	Bloomington	12	98	6	19	19	5	22	22
	Manhattan	14	86	6	23	27	7	18	21
	All Sites	36	91	18	18	20	15	18	20

(a) Column headings: N, number of samples; m mean dustfall; df, degrees of freedom; s_B , standard deviation of between-laboratory measurements; CV, coefficient of variation; s_W , standard deviations of within-laboratory measurements.

for all sites of 95 percent. The average standard deviations of between- and within-laboratory determinations are 37 and 21 percent, respectively, and the respective coefficients of variation are 39 and 22 percent.

The mean recovery of water insoluble dustfall ranged from 86 percent at Manhattan to 99 percent at Los Angeles. The pooled mean for all sites is 91 percent. The between- and within-laboratory variability in the determination of the water soluble dustfall is represented by average standard deviations of 18 percent in both cases and by coefficients of variation of 20 percent.

Analysis of Between-Rack and Within-Rack Variability

The statistical designs in Tables 1, 2, and 3 do not provide for evaluating the effect of location of sampling stations. The number of determinations allotted to the dustfall test method is insufficient to support any analysis capable of isolating the separate effects of laboratory, geographical site, rack location, and location of sampling stations within racks. Out of physical considerations, the last two variables, between-rack and within-rack variability, are not expected to contribute significantly to the observed measurements of reproducibility, repeatability, and recovery.

DISCUSSION AND CONCLUSIONS

The conclusions regarding the accuracy and precision of ASTM Method D 1739 for determining dustfall (settleable particulates) which may be drawn from the interlaboratory study are:

- (1) The between-laboratory component of the variability (reproducibility) inherent in the measurement of total dustfall by ASTM Method D 1739 over the range of 3.28 to 10.47 g/m²-month is represented by a coefficient of variation of 23 percent.
- (2) The within-laboratory component of the variability (repeatability) inherent in total dustfall measurements by ASTM Method D 1739 over the range of 3.28 to 10.47 g/m²-month is represented by a coefficient of variation of 16 percent.

- (3) The average results of the analysis of spiked samples indicates that recovery and analysis of the total collected particulate matter can be performed with an accuracy which is four percentage points less than the true value.
- (4) The variability of between-laboratory measurements of the water soluble dustfall component is greater than the variability of the corresponding water insoluble determinations. Since total dustfall is calculated from the sum of the water soluble and water insoluble components, the variability of the water soluble dustfall measurement exercises the greatest influence on the overall precision of the method.
- (5) The use of copper sulfate as an algicide does not appear to have measureable effect on the accuracy or precision of the water soluble, water insoluble, or total dustfall determinations. Copper sulfate was used at Los Angeles but was not used in the Bloomington and Manhattan tests.

In general, the study shows that ASTM Method D 1739 yield results with an accuracy and precision which is usually considered to be associated with more sophisticated techniques. This demonstrated competence of the Dustfall Method should bring renewed credence in validity of past measurements and should establish the usefulness of the technique for particulate measurements in the future.

RECOMMENDATIONS

The results of this study demonstrate that no major changes are required in ASTM Method D 1739 to obtain dustfall measurements of satisfactory accuracy and precision. Results with statistical characteristics comparable to those reported here can be achieved with the Test Method as it is presently written and performed. However, our experience and the experience of other users suggests the following slight modifications of the D 1739 procedure.

- (1) Since concentration of particulate material may vary considerably, guidelines for selecting the height of the collector should be more specific.
- (2) A statement should be included which suggests careful placement of the dustfall jar openings in a horizontal plane.
- (3) Many users are concerned with the effect of copper sulfate on the accuracy and precision of the dustfall data. Copper sulfate should be eliminated if deleterious effects can be proven and another algicide should be specified.
- (4) It is implied in Paragraph 7.1.6 of the Method that the correction for copper sulfate used as an algicide should be applied to the water insoluble dustfall while, in fact, it should be applied to the water soluble dustfall.

Finally, it is recommended that statements of accuracy and precision based on this interlaboratory study be incorporated in the description of the Test Method.

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APPENDIX

REPRINT OF ASTM

STANDARD METHOD FOR COLLECTION AND ANALYSIS
OF DUSTFALL (SETTLABLE PARTICULATES)

ASTM Designation: D 1739-70



Standard Method for COLLECTION AND ANALYSIS OF DUSTFALL (Settleable Particulates)¹

This Standard is issued under the fixed designation D 1739; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers a procedure for the field collection of particulates settling from atmosphere, and for preliminary characterization of the sample matter. As further analytical methods are developed for components these will be added to this method.

2. Summary of Method

2.1 Open-top collectors of a specified size and shape are located carefully outdoors to provide particulate samples that are representative of the area being studied. Collected material is taken to the laboratory in a closed container for weighing and analysis. Procedures are described for the determination of pH, total weight of settleable particulates, total water and benzene solubles, and total combustible and noncombustible matter.

3. Definitions

3.1 *settleable particulates*—for this method, any particles, liquid or solid, small enough to pass through a 1-mm screen and large enough to settle in the collector.

3.2 For definitions of other terms used in this method, refer to ASTM Definitions D 1356, Terms Relating to Atmospheric Sampling and Analysis.²

4. Interferences

4.1 Care must be taken to avoid matter from trees, bird droppings, and other such deposits. Loss of material from the collector by action of wind must be prevented. If a glass collector is used, protection against breakage by freezing of liquid sample should be provided when necessary. The sample

collector should be protected from vandalism. The criteria for selecting the sampling site (see 6.5) must be adhered to.

5. Apparatus and Materials

5.1 *Collector*—The collector shall be an open-topped cylinder with vertical sides and flat bottom. Cylinders shall be not less than 6 in. in diameter. Height of the cylinder shall be from two to three times the diameter dimension. Collectors may be made of glass, plastic, or stainless steel. (Glass, although permitted, is not preferable because of its fragility.) A holder shall be provided to secure and ensure safe positioning of the collector. The top of the collector should be at least 3 in. above any part of the holder. The holder should not interfere with operation of the collector in any way. A bird ring shall be provided on the holder (see Fig. 1).

NOTE 1—No definitive aerodynamic studies have been made of collector design, so the above specifications cannot assure optimum collection. Pending such studies, any network of stations within which comparisons are to be made should use identical collectors. If especially high winds characterize the study area, frequent inspection will reveal obvious reentrainment of dust.

5.2 *Sieve*, No. 18 (1 mm) (chemically inert), conforming to the requirements of ASTM Specification E11, for Wire-Cloth Sieves for Testing Purposes.³

5.3 *Oven*, thermostatically controlled.

¹ This method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres. A list of members may be found in the ASTM Yearbook. Current edition effective Oct. 15, 1970. Originally issued 1960. Replaces D 1739 - 62 (1967).

² *Annual Book of ASTM Standards*, Part 23.

³ *Annual Book of ASTM Standards*, Part 10.



5.4 *Filter Paper*, soft, open, rapid filtering.

5.5 *Soxhlet Extraction Apparatus* or other efficient extractor.

5.6 *Water*, conforming to ASTM Specifications D 1193, for Reagent Water.²

5.7 *Antifreeze*—Isopropyl alcohol reagent grade.

NOTE 2—Ethyl alcohol may be used, provided that the collector is polished with efficient frequency to ensure that it is not permitted to dry out and that the correct concentration of antifreeze is maintained during the sampling period. In areas where organic matter is not being studied ethylene glycol may be used.

6. Sampling

6.1 *General Sampling Principles*—Application of this method shall be guided by ASTM Recommended Practice D 1357, for Planning the Sampling of the Atmosphere.²

6.2 *Preparation of the Collector*:

6.2.1 Thoroughly rinse the collector. Place distilled water in the collector so that the level stands at one half the collector depth when the test is started. In cold weather mix a sufficient volume of antifreeze to prevent freezing with the water. In warm weather add sufficient copper sulfate as an algicide to give 15 mg/liter if the collector fills. Under the latter condition copper cannot be determined.

6.2.2 Collector liquid should be kept at a reasonable level during the testing period (at least 1 in. of water at all times).

6.3 *Sampling Time*—A sampling period shall be 1 calendar month corrected to 30 days. Allowance of ± 2 days is permissible for setting out or collecting sampling jars, or both.

6.4 *Handling Collected Sample*—No attempt shall be made to remove collected particulate sample from the collector at the field site. Collectors shall be covered and taken to the laboratory for analysis of the contents.

6.5 *Selection of Sampling Site (2,3)*⁴—The following specific recommendations shall be used as a guide in the selection of a site. If conditions do not permit application of these recommendations, note shall be made of this.

6.5.1 The sampling station shall have a free exposure so that the sample is collected by gravity settling only. It must be free from undue local sources of pollution and free from interference from buildings or other higher objects or structures. Accessibility and secu-

rity (freedom from tampering) are major considerations in the selection of a site.

6.5.2 The top of the settleable particulates container shall be a minimum of 8 ft and a maximum of 50 ft above the ground. It shall be 4 ft above any other surface, such as a roof. Higher objects, such as parapets, signs, penthouses, and the like, shall not be more than 30 deg from the horizontal, as measured in 6.5.4.

NOTE 3—Available evidence suggests, but does not prove, that the measured particulates will vary markedly over the height limits of 8 to 50 ft. It is recommended, therefore that every attempt be made to keep collector heights as constant as possible within a given network.

6.5.3 Public buildings such as schools, fire stations, and libraries, are most favorable to public agencies because of their accessibility and security.

6.5.4 Take care to avoid undue influence from one chimney, (for example, the chimney on the building of the sampling station). Whenever possible, the sampling container shall be set more than ten stack lengths from an operating stack and upwind from the prevailing wind.

6.5.5 When higher buildings in the immediate vicinity cannot be avoided, the top of any building shall be not more than 30 deg above a sampling point. That is, a line drawn from the sampling jar to the nearest edge of the highest point on any building shall form not more than 30 deg angle with the horizontal.

6.5.6 Sampling shall not be done where there is a possibility of contamination by motor traffic.

6.6 *Number of Sampling Stations (1)*—For each area or zone to be tested, a minimum of four sampling stations shall be provided. An orderly spacing of the stations shall be made so that they are approximately equally distant from each other and from boundaries of the area. Record vertical distance from collector to ground for each sampling station.

6.7 *Auxiliary information*—Weather data, including wind velocity and direction, rainfall, snowfall and barometric readings, air pollution information, and other information of interest and value should be recorded during

⁴ The boldface numbers in parentheses refer to a list of references at the end of this method.



the sampling period.

7. Procedure

7.1 Total Water Insolubles—Filtration of the sample and determination of the weight of insolubles, sample volume, and pH may be achieved in common operations. Use a filter paper for filtration. Dry the filter paper in a weighing bottle overnight at 105 C in a properly adjusted oven. After determining the tare weight of the filter paper and weighing bottle seat the paper in the funnel and filter the sample, passing it first through the No. 18 sieve, collecting the liquid in a conveniently sized volumetric cylinder. Discard any material retained on the sieve.

7.1.2 Ascertain the volume of the sample before beginning quantitative washing of any residue that may have adhered to the bottom and sides of the collector. Wet the paper when seating it and assume no significant loss during transfer.

7.1.3 The diameter of the paper and size of the filter funnel are a matter of convenience and will not influence the accuracy of this operation.

7.1.4 Before final quantitative policing of the collector with distilled water, take a portion for determination of pH, or determine the latter by direct immersion of the electrodes in the filtrate.

NOTE 4—Determination of pH is traditional but usually meaningless, since it is affected by rainfall, evaporation, the presence of antifreeze or algicide, or both, and by materials leached from large particles subsequently removed by sieve according to the procedure above. On the other hand, really extreme values (below 3 or over 11, for example) can be indicative of hazardous local conditions, so it may be worthwhile to continue making this measurement.

7.1.5 If desired, determine the acidity or alkalinity of the sample by titration of a portion taken at this point. Such determination should be in accordance with ASTM Methods D 1067, Tests for Acidity of Alkalinity of Water.² Report these data separately.

7.1.6 Finally, using a rubber policeman, free the walls of the collector from any particles, which should be washed through the sieve with distilled water. Add the washings to the filtrate through the filter. If the filtrate is to be further analyzed for specific components, adjust the volume to a convenient value

and remove portions from that. Return the filter paper containing insoluble dustfall to the weighing bottle and dry overnight at 105 C. The net weight after deducting the weight of the paper and weighing bottle represents the total water insoluble dustfall that has settled on a circular area represented by the inside diameter of opening of the collector (after correcting for copper sulfate added as an algicide).

NOTE 5—If antifreeze has been added to the collecting water, the whole sample shall be evaporated to a small volume on a hot plate, or with infrared heating lamps. The remaining evaporation should be made on a steam bath or in a 105 C oven. The residue then should be brought up to volume with distilled water and the mixture boiled and filtered as described in 7.1.

7.1.7 Ascertain all weights to the nearest 1 mg.

7.2 Benzene Solubles—Extract the filter paper containing water insoluble residue in a Soxhlet extraction apparatus, Wiley-Richardson Extractor as shown in Fig. 2 of ASTM Method D 494, for Acetone Extraction of Phenolic Molded or Laminated Products, or other suitable extraction apparatus. Introduce 50 ml of reagent grade benzene into a tared extraction flask. Heat over a water bath at a temperature sufficient to maintain a cup loading rate that would permit 10 to 15 changes of solvent in an hour. Continue the extraction for at least 2 h. Cellulose filters contain benzene soluble materials, so that a blank must be extracted and a correction made.

NOTE 6: Caution—Because of flammability and toxicity extreme caution must be taken during the manipulations. The extraction must be performed in a fireproof fume hood, away from all flame and open wiring, and with the hood fan in operation.

NOTE 7—The filter paper should be opened, folded upon itself several times in each direction, and secured with a degreased paper clip or length of bare copper wire to prevent mechanical loss of insoluble matter during extraction.

7.2.2 Remove the filter paper and its residue from the cup, and air dry carefully for at least 2 h in the fume hood, and further dry in a drying oven at 105 C. Now weigh the filter paper and record the loss in weight due to extraction by benzene as benzene solubles.

NOTE 8—This quantity may be obtained also by evaporating off the benzene from the extraction flask, using a water bath, and determining the weight of the flask and contents. This weight, less the tare weight of the flask, will yield the total



weight of benzene solubles. The eluted matter in the flask may be analyzed further after evaporation of the benzene.

7.3 Combustibles and Volatile Particulates Other than Benzene Solubles—Ash the filter paper in a tared crucible and report the loss in weight as: “Combustibles and volatile particulates other than benzene soluble.”

7.3.1 If further analyses for specific materials are desired, ignite an aliquot of the filter paper and determine “combustibles and volatile particulates other than benzene solubles” from this as in 7.3.1. Use the remaining portion for further analyses.

7.3.2 Take the aliquot as follows: Open and cut the filter paper into eight equal radial segments. Use four alternate segments in this determination, and reserve the other four for the further analyses.

7.4 Insoluble Matter—Report the net residue after deducting the weight of the crucible in the above step as “inorganic insoluble particulates.” Retain this for further analysis.

7.5 Total Water Soluble—Make up the water soluble filtrate to a convenient definite volume in a volumetric flask. Take a suitable aliquot to determine soluble salts. Transfer by means of a pipet to a weighed borosilicate evaporation dish as described in ASTM Method D 381, Existing Gum in Fuels by Jet Evaporation.⁵ If fluorides or caustic materials are suspected to be present, conduct the evaporation, in a platinum dish of convenient size. Conduct the evaporation slowly on a hot plate, or under an infrared heat lamp, until the volume is about 25 ml. Complete the evaporation on a steam bath or a thermoregulated hot plate set at a temperature not greater than 99 C. When dry, heat the dish in a 105 C oven

for a period of 2 h, cool in a desiccator, and weigh. Continue the drying procedure to constant weight.

7.5.1 Report the gain in weight of the evaporating dish, adjusted for aliquot portion, and correct for any solids present in a distilled water blank as total water solubles.

7.6 Total Inorganic Particulates—Report the combined weight of water insolubles and soluble matter corrected for any solids present in a distilled water blank as total inorganic matter.

7.7 Unit for Data—Express settleable particulates as “grams per square meter per month.”

8. Calculation

8.1 Calculate the settleable particulates, D , in grams per square meter per month as follows:

$$D = W_m/A_c$$

where:

D = settleable particulates, g/m² · month,
 W_m = total weight of settleable particulates (sum of total insolubles and water solubles) (7.1 and 7.5), and

A_c = sampling area, m²

NOTE 9—Settleable particulates may be converted to tons per square mile per months by means of the following conversion units:

$$1 \text{ g} = 1.1023 \times 10^{-6} \text{ tons, and}$$

$$1 \text{ m}^2 = 3.8608 \times 10^{-7} \text{ miles}^2$$

8.2 Express all other data in appropriate units, or as percentage of settleable particulates, D .

⁵ Annual Book of ASTM Standards, Part 17.

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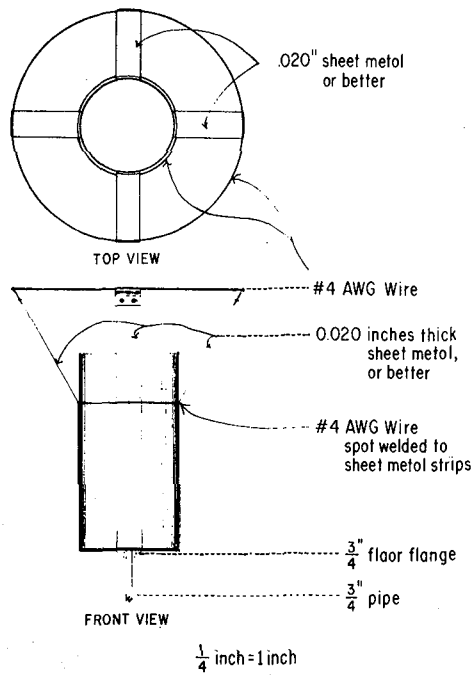


FIG. 1 Dust Fall Jar and Stand

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