A SUMMARY OF THE 1981 EPA NATIONAL PERFORMANCE AUDIT PROGRAM ON SOURCE MEASUREMENTS

by

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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 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.

The major task of this study was to report the results of the national quality assurance audit program for stationary source test methods. Audits were designed to estimate the minimal analytical and computational accuracy that can be expected with EPA Method 5 (dry gas meter only), Method 6 (sulfur dioxide), Method 7 (nitrogen oxides), and Method 19 (coal). Statistical analysis was used to characterize the data.

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ABSTRACT

In the spring and fall of 1981 the Quality Assurance Division (QAD) conducted its semi-annual National Audits for certain Stationary Source Test Methods. The audit materials consisted of a critical orifice for Method 5 (dry gas meter only), five simulated, liquid samples each for Method 6 (SO_2) and Method 7 (NO_{X}), and two coal samples for Method 19. Laboratories participating in the audits sent their data to the Source Branch and later received written reports comparing their results to EPA's.

In the Method 5 spring audit, the mean for all participants differed by 13.6% from the true (EPA) value. For the fall audit, the participants' mean was 4.3% from the true value. In the two Method 6 audits, the median values measured for 9 of 10 samples differed by less than 1% from the true value, whereas the median values for all 10 samples used in the two Method 7 audits were within 2% of the true value.

This was the first coal audit conducted by QAD. For the sulfur, ash, and moisture analysis, the participants' accuracy were consistently better for the higher concentration samples than for the lower concentration samples.

CONTENTS

Foreword	
Abstract	
Figures	
Tables .	<i></i>
Acknowled	gments
1.	Introduction
2.	Summary
3.	Dry Gas Meter Audit
4.	Method 6 Audit
5.	Method 7 Audit
6.	Coal Audit
Reference	s
Appendice	s
Α.	Frequency distributions
В.	Instructions for EPA audit materials
C.	Coal audit statistics

FIGURES

Numbe	<u>r</u>	P	age
1	Cumulative accuracy for the participants in the Method 5 audits, 0381 and 0981	•	8
2	Previous results of Method 5 audits		9
3	Results of the Method 5 Audit, 0381		10
4	Results of the Method 5 Audit, 0981		11
5	Previous results of Method 6 audits		14
6	Previous results of Method 7 audits		18

TABLES

Number	<u>r</u>	<u>P</u>	age
1	Participants' Results for Method 5 Audit (All data - no outliers removed)	•	2
2	Participants' Results for Methods 6 and 7 Audits (All data - no outliers removed	•	3
3	Participants' Results for Method 19 Audit (All data - no outliers removed	•	5
4	Method 5 Audit Participants		6
5	Method 6 Audit Participants		13
6	Summary of Source SO ₂ Audits		13
7	Method 7 Audit Participants		16
8	Summary of Source NO _x Audits		16
9	Coal Audit Participants		20
10	Source Coal Audit - 0781		21

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SECTION 1

INTRODUCTION

In 1977, the Environmental Monitoring Systems Laboratory (EMSL) of EPA established a performance audit program to evaluate the performance of companies that conduct compliance testing using EPA Reference Methods. The audits verify the analytical accuracy of EPA Reference Methods 6, 7 and 19 and the calibration accuracy of the Method 5 control console (1). By participating in this free and voluntary program, testing companies can compare their performance to other laboratories conducting similar measurements.

In 1981, two audits each were conducted for Methods 5, 6, and 7 and one audit was conducted for Method 19. Each participating laboratory received an audit package consisting of the audit sample, a data card, instructions, and an envelope for returning the data to EPA. For the Method 5 audits, a label was also included for returning the audit device. Participants had eight weeks to return data to EPA. At the end of this period, all data received were statistically analyzed to determine the participants' precision and accuracy.

This report summarizes the results of the 1981 source audits. Individual coal results reported by each participant are contained in the appendices to this report.

SECTION 2 SUMMARY

In the spring and fall of 1981, EPA's Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, conducted National Quality Assurance Audits for Stationary Source Test Methods 5 (dry gas meter only), 6 (SO_2), 7 (NO_{X}), and 19 (coal). Industrial laboratories, contractors, universities, foreign countries, and local, state, and Federal agencies participated.

Two Method 5 audits were conducted in 1981. The overall results (no outliers removed) are summarized in Table 1. In the spring and fall audits, the means for all participants were 13.6% and 4.3%, respectively, from the true value. After correcting for outliers, the means for spring and fall audits were 2.9% and 2.7% from the true value. The participants' performances were not significantly different statistically from previous national audits (2, 3, 4).

TABLE 1. PARTICIPANTS' RESULTS FOR METHOD 5 AUDIT (all data - no outliers removed)

Type of sample	Parameter	Audit date	No. of analyses	Mean (percent	Median from EPA Values)	Std. dev.
Orifice	Volume	0381	738	13.6	2.7	110.2
		0981	723	4.3	2.5	9.7

Table 2 presents the data (no outliers removed) from the two 1981 Method 6 audits. This audit procedure requires the participants to determine the sulfate content in five aqueous solutions by the Method 6 titration procedure. For each sample, the participants' means were 7%-25%

higher than the true value, but in contrast the median value in 9 out of 10 differed from the true value by less than 1%. The participants' accuracy was lowest for the lowest concentration sample, i.e., only 47% of the participants in the first audit and 36% in the second audit measured within 2% of the true value. For the four higher concentration samples, 55%-60% of the participants achieved this level of accuracy in both audits. As in the 1981 Method 5 audits, the results from the 1981 Method 6 audits do not differ significantly from those obtained in previous audits (2, 3, 4).

TABLE 2. PARTICIPANTS' RESULTS FOR METHODS 6 AND 7 AUDITS (all data - no outliers removed)

Type of		Audit	No. of	EPA (true)	Par	ticipant r	esults
sample	Parameter	date	analyses	value	Mean	Median	Std. dev.
Aqueous sulfate	so ₂	0281 0881	120 98	305.0 190.7	331.9 245.9	306 193.4	269.9 406.5
(Method	6)	0281 0881	120 98	762.6 610.1	819.6 770.7	757.9 608.3	668.9 1294.6
		0281 0881	120 98	1334.6 1296.4	1424.2 1614.8	1326.0 1285.9	1179.6 2677.8
		0281 0881	121 98	1830.3 1792.1	1997.9 2235.8	1823.8 1779.0	1613.7 3744.0
		0281 0881	120 98	2287.8 2402.2	2448.6 3011.2	2267.9 2385.8	2009.2 5090.5
Aqueous nitrate (Method	NO _X	0481 1081	89 75	119.5 159.3	147.1 164.2	118.0 162.0	165.6 56.8
(Me chod	,,	0481 1081	86 7 4	298.6 378.3	383.5 377.0	301.8 383.3	436.7 96.3
		0481 1081	88 75	497.7 557.4	625.4 566.1	502.9 562.3	706.0 150.9
		0481 1081	89 7 5	696.8 776.4	888.5 777.2	710.2 781.0	1095.8 285.4
		0481 1081	87 76	895.9 955.6	1108.8 961.5	900.0 961.3	1211.2 414.1

Table 2 also presents the data (no outliers removed) from the two Method 7 audits in 1981. This audit procedure requires the participants to determine the nitrate content in five aqueous solutions. The overall positive bias observed in Method 6 for the mean was also observed in Method 7, but the median value differed from the true value by less than 2% for all 10 samples. In contrast, the mean value for some samples was 28% higher than the true value. The participants' level of accuracy was consistent for all five samples in both audits; 31-40% of the testers measured within 3% of the true value on all samples. The participants' performance on the lowest concentration sample improved slightly compared to the previous national audits (2, 3, 4), but was not significantly different for the other four samples. On an absolute basis the accuracy for the lowest concentration sample was approximately the same as for the four higher concentration samples.

The results of the first coal audit conducted by QAD are summarized in Table 3. Participants analyzed each coal sample twice for BTU content and percent sulfur, moisture, and ash. The participants achieved results that agreed closely with the true value for sulfur and BTU determinations. However, the mean values for moisture and ash content in the low-concentration samples were as much as 11% and 20%, respectively, from the true values.

TABLE 3. PARTICIPANTS' RESULTS FOR METHOD 19 AUDIT (all data - no outliers removed)

Type of	Audit		No. of		EPA (true)	Part	icipants	' results
sample	date	Parameter	analyses	Replicate	value	Mean	Median	Std. dev.
Coal	0781	% S	98 83	1 2	1.62 1.62	1.55 1.55	1.57 1.59	0.16 0.14
		% S	97 81	1 2	0.32	0.34 .34	0.35 0.35	0.09 0.09
		%H ₂ 0	97 81	1 2	1.42 1.42	1.58 1.56	1.69 1.67	0.50 0.45
		%H ₂ 0	96 80	1 2	18.42 18.42	17.63 17.50	18.47 18.57	3.74 3.12
		%Ash	98 81	1 2	22.14 22.14	22.65 22.73	22.08 22.09	5.68 6.22
		%Ash	97 81	1 2	4.78 4.78	5.73 5.67	4.70 4.69	9.40 10.00
		BTU/1b	93 77	1 2	11339 11339	11397 11088	11269 11265	741.2 731.1
		BTU/1b	92 77	1 2	12084 12084	11684 11685	11981 11987	870.9 904.1

SECTION 3 DRY GAS METER AUDIT

In the Method 5 audit procedure, participants use a critical orifice to check the calibration of the dry gas meter in their EPA Method 5 control console (meter box). They insert the orifice in the Method 5 meter box, allow the box to warm up, and then make three 15-min volume measurements. Using Equation 5-1 of Method 5, they convert each of the three volumes to cubic meters at standard conditions, record the volumes on the data card, and mail the device and the data card to EPA for statistical analysis.

In the spring audit (0381), 77% of the 170 laboratories that received the audit package returned data. In the fall audit (0981), 75% of the 180 laboratories returned data. These percentages are similar to those encountered in previous audits (2, 3, 4). Table 4, which classifies the participants into general categories, shows the number of laboratories that requested to participate in the Method 5 audit and the number that actually returned data.

TABLE 4. METHOD 5 AUDIT PARTICIPANTS

	No. request	ting samples	No. returning data		
	0381	0981	0381	0981	
Contractors	92	89	67	62	
Industry	40	52	34	43	
Foreign	4	5	4	5	
Federal	3	4	2	3	
State	24	22	19	16	
Local	7	8	5	6	
TOTAL	170	180	131	135	

Figure 1, a cumulative histogram, shows the absolute accuracy obtained by participants in the 0381 and 0981 Method 5 audits, expressed as the percentage of participants whose measurements agree with the true (EPA) value at various levels of accuracy. The Code of Federal Regulations (1) requires that the dry gas meter be calibrated with an accuracy of \pm 2 percent. Figure 1 shows that only 42% of the reporting laboratories in the 0381 audit and 44% in the 0981 audit obtained this accuracy. These results are similar to those reported in previous audits (Figure 2). One hundred and one laboratories participated in both audits.

The histograms in Figures 3 and 4 show how the individual results of the 0381 and 0981 audits compared to the mean and the median values for all participants. The majority of the laboratories reported values lower than the EPA value. The standard deviation of the triplicate analyses (precision) by each laboratory indicated that for the 0381 audit, 65% of the standard deviations for each set were within 0.3%. For the 0981 audit, 68% of the standard deviations were within 0.3%. Six percent of the 0381 data and 5% of the 0981 data were identified as outliers using Chauvenet's Criterion (5). Before the outliers were removed, the mean values for the 0381 and 0981 data differed by 13.6% and 4.3% from the true value, respectively. After deletion of outliers, these values were reduced to 2.9% and 2.7%, respectively.

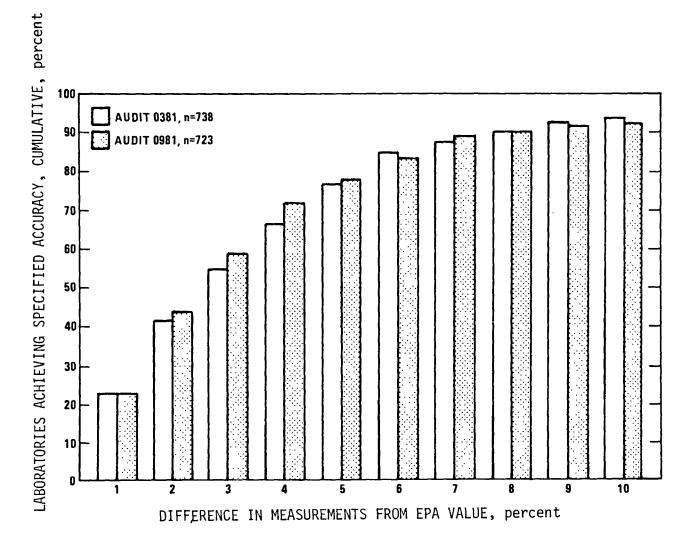


Figure 1. Cumulative accuracy for participants in the Method 5 audits, 0381 and 0981.

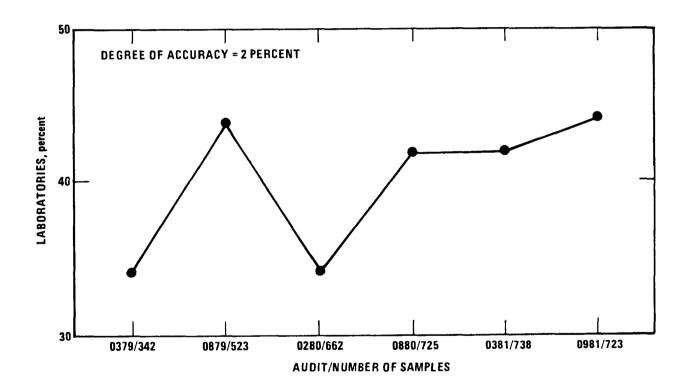


Figure 2. Previous results of Method 5 audit

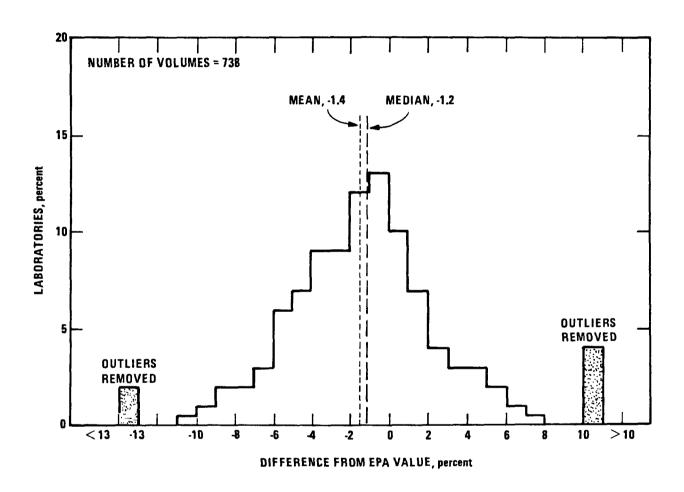


Figure 3. Results of the Method 5 Audit 0381

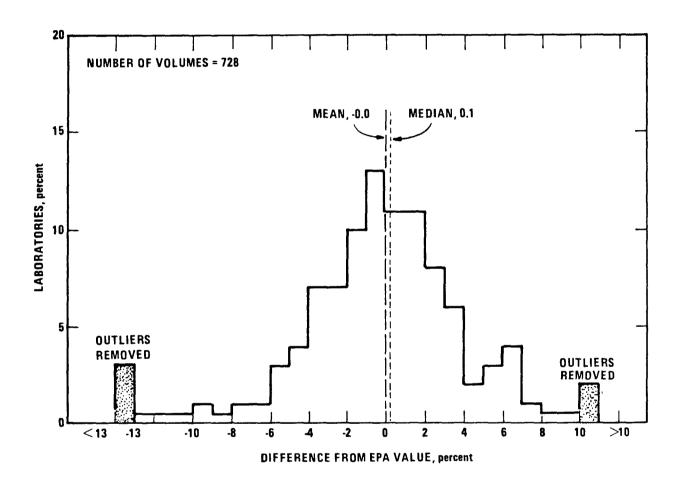


Figure 4. Results of the Method 5 Audit 0981

SECTION 4

METHOD 6 AUDIT

This audit checks the participants' ability to analyze a Method 6 sample for sulfate. The audit set consists of five dilutions of 10 N sulfuric acid ($\rm H_2SO_4$) in 25-ml sealed glass ampoules. These five ampoules contained different concentrations, ranging from 0 to 3000 mg $\rm SO_2$ per dry standard cubic meter (DSCM). The analyst withdraws 5.0 ml from each ampoule, adds 30 ml of 3% hydrogen peroxide, and dilutes the sample to 100 ml with distilled water. A 20-ml aliquot is then withdrawn from the diluted sample, 80 ml of 100% isopropanol and thorin indicator are added, and the sample is titrated with barium perchlorate ($\rm Ba[ClO_4J_2)$) to a pink endpoint. To calculate the results, the participants assume they had an original sample volume of 100 ml, and that they had sampled 21 x $\rm 10^{-3}$ DSCM of stack gas.

In the spring audit (0281), 79% of the 154 laboratories that received the audit package returned data. In the fall audit (0881), 62% of the 157 laboratories returned data. These percentages are similar to those encountered in previous audits (2, 3, 4). Table 5, which classifies the participants into general categories, shows the total number of laboratories requested to participate in the Method 6 audit and the number that returned data. Seventy-three laboratories participated in both audits and returned data.

Table 6 presents the percent of laboratories that achieved 2% and 5% accuracy for each of the five different concentrations in the two 1981 Method 6 audits. In the 0281 audit, 47% of the reporting laboratories achieved an accuracy within 2% for the lowest concentration and in the 0881 audit, 36% of the laboratories achieved an accuracy within 2%. However, in

both audits, approximately 58% of the participants achieved an accuracy within 2% for the four higher concentration samples. The poor accuracy obtained for the lowest sample likely results from the difficulty in determining the thorin endpoint. Approximately 80% of the laboratories were able to achieve an accuracy level within 5%.

TABLE 5. METHOD 6 AUDIT PARTICIPANTS

	No. reques	ting samples	No. returning data		
	0281	0881	0281	0881	
Contractors	87	84	69	55	
Industry	37	42	27	26	
Foreign	4	2	1	1	
Federal	1	2	1	1	
State	16	18	14	8	
Local	9	9	8	7	
TOTAL	154	157	121	98	

TABLE 6. SUMMARY OF SOURCE SO_2 AUDITS

	0281		0881	
Concentration	±2%	±5%	±2%	±5%
0 - 500 mg/DSCM	47%	77%	36%	64%
501 - 1000 mg/DSCM	55%	81%	58%	79%
LOO1 - 1500 mg/DSCM	55%	82%	60%	87%
L501 - 2000 mg/DSCM	58%	80%	59%	86%
2001 - 3000 mg/DSCM	58%	79%	57%	88%
n	12	1	9	8

The results obtained in the 1981 Method 6 audit do not differ significantly from those obtained in previous audits (Figure 5).

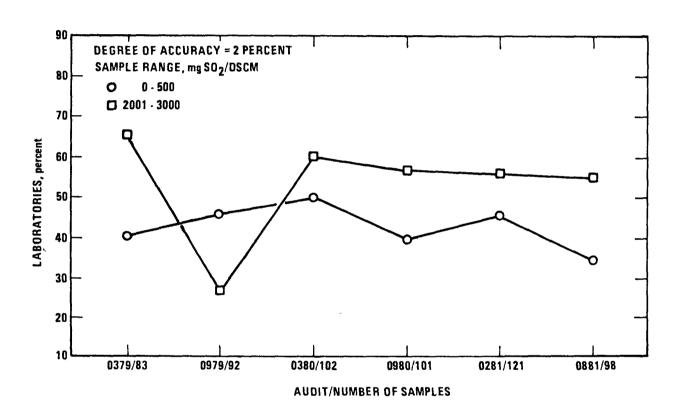


Figure 5. Previous results of Method 6 audits

SECTION 5

METHOD 7 AUDIT

This audit checks the participants' ability to analyze a Method 7 sample for nitrate. The NO_{X} audit set consists of five dilutions of potassium nitrate (KNO $_3$) stock solution in 25-ml glass ampoules that are autoclaved after sealing to destroy bacteria that might attack the nitrate. The five samples in the set simulate source samples ranging from 0 to 1000 mg $\mathrm{NO}_2/\mathrm{DSCM}$. The analyst withdraws 5.0 ml from an ampoule, adds this and 25 ml of the Method 7 absorbing solution to a flask, adjusts the pH to 9-12 with NaOH, and then dilutes the solution to 50.0 ml with distilled water. Then the analyst withdraws a 25-ml aliquot from the diluted sample, places it in an evaporating dish, and treats it as described in Section 4.3 of Method 7. After the treatment is completed, the absorbance is measured at 410 nm. For the concentration calculations, the participant assumes that 2000 ml of stack gas has been sampled.

In the spring audit (0481), 72% of the 124 laboratories that received the audit package returned data. In the fall audit (1081), 60% of the 126 laboratories returned data. These percentages are similar to those encountered in previous audits (2, 3, 4). Table 7 shows the total number of laboratories requesting participation and the number that returned data for Method 7 audits 0481 and 1081.

TABLE 7. METHOD 7 AUDIT PARTICIPANTS

		ting samples	No. returning data		
	0481	1081	0481	1081	
Contractors	75	78	50	48	
Industry	26	26	22	13	
Foreign	3	4	2	2	
Federal	1	1	1	0	
State	11	10	8	7	
Local	7	7	6	6	
TOTAL	124	126	89	76	

Table 8 shows the percentages of laboratories that achieved 3% and 7% accuracy for each of the five concentrations. For the 0481 and 1081 audits, 31% and 36% of the reporting laboratories achieved accuracy within 3% for the lowest concentration samples. Sixty percent of the laboratories were able to achieve accuracy within 7% for all samples in both audits.

TABLE 8. SUMMARY OF SOURCE NO_{χ} AUDITS

	0481		1081	
Concentration	±3%	±7%	±3%	±7%
0 - 200 mg/DSCM	31%	57%	36%	55%
201 - 400 mg/DSCM	40%	64%	39%	58%
401 - 600 mg/DSCM	36%	64%	34%	59%
601 - 800 mg/DSCM	35%	67%	38%	59%
801 - 1000 mg/DSCM	33%	60%	34%	58%
n	8	9	7	6

Figure 6 compares the results of the 1981 audit to those of the past six audits. For the four highest concentrations, the percentage of laboratories obtaining 3% accuracy was between 30% and 35%. The percentage of laboratories obtaining 3% accuracy for the lowest concentration has varied widely from audit to audit, but seems to have improved in the 1981 audits.

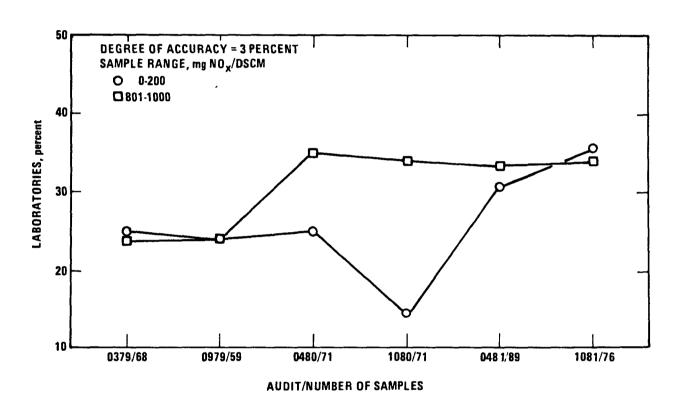


Figure 6. Previous results of Method 7 audit

SECTION 6

COAL AUDIT

Method 19 of Appendix A in Part 60, Title 40 of the Code of Federal Regulations (CFR) allows coal sampling and analysis to serve as an acceptable method to determine the scrubber inlet sulfur emission rate for new large coal-fired power plants. The coal audit checks the participant's ability to analyze a coal sample for sulfur, ash, moisture, and BTU content. Acceptance Testing on the NBS-supplied audit samples was done by an EPA contractor using the following instrumentation: LECO SC132 (sulfur), Fisher Model 490 (ash and moisture), and Parr Model 1241 Calorimeter (BTU content). This was the first coal audit conducted by QAD.

Each set of coal samples consisted of two bottles containing 13 grams of 60 mesh coal. Participants measured sulfur, moisture, ash, and gross calorific value of each sample. The following American Society for Testing and Materials (ASTM) procedures were recommended, but not necessarily mandated (6).

- ASTM D-3177 (Standard Test Method for Total Sulfur in the Analysis of Coal and Coke);
- ASTM D-3174 (Standard Test Method for Ash in the Analysis Sample of Coal and Coke);
- · ASTM D3173 (Test for Moisture in the Analysis Sample of Coal); and
- ASTM D-2015 (Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Method) (9).

The participants reported their results for moisture (%) on an as received basis, and their results for sulfur (%), ash (%), and gross calorific value (BTU/1b) on a dry basis.

Table 9 shows the total number of laboratories requesting samples and the number that returned data (83%). Due to the high cost of shipping coal samples outside of the United States, no foreign laboratories were able to participate.

TABLE 9. COAL AUDIT PARTICIPANTS

	No. requesting samples 0781	No. returning data 0781
Contractors	28	22
Industry	32	28
Foreign	0	0
Federal	2	1
State	11	11
Local	4	2
TOTAL	77	64

Table 10 summarizes the coal audit results. The numbers of analyses in Table 10 are greater than the number of participants because some companies had more than one laboratory participating. In this case, each laboratory received its own set of samples and each was asked to analyze the samples in duplicate. However, some laboratories exhausted their sample set on the first analysis. Therefore, accuracies of 5% and 10% were chosen for the precision criterion for each of the four parameters.

Only 20% of the laboratories were able to analyze the sulfur and moisture content of the low level samples within 10% of the NBS value. In contrast, more than 85% of the laboratories were able to analyze the higher concentration sample within 10% of the NBS moisture and sulfur values.

For the ash analysis, 77% and 98% of the participants achieved an accuracy within 10% for the lower and higher ash samples, respectively. Ninety-five percent of the participants analyzed the lower BTU sample within 10% and 90% analyzed the higher BTU sample within 10% of the NBS value.

Table 10 also shows that the participant's accuracy was consistently better for the higher concentration samples for the sulfur, ash, and moisture analysis than for the lower concentration samples. For those that did duplicate analyses, the within-laboratory precision showed no correlation with concentration. Therefore, the standard deviation (precision) was independent of the sample concentration for all four parameters.

TABLE 10. SOURCE COAL AUDIT - 0781

Expected Value	Number analys		Laboratories accurate within ±5%	Laboratories accurate within ±10%
		S	ULFUR	
0.32%		97 81	15% 17%	21% 20%
1.62%		98 83	57% 65%	82% 87%
		MO	ISTURE	
1.42%	• ,	97 81	15% 17%	21% 20%
18.42%		96 80	76% 83%	87% 91%
			ASH	
4.78%		97 81	55% 54%	76% 78%
22.14%		98 81	97% 99%	98% 99%
		- GROSS	CALORIFIC	
1339 BTU/1b		93 77	90% 9 4 %	95% 96%
2084 BTU/1b	` '	92 77	84% 86%	90% 90%

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APPENDIX A

FREQUENCY DISTRIBUTIONS

DGM FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCE - 0381

#SAMP	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD. DEV.	SKEWNESS	MEDIAN
738	. 0	. 4	. 9	1.4	1.9	2.7	3.4	4.3	5.3	7.6	1504.0	13.6	110.2	11.87	2.7
732	.0	. 4	.9	1.4	1.9	2.7	3.3	4.2	5.3	7.3	42.8	3.9	5.3	00	2.7
711	. 0	. 4	. 9	1.3	1.8	2.6	3.2	4.1	5.0	6.6	15.7	3.2	2.8	00	2.6
695	.0	. 4	. 9	1.3	1.8	2.4	3.1	3.9	4.9	5.9	11.8	2.9	2.3	01	2.4
693	.0	. 4	.9	1.3	1.8	2.4	3.1	3.9	4.9	5.9	9.9	2.9	2.2	01	2.4

DGM FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCE - 0981

#SAMP	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD. DEV.	SKEWNESS	MEDIAN
723	.0	. 4	.8	1.3	1.8	2.5	3.1	3.8	5.3	7,5	94.4	4.3	9.7	7.65	2.5
714	.0	. 4	. 8	1.3	1.8	2.5	3.0	3.6	5.2	7.0	17.6	3.3	3.3	07	2.5
696	. 0	. 4	. 8	1.2	1.7	2.4	2.9	3.5	4.9	6.6	14.0	3.0	2.7	15	2.4
685	. 0	. 4	. 8	1.2	1.7	2.3	2.9	3.4	4.7	6.3	11.7	2.9	2.4	24	2.3
680	.0	. 4	.8	1.2	1.7	2.3	2.8	3.4	4.6	6.1	10.5	2.8	2.3	32	2.3
678	. 0	. 4	. 8	1.2	1.6	2.3	2.3	3.4	4.5	6.1	10.2	2.8	2.2	40	2.3
676	.o	. 4	.8	1.2	1.6	2.3	2.8	3.4	4.4	6.0	10.1	2.8	2.2	48	2.3
675	.0	. 4	.8	1.2	1.6	2.3	2.8	3.4	4.4	5.9	10.0	2.7	2.2	55	2.3

SOURCE ${\bf so_2}$ frequency distribution of percent difference with outliers removed - 0281

LEVEL (MG/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1 - 500.0	103	. 00	. 23	. 46	. 66	1.31	1.64	2.23	2.92	4.26	4.92	7.41	2.27	1.96
500.1 - 1000.0	102	. 00	. 29	. 62	. 80	1.02	1.49	1.74	2.02	2.49	3.54	5.49	1.69	1.31
1000.1 - 1500.0	101	.00	. 16	.40	. 58	1.09	1.38	1.74	2.14	2.56	3.72	5.18	1.66	1.34
1500.1 - 2000.0	101	. 08	. 32	. 40	. 68	1.04	1.25	1.50	1.93	2.58	4.00	5.29	1.69	1.40
2000.1 - 3000.0	103	. 00	. 20	. 47	. 79	1.25	1.48	1.67	2.08	2.77	4.24	6.34	1.85	1.57

SOURCE ${
m SO}_2$ FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE WITH OUTLIERS REMOVED - 0881

LEVEL (MG/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1 - 500.0	87	.00	. 37	1.21	1.63	1.94	2.36	3.30	4.46	5.66	8.60	9.86	3.53	2.80
500.1 - 1000.0	84	.00	'.15	. 48	. 67	. 90	1.21	1.67	2.08	2.80	4.44	6.21	1.81	1.67
1000.1 - 1500.0	86	.02	. 28	. 49	. 88	1.05	1.20	1.49	2.04	2.65	3.56	5.03	1.65	1.26
1500.1 - 2000.0	86	. 12	. 34	. 59	. 68	. 90	1.23	1.58	2.28	2.64	3.63	5.17	1.69	1.34
2000.1 - 3000.0	86	. 02	. 25	.51	. 68	. 97	1.26	1.61	2.22	2.59	3.88	4.96	1.71	1.36

SOURCE NO $_{\chi}$ FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE WITH OUTLIERS REMOVED 0481

LEVEL (MG/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1 - 200.0	73	.42	. 42	1.92	2.68	3.77	4.94	5.44	6.78	8.70	19.46	15.48	5.51	3.98
200.1 - 400.0	67	.03	. 20	. 54	1.24	2.08	2.85	3.82	4.19	5.89	7.50	11.19	3.53	3.00
400.1 - 600.0	73	. 06	. 30	1.08	1.89	2.73	3.46	4.26	5.49	7.49	9.60	13.52	4.35	3.47
600.1 - 800.0	75	. 04	. 75	1.81	2.05	2.76	3.46	4.28	5.05	6.92	9.07	12.54	4.34	3.2
800.1 - 1000.0	71	. 12	. 90	1.33	1.83	2.58	3.64	4.79	5.93	7.23	11.62	16.08	4.92	4.1

SOURCE NO_X FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE WITH OUTLIERS REMOVED 1081

EVEL (MG/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1 - 200.0	62	. 19	. 69	1.44	2.26	2.70	3.95	5.40	7.97	8.66	12.99	18.39	5.64	4.82
200.1 - 400.0	58	. 08	. 29	. 69	1.51	1.93	2.83	3.52	4.49	5.21	6.16	10.79	3.45	2.83
400.1 - 600.0	67	. 07	. 43	1.00	2.19	2.76	3.98	5.40	7.32	11.71	13.89	21.10	6.11	5.60
600.1 - 800.0	62	. 04	. 30	. 59	1.08	2.20	3.54	4.89	6.13	8.57	12.70	17.10	5.09	4.93
800.1 - 1000.0	62	. 06	. 83	1.25	1.97	2.45	3.89	5.63	6.45	8.68	14.61	18.99	5.69	5. 2

NATIONAL COAL AUDIT FREQUENCY DISTRIBUTION
OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES - 0781

SAMPLE	NO.	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD. DEV
								SULFUR -						
2000 5000	181 178	.00	. 62 3. 13	1.23 6.25	1.85 9.38	2.47 12.50	3.70 12.50	4.94 15.63	6.79 18.75	8.64 31.25	16.05 43.75	45.06 93.75	6.49 19.95	8.04 19.66
								MOISTURE						
2000 5000	178 176	.00	2.11 .43	9.15 1.03	15.49 1.36	19.72 1.85	23. 94 2. 55	28.17 2.99	33. 10 3. 53	38.73 5.05	47.89 13.14	164. 08 95. 49	27. 15 7. 32	22.36 17.70
								- ASH						
2000 5000	179 178	. 00 . 00	. 09 . 63	. 14 1. 26	. 27 1. 88	. 41 2. 72	. 54 4. 18	. 68 5. 65	. 86 7. 11	1.22 12.34	2.26 18.83	252.08 1876.78	3.76 30.61	26.55 200.52
							GROS	S CALOROF	IC					
2000 5000	170 169	.01 .01	. 21 . 12	. 37 . 31	. 51 . 41	. 59 . 67	. 69 . 89	. 86 1. 18	1. 28 1. 94	1.84 3.91	3. 37 9. 99	46.76 45.86	2.38 3.56	6.31 7.19

APPENDIX B INSTRUCTIONS FOR EPA AUDIT MATERIALS

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY METHOD 5 DRY GAS METER PERFORMANCE TEST DEVICE

NOTE: All procedures referrred to are from revised Method 5 published in the Faderal Register, Vol. 12, No. 160, Part II, Thursday, August 18, 1977, pp. 41776-41782 and references contained therein. This revised method should be adhered to in all details in the use of this quality assurance performance device.

EQUIPMENT: The participant in this study should possess the following equipment, including the performance test device supplied by EPA.

Quantity Item

1 Method 5/Source Sampling Meter Box

Stopwatch, preferably calibrated in decimal minutes

1 Thermometer, ambient range

Barometer. If unavailable, call nearest National Weather Service and request the ABSOLUTE barometric pressure. (Corrected for temperature and acceleration due to gravity, but not corrected for altitude.)

Performance Test Device. A calibrated flow orifice housed in a quick-connect coupling and identified with an engraved three-digit serial number.

WARNING: THE DEVICE MUST NOT BE DISASSEMBLED UNDER ANY CIR-CUMSTANCES. Use these devices at room temperature.

PROCEDURE:

- 1. Remove the performance test device from its case and insert it into the gas inlet quick-connect coupling on the source sampling meter box.
- 2. Turn the power to the meter box on and start the pump.
- 3. Adjust the coarse flow rate control valve and the fine flow rate control valve to give a maximum vacuum reading. CAUTION: A vacuum reading of less than 17 inches Hg will result in flow rate errors.
- 4. Allow the orifice and source sampling meter box to warm up for 45 minutes with flow controls adjusted as described in Step 3 before starting quality assurance runs.
- 5. Make triplicate quality assurance runs. For each run, record initial and final dry gas meter volumes, dry gas meter inlet and outlet temperatures, internal orifice pressure drop (△H), ambient temperature, and barometric pressure. Run duration should be slightly greater than 15 minutes. The following procedure is recommended. Fifteen minutes after a run is started, the participant watches the dry gas meter needle closely. As the needle reaches the zero (12 o'clock) position, the pump and stopwatch are stopped simultaneously. The dry gas meter volume and time are recorded.
 - This complete run procedure is performed three times to provide the required triplicate quality assurance runs.
- 6. Calculate the corrected dry gas volume for each run using equation 5.1 of the above-refer enced Method 5. For each replicate, record the corrected dry gas volume in dry standard cubic meters, the sampling time in decimal minutes, the barometric pressure in mm Hg, and the ambient temperature in degrees Celcius on the enclosed data card. Be sure to record the performance test device serial number on the data card in the column headed "Orifice Number."
 - NOTE 1: If you calculate dry gas volume in English Units, use the conversion factor of 0.02832 m³ ft³ to obtain the volume in metric units.
 - NOTE 2: If your stopwatch is not in decimal minutes, be sure to convert (e.g. 15 minutes 20 seconds is reported as 15.33 minutes).
- 7. After recording the requested data on the enclosed data form, return the data form and the performance test device to:

Quality Assurance Division (MD-77) Environmental Monitoring Systems Laboratory Environmental Protection Agency Research Triangle Park, North Carolina 27711

Attention: Robert G. Fuerst

A postpaid return envelope and label are enclosed for this purpose.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE SO₂ REFERENCE SAMPLES

Note: All Method 6 procedures referred to are from the amended method published in the Federal Register Vol. 42, No. 160, Part II, Thursday, August 18, 1977, pp 41782-41784. This amended method should be adhered to in all details in the analysis of these reference standards.

- 1. Prepare 3-percent hydrogen peroxide according to Section 3.1.3 of the method (30 ml is required for each sample and each blank).
- 2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampule and with the ampule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampule pipette exactly 5 ml of the reference sample into a 100-ml volumetric flask. Add 30 ml of 3-percent hydrogen peroxide solution. Dilute exactly to the mark with deionized, distilled water. Analyze the sample in accordance with the procedure detailed in Section 4.3 of the method, beginning with "Pipette a 20-ml aliquot of this solution...." (Note: If more than 50 ml of barium perchlorate titrant is required for any sample analysis, a smaller aliquot should be selected to allow titration with less than 50-ml titrant.)
- 3. Calculate the concentration, C_{SO_2} (concentration of sulfur dioxide, dry basis, corrected to standard conditions, mg/dscm), using Equation 6-2. A value of 21 X 10^{-3} dscm should be used for $V_{m(std)}$, in the equation. A value of 100 ml should be used for V_{soln} in the equation.
- 4. Record the reference standard sample numbers and their corresponding SO₂ concentrations in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD 77)
Environmental Monitoring Systems Laboratory
Environmental Protection Agency
Research Triangle Park, N.C. 27711
Attn: Robert G. Fuerst

If other than EPA Method 6 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE NO_x REFERENCE SAMPLES

Note: All Method 7 procedures referred to are from the amended method published in the Federal Register Vol. 42, No. 160, Part II, Thursday, August 18, 1977, pp 41784-41786. This amended method should be adhered to in all details in the analysis of these reference standards.

- 1. Prepare absorbing solution according to Section 3.1 of the method.
- 2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampule and with the ampule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampule pipette exactly 5 ml of the reference sample into a 100-ml beaker. Add 25 ml absorbing solution to the beaker; adjust the pH to 9-12 (using pH paper as indicated in Section 4.2 of the method) by dropwise addition of sodium hydroxide (1N). Quantitatively transfer the contents of the beaker to a 50-ml volumetric flask and dilute exactly to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of the diluted sample into a porcelain evaporating dish. Beginning with the evaporation step in Section 4.3, complete the sample analysis.
- 3. Calculate total μ g NO₂ per sample using Equation 7-3. Calculate the sample concentration, C (concentration of NO_X as NO₂, dry basis, corrected to standard conditions, mg/dscm), using Equation 7-4. A value of 2000 ml should be used for V_{SC} in Equation 7-4.
- 4. Record the reference sample numbers and their corresponding concentrations, C, in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD 77)
Environmental Monitoring Systems Laboratory
Environmental Protection Agency
Research Triangle Park, N.C. 27711
Attn: Robert G. Fuerst

If other than EPA Method 7 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

COAL AUDIT PROGRAM INFORMATION

- 1. There is approximately 13 grams of 60 mesh coal per bottle.
- 2. Analyze the coal samples for moisture and on a <u>dry basis</u> for ash, sulfur and gross calorific value. Report moisture, ash, and sulfur in weight percent with gross calorific value reported as BTU/lb.
- 3. All methods used in the analysis of these coal samples should follow American Society for Testing and Materials (ASTM) recommended procedures or an accepted automatic analytical device.
- 4. Suggested procedures are:

Moisture D-3173
Ash D-3174
Sulfur D-3177
Gross Calorific Value ... D-2015

Please note on the data card (columns 17-32) the ASTM method number. If an ASTM method was not used for analysis note that on the back of the data card. Be parameter specific.

- 5. If you cannot analyze the coal sample for all four parameters, analyze for what you can. Analysis of moisture is necessary to calculate on a dry basis any of the other three parameters. Analysis of sulfur is also necessary for the calculation of gross calorific value.
- 6. Analyze each sample in duplicate (if possible) and record results as analysis 1 and analysis 2 for each parameter.
- 7. Most laboratories will use site number 001. Multiple site numbers are used by laboratories that receive more than one set of samples. These central laboratories have requested auditing of their satellite laboratories.
- 8. After recording the requested data on the enclosed data card, return the data card to:

Mr. Robert G. Fuerst Quality Assurance Division (MD-77) Environmental Monitoring Systems Laboratory U.S. Environmental Protection Agency Research Triangle Park, NC 27711

A postpaid return envelope is enclosed for this purpose.

9. If you have any questions concerning this or any source method audit, please call (919/541-2220).

APPENDIX C

COAL AUDIT STATISTICS

Parameter: Sulfur Sample Number: 20 Analysis: 1 Number of OBS: 98		Expected Value: Mean: 1.55 Median: 1.57 Variance: .03		Coef. Var.: 10.53 Skewness: -1.14	
		DATA IN ASCENDIN	G ORDER		
.89 1.25 1.37 1.46 1.48 1.51 1.53 1.54 1.56 1.57 1.58 1.60 1.60 1.61 1.62 1.63 1.65 1.69 1.71 1.86	1.02 1.26 1.39 1.47 1.49 1.51 1.53 1.54 1.56 1.57 1.58 1.60 1.61 1.61 1.63 1.64 1.65 1.65 1.74	1.13 1.28 1.39 1.47 1.50 1.53 1.54 1.55 1.56 1.57 1.60 1.61 1.61 1.63 1.64 1.66 1.70 1.76 2.00		1. 15 1. 34 1. 45 1. 48 1. 50 1. 53 1. 54 1. 56 1. 57 1. 59 1. 60 1. 61 1. 62 1. 63 1. 65 1. 67 1. 71	1.25 1.35 1.46 1.48 1.50 1.53 1.54 1.56 1.58 1.60 1.61 1.62 1.63 1.65 1.71 1.86
Parameter: Sulfur Sample Number: 20 Analysis: 2 Number of OBS: 83	000	Expected Value: Mean: 1.55 Median: 1.59 Variance: .02		Std. Dev.: .14 Coef. Var.: 9.15 Skewness: -2.15 Accuracy: -1.85	
		DATA IN ASCENDIN	G ORDER		
.91 1.33 1.46 1.49 1.51 1.54 1.55 1.58 1.59 1.60 1.61 1.62 1.64 1.65 1.67 1.72	1.00 1.33 1.46 1.50 1.52 1.54 1.56 1.58 1.59 1.60 1.60 1.62 1.63 1.64 1.65 1.68	1.14 1.33 1.48 1.50 1.52 1.54 1.57 1.58 1.59 1.60 1.61 1.62 1.63 1.64 1.65 1.68		1.30 1.43 1.48 1.51 1.52 1.54 1.57 1.58 1.59 1.60 1.61 1.62 1.64 1.64	1.31 1.44 1.49 1.51 1.53 1.55 1.57 1.59 1.60 1.61 1.62 1.64 1.64

Parameter: Sul Sample Number: Analysis: 1 Number of OBS:	5000	Expected Value: .32 Mean: .34 Median: .35 Variance: .01	Std. Dev.: .09 Coef. Var.: 25.29 Skewness:48 Accuracy: 9.38
************		DATA IN ASCENDING ORDER	
. 05	. 07	.07	.19 .27
. 22	. 23	. 24	.25 .25
. 26	. 26	. 27	.28 .28
. 28	. 28	. 28	. 29 29
. 29	. 29	. 30	.30 .30
. 30	.31	.31	.31 .31
. 31	. 32	. 32	. 32 32
. 32	. 33	. 33	.33 .33
. 33	. 34	. 34	.34 .34
. 34	. 34	. 34	. 35 . 35
. 35	. 35	. 35	.35 .36
. 36	. 36	. 36	.36 .36
. 36	. 36	. 37	. 37
. 37	. 37	. 37	.37 .37
. 37	. 38	. 38	. 38
. 38	. 38	. 38	. 39
.40	.41	. 42	.42 .43
. 43	. 46	. 46	. 46
. 47	. 47	. 47	. 49 . 52
. 54	.60		
Parameter: Sul		Expected Value: .32	Std. Dev.: .09
Sample Number:	50 00	Mean: .34	Coef. Var.: 25.50
Analysis: 2		Median: .35	Skewness:56
Number of OBS:	81	Variance: .01	Accuracy: 9.38
***********		DATA IN ASCENDING ORDER	
. 05	. 07	.08	.18 .22
. 24	. 25	. 26	. 26
. 27	.27	.28 .31 .32 .33 .34 .34 .35 .36 .36 .37 .38 .40	. 26
. 30	. 30	.31	.31 .31
.31	.31	. 32	. 32
. 32	. 32	. 33	. 33
. 34	. 34	. 34	. 34
. 34	. 34	. 34	. 34 . 35
. 35	. 35	. 35	. 35
.31 .32 .34 .34 .35 .35 .36	.35 .35 .36	. 30	.35 .36 .36 .36 .37
. 30 70	. 30 27	. 30 7.	. 3/ . 3/
.3/	.37	. 37	.37 .38
.38	.38	. 38	.38 .38
.40	.40	. 4U	.42 .42
.43	.43	.43	.43 .45
.45	.49	. 49	.51 .53
. 62			

Parameter: Mois Sample Number: Analysis: 1 Number of OBS:	2000	Expected Value: Mean: 1.58 Median: 1.69 Variance: 31.48		Std. Dev.: .50 Coef. Var.: 31.4 Skewness:30 Accuracy: 19.01	18
	****	DATA IN ASCENDIN	G ORDER		
.03 .65 1.00 1.21 1.35 1.42 1.45 1.53 1.62 1.65 1.69 1.73 1.77 1.81 1.84 1.87 1.92 2.00	.03 .82 1.03 1.21 1.36 1.43 1.47 1.55 1.64 1.68 1.70 1.74 1.78 1.82 1.84 1.88 1.96 2.01	.10 .87 1.10 1.29 1.38 1.44 1.47 1.57 1.65 1.69 1.71 1.75 1.79 1.82 1.85 1.89		.54 .87 1.12 1.30 1.41 1.44 1.49 1.60 1.65 1.69 1.71 1.75 1.79 1.82 1.85 1.89	.63 .91 1.20 1.33 1.41 1.51 1.65 1.69 1.71 1.76 1.81 1.83 1.86
2.07 2.21	2.01 2.07 3.75	2.01 2.10		2.02 2.18	2.04 2.19
Parameter: Moist Sample Number: 2 Analysis: 2 Number of OBS: 8	2000 31	Expected Value: Mean: 1.56 Median: 1.67 Variance: .20		Std. Dev.: .45 Coef. Var.: 28.8 Skewness: -1.65 Accuracy: 17.61	
		DATA IN ASCENDING	JUNDER		
.03 .85 1.15 1.39 1.43 1.45 1.58 1.64 1.67 1.73 1.76 1.79 1.86 1.91 1.94 1.97 2.19	.04 .86 1.20 1.40 1.43 1.46 1.65 1.69 1.73 1.77 1.80 1.87 1.92 1.95	.12 .91 1.24 1.40 1.44 1.61 1.65 1.69 1.74 1.78 1.82 1.88 1.92 1.95 2.05		.44 .93 1.34 1.41 1.44 1.53 1.61 1.65 1.71 1.74 1.79 1.83 1.91 1.93 1.96 2.10	.61 .96 1.36 1.42 1.45 1.57 1.64 1.71 1.75 1.79 1.83 1.91 1.93 2.18

Parameter: Moi Sample Number: Analysis: 1 Number of OBS:	5000 96	Expected Value: 18.42 Mean: 17.63 Median: 18.47 Variance: 13.99	Skewness: -3. Accuracy: .30	1.22 51
		- DATA IN ASCENDING ORDE	R	
	25	1 66	1.78	10.63
. 83	. 85	1.66 15.82	16.00	16.50
11.39	15.74	17.28	17.37	17.41
17.03	17.22 17.58	17.60	17.61	17.63
17.43	17.83	17.83	17.90	17.90
17.66 17.95	17.95	18.03	18.05	18.12
18.15	18.15	18.17	18.23	18.23
18. 26	18.29	18.29	18.30	18.38
18.39	18.39	18.39	18.46	18. 4 6
18.47	18.47	18.47	18.48	18. 4 8
18.49	18.50	18.50	18.53	18.58
18.59	18.64	18.67	18.67	18.69
18.74	18.74	18.76	18.76	18.77
18.77	18.77	18.82	18.82	18.82
18.82	18.84	18.93	18.93	18.95
18.96	18.97	18.98	18.99	18.99
19.00	19.01	19.06	19.07	19.07
19.12	19.17	19.19	19.34	19.35 22.90
19.42	19.44	19.46	21.98	22.30
23.00				
Danier dans Mai	ictum	Expected Value: 18.42	Std. Dev.: 3.	12
Parameter: Mo	5000	Mean: 17.90	Coef. Var.:	L7.43
Sample Number: Analysis: 2	3000	Median: 18.57	Skewness: -4.	. 23
Number of OBS:	80	Variance: 9.73	Accuracy: .8	
			-	
		- DATA IN ASCENDING ORDE	ER	
			11.42	15.78
. 85	. 85	10.84	17.29	17.37
16.60	16.90	17.24 17.76	17.80	17.93
17.47	17.72	17.76	17.99	18.05
17.93	17.95 18.10	18.15	18.15	18.19
18.09	18.21	18.23	18.23	18.25
18.20 18.26	18.28	18.30	18.31	18.32
18.42	18.44	18.48	18.51	18.53
18.61	18.62	18.63	18.63	18.64
18.66	18.66	18.67	18.67	18.68
18.68	18.68	18.70	18.73	18.73
18.78	18.79	18.85	18.85	18.89
18.91	18.91	18.92	18.95	18.96
18.97	18.97	18.98	18.98	18.98
19.00	19.05	19.07	19.19	19.20
19.24	19.29	19.41	22.21	23.15

Parameter: Ash Sample Number: 2000 Analysis: 1 Number of OBS: 98	Expected Value: 22.14 Mean: 22.65 Median: 22.08 Variance: 32.26	Coef. Var.: 25.07 Skewness: 9.42	
~~~~~~~~	DATA IN ASCENDING ORDER -		-
20.92       21.18         21.57       21.58         21.72       21.72         21.85       21.86         21.92       21.92         21.96       21.96         22.00       22.00         22.02       22.02         22.04       22.05         22.07       22.07         22.09       22.11         22.11       22.12         22.13       22.14         22.14       22.16         22.17       22.17         22.18       22.19         22.20       22.20         22.23       22.24         22.28       22.29	21.41 21.62 21.73 21.87 21.92 21.97 22.01 22.03 22.06 22.08 22.11 22.12 22.14 22.16 22.17 22.20 22.21 22.20 22.21	21.53       21.5         21.66       21.6         21.76       21.8         21.90       21.9         21.93       21.9         21.97       21.9         22.02       22.0         22.03       22.0         22.07       22.0         22.08       22.0         22.11       22.1         22.12       22.1         22.16       22.1         22.18       22.1         22.20       22.2         22.21       22.2         22.22       22.2         22.36       22.4	83048237912468038
22.64 28.00  Parameter: Ash Sample Number: 2000	77.94  Expected Value: 22.14  Mean: 22.73	Std. Dev.: 6.22 Coef. Var.: 27.34	
Analysis: 2 Number of OBS: 81	Median: 22.09 Variance: 38.64	Skewness: 8.65 Accuracy:23	
	DATA IN ASCENDING ORDER		-
21. 24       21. 49         21. 60       21. 66         21. 78       21. 79         21. 92       21. 93         21. 96       21. 97         21. 98       21. 99         22. 01       22. 01         22. 03       22. 04         22. 09       22. 09         22. 11       22. 12         22. 13       22. 14         22. 21       22. 22         22. 23       22. 23         22. 24       22. 26         22. 35       22. 38	21.51 21.68 21.87 21.94 21.97 21.99 22.02 22.05 22.10 22.12 22.14 22.19 22.22 22.24 22.28 22.40	21.51       21.5         21.74       21.7         21.87       21.9         21.95       21.9         21.97       21.9         22.00       22.0         22.02       22.0         22.06       22.0         22.11       22.1         22.12       22.1         22.20       22.2         22.23       22.2         22.24       22.2         22.43       22.8	75 90 95 98 98 90 93 98 91 12 12 12 12 13 12 13 13 14 14 15 16 16 17 17 18 18 18 18 18 18 18 18 18 18 18 18 18

Parameter: Ash Sample Number: Analysis: 1 Number of OBS:	5000	Expected Value: 4 Mean: 5.73 Median: 4.70 Variance: 88.38		Std. Dev.: 9.40 Coef. Var.: 164.04 Skewness: 8.78 Accuracy: -1.67	1
		DATA IN ASCENDING	ORDER		
2.70 3.84 3.92 4.05 4.40 4.49 4.50 4.58 4.65 4.66 4.70 4.73 4.76 4.78 4.86 4.94 5.05 5.16	2.76 3.86 3.93 4.14 4.41 4.49 4.52 4.60 4.65 4.67 4.71 4.73 4.77 4.79 4.84 4.86 4.96 5.08 5.22	3.45 3.87 3.93 4.22 4.42 4.50 4.55 4.61 4.65 4.69 4.71 4.74 4.77 4.79 4.85 4.87 4.98 5.12 5.23		3.72 3.87 3.97 4.24 4.45 4.50 4.55 4.61 4.65 4.70 4.72 4.75 4.77 4.82 4.85 4.90 5.00 5.15 5.35	3.88 3.98 4.38 4.48 4.50 4.57 4.65 4.66 4.70 4.72 4.74 4.83 4.86 4.91 5.00 5.16 5.35
27.40	94.42				3.33
Parameter: Ash Sample Number: Analysis: 2 Number of OBS:	5000	Expected Value: 4 Mean: 5.67 Median: 4.69 Variance: 100.07		Std. Dev.: 10.00 Coef. Var.: 176.4 Skewness: 8.64 Accuracy: -1.86	l
	-4	DATA IN ASCENDING	ORDER	******	
2.65 3.80 3.97 4.33 4.45 4.52 4.61 4.65 4.69 4.72 4.75 4.80 4.82 4.87 4.99 5.07	2.80 3.92 3.98 4.38 4.46 4.53 4.62 4.66 4.69 4.73 4.77 4.81 4.82 4.89 4.99 5.08	3.55 3.94 4.07 4.39 4.47 4.54 4.65 4.67 4.69 4.73 4.78 4.81 4.82 4.91 5.02 5.09		3.60 3.94 4.18 4.40 4.47 4.56 4.65 4.67 4.70 4.73 4.78 4.81 4.83 4.91 5.03 5.38	3.77 3.96 4.19 4.44 4.51 4.60 4.65 4.67 4.71 4.74 4.79 4.81 4.86 5.04 5.52

Parameter: Gro Sample Number:	ss Cal 2000	Expected Value: Mean: 1139.74	Coef. Var.	: 6.65
Analysis: 1	0.3	Median: 11269.0		
Number of OBS:	93	Variance: 54937	7.38 Accuracy:	<b>-</b> .62
***********		DATA IN ASCENDIN	G ORDER	
6048.00	8273.00	9653.00	10040.00	10287.00
10500.00	10587.00	10607.00	10957.00	11012.00
11027.00	11052.00	11052.00	11059.00	11085.00
11101.00	11105.00	11113.00	11120.00	11131.00
11133.00	11140.00	11143.00	11176.00	11181.00
11183.00	11206.00	11208.00 11229.00	11209.00 11232.00	11215.00 11233.00
11219.00 11238.00	11220.00 11248.00	11250.00	11255.00	11256.00
11256.00	11256.00	11250.00	11261.00	11263.00
11269.00	11269.00	11270.00	11270.00	11271.00
11272.00	11272.00	11274.00	11276.00	11278.00
11278.00	11279.00	11281.00	11281.00	11282.00
11282.00	11284.00	11285.00	11286.00	11290.00
11296.00	11297.00	11301.00	11302.00	11303.00
11303.00	11305.00	11307.00	11312.00	11315.00
11315.00	11321.00	11324.00	11326.00	11326.00
11330.00	11334.00	11352.00	11369.00	11380.00
11380.00	11381.00	11435.00	11484.00	11500.00
<b>1</b> 1674.00	11799.00	14098.00		
110/1.00	11/33.00	14030.00		
			11339.00 Std. Dev.:	731.14
Parameter: Gro	ss Cal	Expected Value: Mean: 1108.84	Coef. Var.	: 6.58
	ss Cal 2000	Expected Value: Mean: 1108.84 Median: 11265.0	Coef. Var. O Skewness:	: 6.58 -5.48
Parameter: Gro Sample Number:	ss Cal 2000	Expected Value: Mean: 1108.84	Coef. Var. O Skewness:	: 6.58 -5.48
Parameter: Gro Sample Number: Analysis: 2	ess Cal 2000 77	Expected Value: Mean: 1108.84 Median: 11265.0	Coef. Var. Skewness: Accuracy:	: 6.58 -5.48
Parameter: Gro Sample Number: Analysis: 2	ess Cal 2000 77	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456	Coef. Var. 0 Skewness: 1.68 Accuracy: G ORDER	: 6.58 -5.48 65
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456 DATA IN ASCENDIN 9702.00 11047.00	Coef. Var.  Skewness: Accuracy:  G ORDER  10631.00 10050.00	: 6.58 -5.48 65 
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456 DATA IN ASCENDIN 9702.00 11047.00 11147.00	Coef. Var.  Skewness: Accuracy:  G ORDER  10631.00 10050.00 11148.00	: 6.58 -5.48 65 0700.00 11074.00 11148.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	77 7928.00 10927.00 11130.00 11159.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456 DATA IN ASCENDIN 9702.00 11047.00 11147.00 11176.00	Coef. Var.  Skewness: Accuracy:  G ORDER  10631.00 10050.00 11148.00 11187.00	10700.00 11074.00 11201.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 111222.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456 DATA IN ASCENDIN  9702.00 11047.00 11176.00 11227.00	Coef. Var.  Coef. Var.  Skewness: Accuracy:  GORDER  10631.00 10050.00 11148.00 11187.00 11235.00	10700.00 11074.00 11201.00 11242.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456 DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00	Coef. Var.  Coef. Var.  Skewness: Accuracy:  GORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00	10700.00 11074.00 11148.00 11242.00 11255.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 111222.00 11257.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00	Coef. Var.  Skewness:  Accuracy:  G ORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00 11261.00	10700.00 11074.00 11148.00 11201.00 11242.00 11255.00 11262.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00 11257.00 11263.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00 11263.00	Coef. Var.  Skewness:  Accuracy:  G ORDER  10631.00 10050.00 11148.00 11187.00 11249.00 11249.00 11261.00 11265.00	: 6.58 -5.48 65 10700.00 11074.00 11148.00 11201.00 11242.00 11255.00 11262.00 11265.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00 11257.00 11263.00 11269.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00 11263.00 11271.00	Coef. Var.  0 Skewness: 1.68 Accuracy:  G ORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00 11261.00 11265.00 11272.00	10700.00 11074.00 11201.00 11242.00 11262.00 11265.00 11274.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00 11257.00 11263.00 11269.00 11275.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11176.00 11271.00 11259.00 11263.00 11271.00 11276.00	Coef. Var.  Skewness: 1.68 Accuracy:  G ORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00 11261.00 11265.00 11272.00 11276.00	10700.00 11074.00 11148.00 11242.00 11262.00 11265.00 11274.00 11276.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00 11263.00 11269.00 11275.00 11279.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00 11263.00 11271.00 11276.00 11280.00	Coef. Var.  Coef. Var.  Skewness: Accuracy:  GORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00 11261.00 11272.00 11276.00 11281.00	10700.00 11074.00 11201.00 11242.00 11262.00 11265.00 11274.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00 11257.00 11263.00 11269.00 11275.00 11279.00 11286.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00 11263.00 11271.00 11276.00 11280.00 11288.00	Coef. Var.  Coef. Var.  Skewness: Accuracy:  GORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00 11261.00 11265.00 11272.00 11276.00 11281.00 11290.00	10700.00 11074.00 11148.00 11201.00 11242.00 11255.00 11262.00 11274.00 11276.00 11282.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00 11257.00 11263.00 11275.00 11279.00 11286.00 11302.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00 11263.00 11271.00 11276.00 11280.00	Coef. Var.  Coef. Var.  Skewness: Accuracy:  GORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00 11261.00 11272.00 11276.00 11281.00	: 6.58 -5.48 65 10700.00 11074.00 11148.00 11201.00 11242.00 11255.00 11262.00 11274.00 11276.00 11282.00 11292.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 11222.00 11245.00 11257.00 11263.00 11269.00 11275.00 11279.00 11286.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00 11263.00 11271.00 11276.00 11280.00 11280.00 11305.00	Coef. Var.  Note that the content of	10700.00 11074.00 11148.00 11201.00 11242.00 11255.00 11262.00 11274.00 11276.00 11282.00 11292.00 11315.00
Parameter: Gro Sample Number: Analysis: 2 Number of OBS: 	7928.00 10927.00 11130.00 11159.00 1122.00 11245.00 11257.00 11263.00 11269.00 11275.00 11279.00 11286.00 11302.00 11330.00	Expected Value: Mean: 1108.84 Median: 11265.0 Variance: 53456  DATA IN ASCENDIN  9702.00 11047.00 11147.00 11176.00 11227.00 11248.00 11259.00 11263.00 11271.00 11276.00 11280.00 11280.00 11305.00 11334.00	Coef. Var.  0 Skewness: 1.68 Accuracy:  G ORDER  10631.00 10050.00 11148.00 11187.00 11235.00 11249.00 11261.00 11265.00 11272.00 11276.00 11290.00 11305.00 11340.00	: 6.58 -5.48 65 10700.00 11074.00 11148.00 11201.00 11242.00 11255.00 11262.00 11265.00 11274.00 11276.00 11282.00 11292.00 11315.00 11346.00

Parameter: Gro Sample Number: Analysis: 1	5000	Expected Value: 1 Mean: 11683.64 Median: 11981.50		Dev.: 870.94 . Var.: 7.45 ness: -3.23
Number of OBS:	92	Variance: 758529.	40 Accu	racy:85
••••••		DATA IN ASCENDING	ORDER	
<b>6</b> 662.00	8701.00	9519.00	9664.00	
9778.00	9825.00	9973.00		10913.00
11112.00	11200.00	11314.00	_	11471.00
11515.00	11516.00	11562.00	11573.00	
11720.00	11757.00	11770.00	11825.00	
11846.00	11880.00	11891.00	11901.00 11938.00	
11925.00	11929.00 11954.00	11933.00 11954.00	11957.00	
11946.00 11969.00	11969.00	11972.00	11977.00	
11981.00	11982.00	11994.00	11997.00	
12003.00	12009.00	12012.00	12014.00	
12022.00	12024.00	12025.00	12026.00	
12031.00	12033.00	12034.00	12037.00	
12042.00	12042.00	12043.00	12049.00	12051.00
12059.00	12060.00	12065.00	12069.00	
12073.00	12075.00	12079.00	12082.00	
12086.00	12096.00	12097.00	12115.00	
12117.00	12127.00	12190.00	12295.00	12319.00
12379.00	12532.00			
Parameter: Gre		Expected Value: 1		Dev.: 904.07
Sample Number:		Mean: 11684.69		7. Var.: 7.74
Analysis: 2		Median: 11986.69		ness: -3.38
Number of OBS:		Variance: 817340.		racy:81
		DATA IN ASCENDING	ORDER	
6542.00	8763.00	9547.00	9727.00	9842.00
9855.00	10247.00	10574.00	10877.00 11567.00	11159.00 11604.00
11429.00	11515.00	11562.00 11811.00	11817.00	11825.00
11683.00 11889.00	11766.00 11899.00	11911.00	11902.00	11911.00
11924.00	11930.00	11944.00	11954.00	11971.00
11972.00	11973.00	11973.00	11975.00	11977.00
11980.00	11980.00	11980.00	11986.00	11994.00
11997.00	12001.00	12001.00	12003.00	12013.00
12018.00	12019.00	12032.00	12034.00	12034.00
12034.00	12037.00	12038.00	12039.00	12042.00
12043.00	12044.00	12047.00	12057.00	12060.00
12063.00	12068.00	12068.00	12070.00	12073.00
12074.00	12074.00	12083.00	12090.00	<b>12096.00</b>
12115.00			4 4 4 4 4 4 4 4	10005 00
12370.00	12119.00 12404.00	12123.00	12187.00	12325.00