ACCURACY CRITERIA RECOMMENDED

FOR THE CERTIFICATION

OF GRAVIMETRIC COAL MINE DUST SAMPLERS

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ABSTRACT

Procedures are recommended for testing the bias and precision of gravimetric Coal Mine Dust Personal Sampling Units (CMDPSUs). The CMDPSU bias relative to any definition for respirable dust can be calculated from the unit's collection efficiency measured with a fluorescein-tagged monodisperse aerosol. The CMDPSU precision is measured by taking replicate samples in a coal dust chamber. Based on this type of test, sampling unit accuracy can be compared to the National Institute for Occupational Safety and Health (NIOSH) criterion of ±25 percent at the 95 percent confidence level, currently used to validate other sampling and analytical methods.

Recommendations are presented for developing a CMDPSU certification program based on the NIOSH accuracy criteria and the newly developed bias and precision tests. In order to evaluate the proposed certification tests, the bias and precision tests were performed on 10 mm cyclone sampling units operated according to the Federal coal mine dust sampling procedures. Such a certification program based on performance criteria could allow the introduction of improved gravimetric samplers and pumps excluded by the current regulations (30 CFR 74).

TABLE OF CONTENTS

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GLOSSARY OF ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
ANOVA	Analysis of Variance
APS	Aerodynamic Particle Sizer
BMRC	British Medical Research Council
CFR	Code of Federal Regulations
CMDPSU	Coal Mine Dust Personal Sampling Unit
DPSE	Division of Physical Sciences and Engineering (NIOSH)
DSR	Division of Safety Research (NIOSH)
EMS	Expected mean square term from an analysis of variance
EPA	Environmental Protection Agency
EXP	Experiment effects in the precision experiment
GSD	Geometric Standard Deviation
JCL	Job Control Language
KHP	Potassium Hydrogen Phthalate
LNC	Natural Log Transform of the (Dust) Concentration
MMD	Mass Median Diameter
MRE	Mining Research Establishment horizontal elutriator
MS	Mean square value from an analysis of variance
MSA	Mine Safety Appliances Company
MSHA	Mine Safety and Health Administration
NAS	National Academy of Sciences

NBS	National Bureau of Standards
NIOSH	National Institute for Occupational Safety and Health
NMD	Number Median Diameter
PCC	Parklawn Computer Center
POS	Position effects in the precision experiment
PFR	Pneumoconiosis Field Research
QC	Quality Control
RAM	Respirable Aerosol Monitor
RESID	Residuals
S	Sampler effects in the precision experiment
SAS	Statistical Analysis System
SCP	Standards Completion Program (NIOSH)
SEM	Scanning Electron Microscope
SIC	Standard Instrument Calibration chamber (NIOSH)
SS	Sum of Squares
T	Target concentration in the precision experiment
TSI	Thermo-Systems Incorporated
TSO	Time-Sharing Option to the IBM 370 computer system
TRANS	Translation
VAR	Variance

GLOSSARY OF KEY SYMBOLS

C_R	=	true concentration of respirable dust (R = BMRC or ACGIH)
C_S	=	sampled concentration of respirable dust
CV_A	=	coefficient of variation (relative standard deviation) from analysis (i.e., weighing)
CV_S	=	coefficient of variation (r.s.d.) from sampling
CV_t	=	total coefficient of variation (r.s.d.) in a single measurement
D	=	particle's aerodynamic diameter
D_{cut}		fifty percent cut diameter in the collection and sampling efficiency curves
dM/dD	=	aerosol size distribution (mass weighted)
dN/dD	=	aerosol size distribution (number weighted)
k	=	calibration coefficient
M_I	=	initial mass of filter
M_F	=	final mass of filter (after dust sampling)
M_S	=	mass of sampled dust
Q	=	sampler flow rate
s_M	=	standard deviation in the dust mass measurement
T	=	sampling time
Δ	=	relative bias in respirable dust concentration
η	=	collection efficiency of the sampler for non-respirable particles
θ_R	=	defined sampling efficiency for respirable dust (R = BMRC or ACGIH)

θ_s = calibrated sampling efficiency of the sampler for respirable dust

σ_ϵ = standard deviation in the log-transformed concentration due to intra-sampler variability

σ_s = standard deviation in log-transformed concentrations due to inter-sampler variability

σ_t = total standard deviation in the log-transformed concentration

I. INTRODUCTION

In this report, procedures are described for testing the accuracy of gravimetric Coal Mine Dust Personal Sampling Units (CMDPSU). The results of these accuracy tests could be used by NIOSH as the basis for future revisions of the regulations (30 CFR 74) for the certification of CMDPSUs for use in sampling respirable coal mine dust as required by the Mine Safety and Health Administration (MSHA).

CERTIFICATION PROCEDURES

The Federal certification of CMDPSUs was established by law over ten years ago. In the Federal Coal Mine Safety and Health Act of 1969, the Dust Standard begins:

> "Each operator of a coal mine shall take accurate samples of the amount of respirable dust in the mine atmosphere to which each miner in the active workings of such mine is exposed. Such samples shall be taken by any device approved by the Secretary [of the Interior, now the Secretary of Labor] and the Secretary of Health, Education and Welfare [now Health and Human Services]..."
>
> Section 202(a)

To approve these sampling devices, the two federal departments set up on March 11, 1970 the CMDPSU certification program, described in Part 74 of the Code of Federal Regulations, Title 30. In Section 74.2 of these regulations, the CMDPSU is defined as "(a) a pump unit, (b) a sampling head assembly, and (c) if rechargeable batteries are used in the pump unit, a battery charger."

In Section 74.3, the design and performance of the CMDPSU is specified, based on the Dorr-Oliver 10mm nylon cyclone, which the Bureau of Mines had been developing for the sampling of respirable coal mine dust.

Testing CMDPSUs for adherence to these specifications in Section 74.3 is the responsibility of NIOSH (Section 74.4(a)). MSHA must also test the CMDPSU for intrinsic safety in methane atmospheres (Section 74.4(b)). After passing the certification tests and meeting the other requirements in 30 CFR 74, a CMDPSU is certified for taking the coal mine dust samples required by Federal regulations.

Within NIOSH, the certification of CMDPSUs is conducted by the Air Sampling and Instrumentation Section, Division of Safety Research (DSR). For this task, tests were developed by McCawley and Roder (1975, 1976). The present certification procedures are based on tests for:

* pump pulsation effects on sample accuracy, 30 CFR 74.3(a)(8)(ii);
* filter resistance, 30 CFR 74.3(b)(2)(2);
* rotameter calibration, 30 CFR 74.3(a)(11) and (12);
* pump flow against resistance, 30 CFR 74.3(a)(14);

* filter capsule preweight, 30 CFR 74.3(b)(2)(ii);

* flow rate consistency, 30 CFR 74.3(a)(13);

* battery charge rundown, 30 CFR 74.3(c)(4);

* pulsation frequency, 30 CFR 74.3(a)(8)(i).

The CMDPSU tests have been the basis for 25 certification and de-certification actions by NIOSH since 1971. (See Table I-1). At the present time, the Mine Safety Appliances Co. (MSA) has one active certified CMDPSU and the Bendix Corporation has 2 active certified units. Six other units are certified but seldom used.

PERFORMANCE CRITERIA FOR CERTIFICATION

The certified CMDPSUs have been the tool for taking millions of dust samples in coal mines from 1971 up to the present. However, the 1971 certification regulations (30 CFR 74) do not allow the use of new dust sampling equipment which is now availible. For example, constant flow pumps are not certified by 30 CFR 74, even though their use in coal mines would eliminate the need to adjust the pumps during a shift. Neither can new designs for the sampler such as impactor size selectors (Treaftis, Tomb and Taylor, 1978 and Marple, 1978) or light scattering dust monitors be considered for certification under the present regulations.

According to a review of all NIOSH certification programs (Brief et al, 1980), the existing programs:

> ". . . . need to be replaced by a conceptually conceived system that places product performance as the sole responsibilty of the manufacturer. The responsibility of NIOSH in this system should be to develop basic performance criteria required for NIOSH certification and to assure adherence of products in usage to these criteria."

Performance criteria for measurement instruments are limits on the device's operating characteristics (such as a limit on inaccuracy), without any reference to the design used to achieve this performance. In developing a new certification system, DPSE has had two models of how performance criteria can be used in approving environmental measurement methods. The first example is the NIOSH procedures for the validation of sampling and analytical methods for workplace toxic substances (Gunderson and Anderson, 1980). The second example is EPA's regulations for "Ambient Air Monitoring, Equivalent and Reference Methods" (40 CFR 53, see also Hauser and Shearer, 1975).

The NIOSH validation procedures are applied to laboratory analytical methods used with industrial hygiene samples. The test applies to the laboratory method, but does not legally restrict the use of commercially available sampling or analytical equipment. In this sense, the NIOSH validation procedures are different from the situation addressed by CMDPSU certification.

Table I-1

History of CMDPSU Certifications

Certification Date	Manufacturer	Model	Current Status
1971	MSA	Model G	Voided 6/15/73
1971	Bendix	Unico	Voided 6/15/73
1971	Bendix	Unico/Koehler	Voided 6/15/73
1971	MSA	G/lamp battery	Voided 6/15/73
1971	Bendix	C-110	Voided 6/15/73
1971	Coal Technology	Universal Head	Revoked before 5/72
1971	Willson	BC	Voided 6/15/73
7/73	Bendix	3900-10 Micronair II	Revoked 4/17/75
7/73	NIOSH/MSA	Model G with Cincinnati damper	Never active in coal mines
7/18/73	Willson	BC	Few sold, not produced now
8/10/73	MSA	Model G	Active
6/28/74	Bendix	Micronair II w/Koehler Connector	Revoked 4/17/75
9/17/74	Bendix	C-115	Revoked 4/17/75
4/16/75	Bendix	Micronair II	Revoked 1/12/77
4/16/75	Bendix	C-115	Revoked 1/12/77
4/16/75	Bendix	Micronair II w/Koehler Connector	Revoked 1/12/77
7/7/75	Bendix	30/31	Revoked 1/12/77
6/23/76	Bendix	30/31	Revoked 1/12/77

Table I-1 (continued)

Certification Date	Manufacturer	Model	Current Status
8/18/76	RAC	2392-PS	Stop Sale 5/28/77, few sold.
5/20/77	Bendix	C-115	Not produced now
5/20/77	Bendix	Micronair II	Active (Koehler conn. also)
5/20/77	Bendix	30/31	Active
4/23/78	Bendix	30/31 (tamper-proof)	Not produced now
5/20/77	Bendix	44	Probably not active in coal mines
3/29/79	MSA	Model G (tamper-proof)	Not produced now

The NIOSH criterion for successful validation is that the results of a sampling and analytical method come within ±25 percent of the "true" value for the contaminant at the 95 percent confidence level. In order to test adherence to this criteria, the Standards Completion Program (SCP) conducted tests for analytical recovery, sampler capacity, storage stability, precision and bias. In the SCP protocol, the precision and bias are measured at 3 concentrations, 0.5, 1 and 2 times the OSHA standard. At each concentration, six samples are collected simultaneously in a test atmosphere. The estimated bias is calculated from the difference between the "true" concentration and the mean of the six test concentrations. In the validation criteria, the bias must be less than 10 percent of the "true" concentration at all 3 concentrations.

The precision is expressed as a coefficient of variation (CV), also called the "relative standard deviation", calculated for each set of 6 replicate samples. If the CV estimates are homogeneous at all 3 concentrations, they are pooled to give a common precision estimate with 15 degrees of freedom. Under the SCP protocol, the bias and precision estimates are then combined into a 95 percent confidence limit of the accuracy in a single sample. If the accuracy confidence limit is inside ±25 percent, the method is validated for use in industrial hygiene measurements.

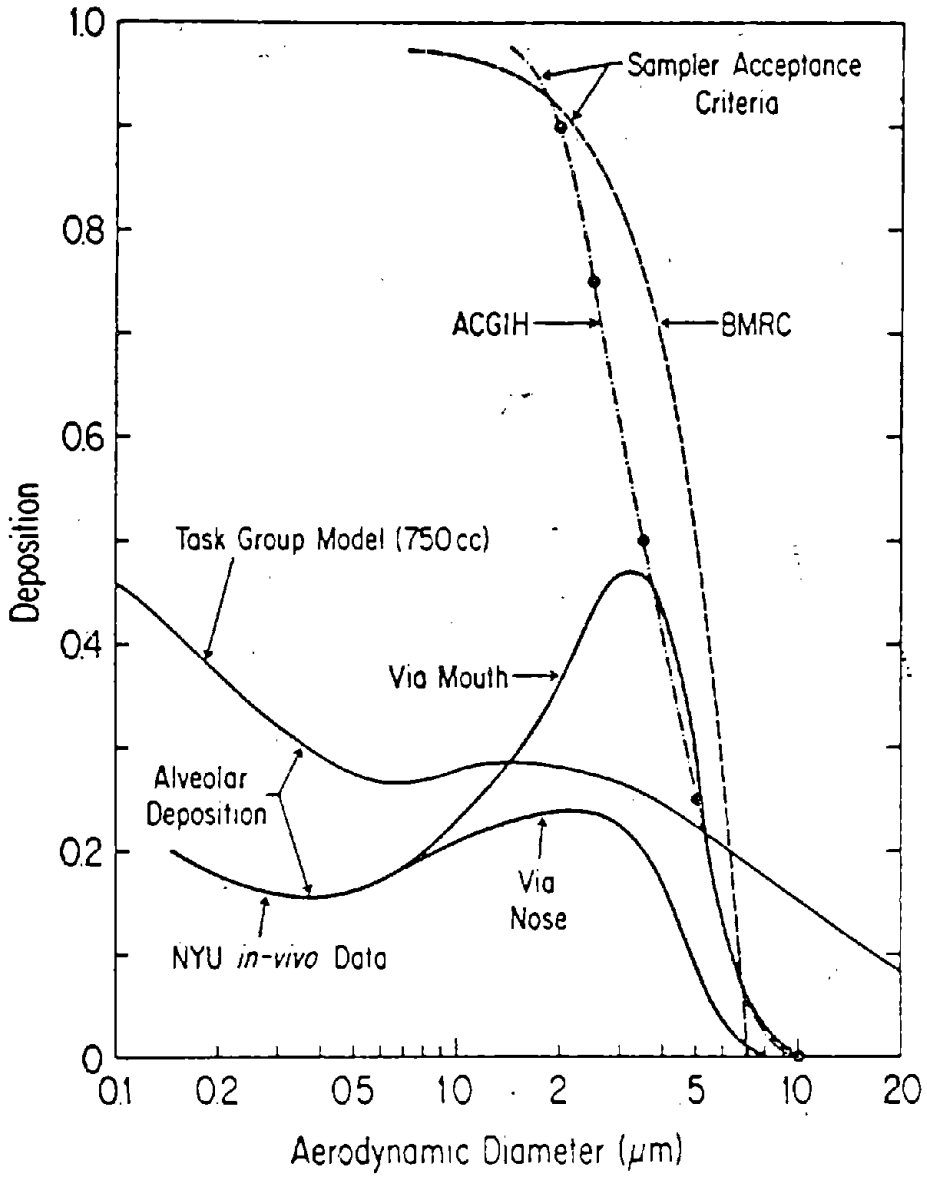
With methods of sampling respirable dust, the NIOSH validation protocol has one failing. In determining the bias, the "true" concentration of respirable dust cannot be directly measured, as the NIOSH protocol assumes. Respirable dust is defined rather by a penetration efficiency curve for the human lung as a function of particle size (see Figure I-1).

Alternative definitions of respirable dust have been created by the British Medical Research Council (BMRC), the American Conference of Governmental Industrial Hygenists (ACGIH), and other organizations around the world. In addition, the penetration curves of respirable dust samplers, such as the MRE horizontal elutriator and the 10mm Dorr-Oliver cyclone, can also be considered as references against which new samplers may be compared. All these definitions of respirable dust are based on the knowledge that only the dust fraction penetrating into the non-ciliated portions of the lower lung have the potential to cause coal workers pneumoconiosis (NAS, 1980).

To calculate the bias in respirable dust sampling, one of these respirable dust definitions must be selected as the reference against which new samplers are to be certified. In addition, the NIOSH validation protocol is based on the bias in the concentration, which depends on both the sampler's penetration curve and the size distribution of the dust being measured. To apply the NIOSH protocol to respirable dust samplers, a method is needed for determining the bias over the entire range of dust distributions found in coal mines. Both these problems must be solved in order to apply the NIOSH validation criteria to CMDPSUs.

Figure I-1: Human respirable deposition curves, compared to the definitions of the ACGIH, BMRC and ICRP Task Group Model.

SOURCE: Lippmann(1976).



EPA has been studying the development of bias criteria for particulate sampling, and also have extensive experience with performance criteria for privately manufactured sampling equipment. Their "equivalent method" system serves the same purpose for ambient air monitors as NIOSH's certification system does for CMDPSUs. The EPA system consists of the following parts:

1. Reference Methods (Appendices to 40 CFR 50). The reference methods have been thoroughly tested by EPA, and the procedures (including the brand names of equipment) are thoroughly specified in the Code of Federal Regulations. If the EPA approach were applied to CMDPSUs, the reference method would be the current Federal sampling procedures in 30 CFR 70 and 71, using the CMDPSUs specified in Part 74.

2. Candidate Method. The candidate method is submitted to EPA for accreditation as a method equivalent to the reference method. Under 40 CFR 53, the candidate method must be tested for equivalence by the manufacturer, and the test results submitted to EPA. The EPA Administrator may decide to have the tests repeated in a government designated laboratory.

   Unlike the present CMDPSU certification system, a candidate method may use new sampling procedures, as well as new equipment. Such freedom will be necessary in CMDPSU certification in order to allow the introduction of novel sampling devices for respirable coal mine dust.

3. Performance Testing (40 CFR 53, Parts B and C). EPA thoroughly specifies the tests which a candidate method must pass. The tests in Part B are for automated monitors, and thus are largely inapplicable to CMDPSUs, except for the precision measurement (which is done with 24 degrees of freedom). The tests in Part C are field tests comparing the candidate and reference methods, and can be considered bias tests with the "true" value defined as the result of the reference method.

4. Performance Criteria. EPA's performance specifications (Table B-1 and Table C-1, 40 CFR 53) are set essentially to be those which can be met by the reference method. These criteria could theoretically be lowered if more accurate methods are available, but this has not yet happened. The performance criteria differ for different substances. EPA has no generic accuracy standard, similar to the NIOSH criteria, which applies to all substances.

The EPA system is not as well developed for particle sampling as it is for gas sampling. The reason is that EPA's reference method for aerosols is the High-Volume Sampler, a simple device without clearly defined size-selection properties. However, EPA has developed a definition for "inhalable particles" in ambient air (Miller et al., 1979), and has organized an extensive effort to develop performance criteria for inhalable particle samplers.

One size selection criterion recommended for this new method (Ranade and Kashdan, 1981) assumes that the sampler penetration efficiency in Figure I-2 can be expressed as a cumulative log-normal function determined by its 50 percent cut diameter $(d_p)_g$ and its geometric standard deviation σ_g. The accuracy criteria are then simply given as allowable ranges for the parameters $(d_p)_g$ and σ_g. Other performance criteria are also proposed for flow rate stability, chemical artifact formation, errors due to wall losses and particle bounce, and size selection characteristics at different wind speeds. The "reproducibility" of the inhalable particle sampler would be tested, as with the other substances in 40 CFR 53, by comparison testing with a reference method.

The present certification testing of CMDPSUs also includes one test for the unit's accuracy, but the measurement is indirect. Lamonica and Treaftis (1972) had shown that insufficiently damped flow oscillations causes a shift in the size distribution and therefore the bias of the dust sample. Therefore, a new specification was added to the certification regulations:

> "...the quantity of respirable dust collected with a sampler unit shall be within ±5 percent of that collected with a sampling head operated with a non-pulsating flow."
>
> 30 CFR 74.3(a)(8)(88)

To test CMDPSUs for adherence to this pump pulsation criterion, McCawley and Roder (1975) devised a test where two CMDPSUs were run at 2.0 L/min in a coal dust chamber with two sampling heads with unpulsated flow regulated by critical orifices. To pass this test, 10 respirable dust samples taken with the CMDPSU at dust concentrations between 0.5-5.0 mg/m^3 must agree within ±5 percent at the 95 percent confidence level with concurrent samples taken with a non-pulsating flow source.

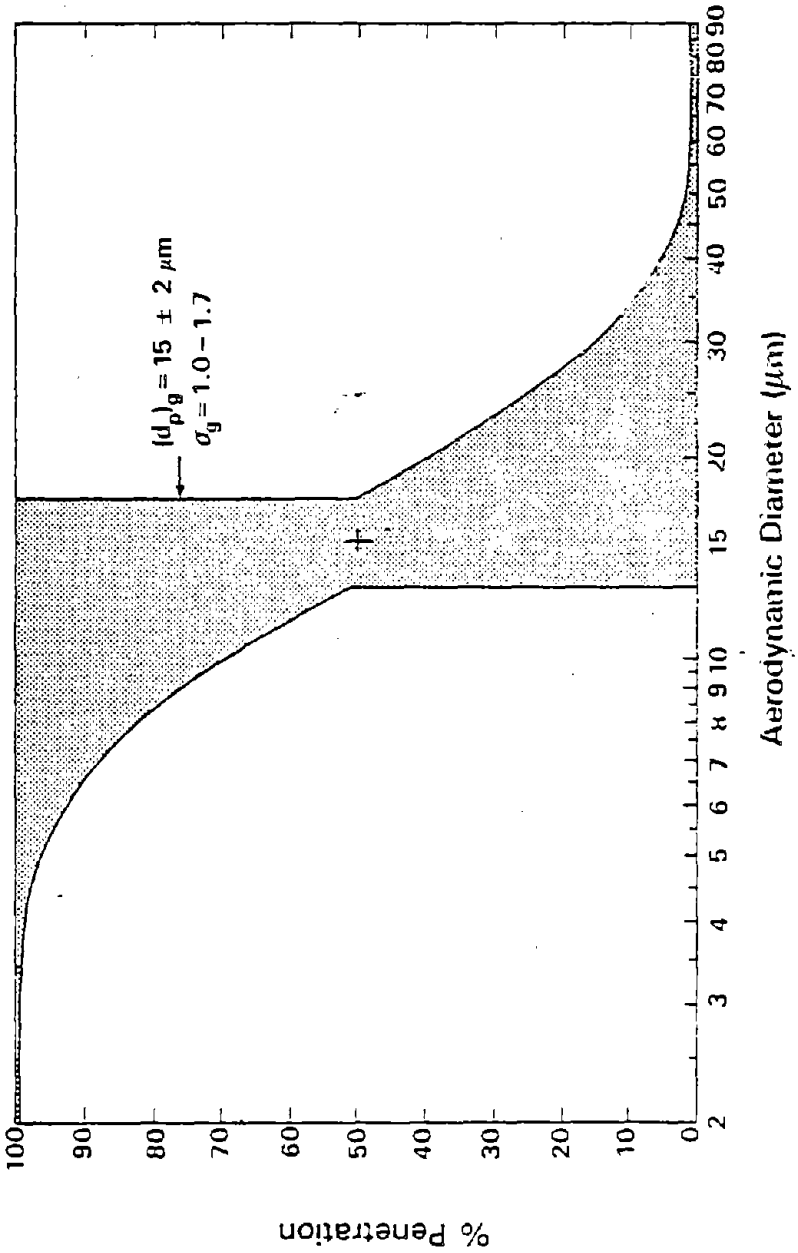
For this test, NIOSH originally used a "standard test coal dust" developed by Los Alamos Scientific Laboratory. Fairchild, Tillery and Ettinger (1977) developed this standard dust to have well-defined properties relative to the ACGIH definition for respirable dust (ACGIH, 1970), and they claim:

> "This standard dust permits evaluating the performance of coal mine air samplers with ±10 percent accuracy."

However, the theory behind this standard dust was never developed to the point where CMDPSU accuracy could be rigorously measured in terms of the standard

Figure I-2: EPA's proposed tolerance limits on the penetration efficiency function of "inhalable particle" samplers.

SOURCE: Renade and Kashdan (1981)



definitions for precision and bias. The statistical characteristics in DSR's pump pulsation test have not been rigorously analyzed either. Furthermore, DSR used up the Los Alamos "standard" dust, and is now using a dust prepared by MSHA. Therefore, the present CMDPSU certification tests contain no test for the absolute accuracy.

PROPOSED ACCURACY CRITERIA

The purpose of this report is to propose laboratory accuracy tests for CMDPSUs and criteria for deciding whether candidate units are accurate enough to be certified. Although the proposed tests were developed with the 10 mm nylon cyclone, they can be applied to any sampler design in which dust is distributed between a filter and a size selector. (These tests cannot be applied to the new light-scattering dust monitors because of the extra variables needed to relate light scattering properties to aerosol mass.) The final certification process will presumably require additional tests of the unit's safety, ruggedness, portability, and reliability.

The recommended accuracy criteria draw in part from other certification programs and from past research on size selective samplers (see the Bibliography), but contain some new elements as well, especially in the mathematical treatment of the test data. The proposed system is outlined in Figure I-3.

As shown in this flow diagram, CMDPSU accuracy is composed of the bias relative to a definition of respirable dust, and of the precision, expressed as a coefficient of variation from replicate samples. Using both bias and precision in expressing measurement accuracy is consistent with present statistical theories of accuracy (Eisenhart, 1963, 1968).

The bias test starts with measurements of the candidate sampler's penetration efficiency (described in Section III.B). The resulting penetration function is then converted into an estimate of the CMDPSU's bias in coal mines, using a computerized mathematical model (described in Section II, D-F).

This bias calculation requires the input of dust size distributions found in coal mines (reviewed in Section II.B) and a definition of respirable dust. Thus, the recommended accuracy criteria require the adoption of an explicit definition for respirable dust. In analyzing the test results (Section IV), the bias calculation uses the BMRC definition, but the proposed system will operate equally well with the ACGIH definition. The definition in this system could also be the measured penetration curve of a reference instrument such as the MRE horizontal elutriator or the 10mm Dorr-Oliver cyclone run at a single flow rate. However, this alternative would require some consideration of the experimental errors in measuring the penetration curve of the reference instrument.

Separately, the candidate CMDPSU is run through the precision test (described in Section III.A). In this test, the samplers are operated according to the manufacturer's directions, so any practical difficulties with the sampler's operation become apparent. In the trial runs of the precision tests, the

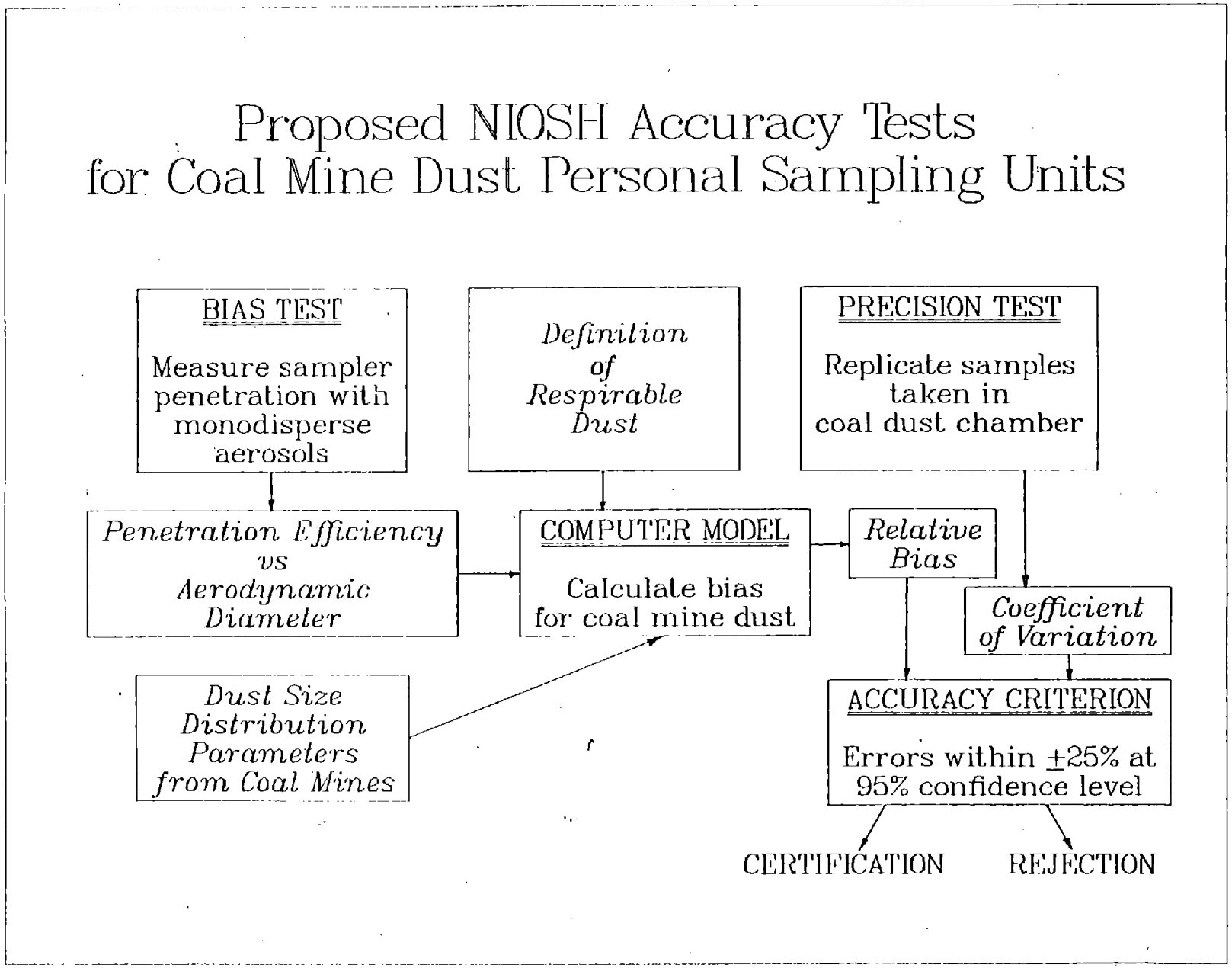


Figure I-3: Recommended procedure for testing the accuracy of Coal Mine Dust Personal Sampling Units.

CMDPSUs are run according to current Federal sampling procedures. Statistical analysis of these test results (Section II.C) provides estimates of the CV for a CMDPSU sample to approximately 15 degrees of the freedom, the standard used in NIOSH's validation program for sampling and analytical methods.

The final step is to combine the bias and precision estimates into an over-all estimate of the accuracy of a single CMDPSU sample at the 95 percent confidence level (described in Section II.G). This accuracy estimate can then be compared to the criterion set for certification. If the NIOSH accuracy criteria is adopted for CMDPSU certification, a candidate sampler must have an estimated accuracy within $\pm$25 percent at the 95 percent confidence level.

II. THEORY

A. INTRODUCTION

The basic philosophy behind this report is to address coal mine dust personal sampling techniques in reference to the present NIOSH criteria (Gunderson and Anderson, 1980) requiring that acceptable sampling/analytical techniques maintain less than 25 percent error at the 95 percent confidence level. This criterion was applied over a set of representative coal dust distributions. As described below, both systematic as well as random errors contribute significantly to the overall error and were evaluated.

In order to select a set of representative coal dusts, a review of size distribution measurements was carried out. Results of this review, together with a rudimentary evaluation of the different experimental procedures involved are presented below. Log-normal parameters are given for specific dust distributions for which the NIOSH accuracy criterion was applied.

Measurement of the random errors or precision of the sampling unit under examination is then discussed. An experimental design is presented for estimating variations in the CMDPSU performance, after removing run and position variations due to the test method. Standard quality control practices are suggested for maintaining a high level of accuracy within the test procedure itself.

Aside from random errors, the systematic error or bias was measured. In general, a sampler to be certified under this program will exhibit a particle size acceptance efficiency which differs from the applicable respirable dust definition (ACGIH or BMRC). Therefore, for almost every specific dust distribution there will exist bias or systematic error between sampler-estimated and true respirable dust concentration. Furthermore, as the coal mine dust size distributions, which will not be measured in detail, are expected to vary from mine to mine, workplace to workplace and day to day, the bias is an unavoidable, unpredictable source of error.

Therefore, details are given as to how the bias and its 95 percent confidence level value are computed for any specific dust size distribution, given collection efficiency data for the sampling unit. Calculation of the sampler's bias alone involves straightforward numerical integration of interpolated collection efficiency data superimposed onto the size distribution of the particular coal mine dust size distribution of interest. However, estimation of the experimental uncertainty in the certification laboratory's bias test is not so simple, because replicate measurements of the collection efficiency are not feasible with present technology. Secondly, unlike the 10 mm cyclone, little may be known about the collection efficiency of the sampler undergoing certification testing. Thus, no detailed mathematical model curve about which to measure the scatter of experimental points is available.

Thus, in order to estimate the bias uncertainty for a general (i.e., not necessarily the 10 mm cyclone) sampling unit, further assumptions are necessary. Firstly, it is assumed that, as with the 10 mm cyclone, uncertainty as to the collection efficiency curve's exact positioning along the particle diameter axis dominates the bias uncertainty. Secondly, this translational uncertainty itself is estimated from certification laboratory measurements of cyclone characteristics. Within these assumptions it is a simple matter to compute the propagation of error into the bias for the general sampler.

B. COAL MINE DUST SIZE DISTRIBUTIONS

SCOPE

The size distribution of the sampled dust is needed for the calculation of the bias in respirable dust samplers. In order to estimate this bias for Coal Mine Dust Personal Sampling Units, the literature on dust size distributions in coal mines is reviewed.

The scope of this literature review is therefore limited to those studies whose results are applicable to respirable dust sampling in coal mines. Size distributions measured in the laboratory or in workplaces other than coal mines are thus excluded. The size distributions of bulk coal samples which have been crushed are also irrelevant to this application.

Also excluded are the size distributions for samples taken with a respirable dust size selector (e.g., a horizontal elutriator). For use in the bias calculations, the size distribution must represent the ambient dust entering the sampler. Although ambient distributions can theoretically be reconstructed from a knowledge of the size selector's collection efficiency, this mathematical operation results in great uncertainty in the distribution parameters, especially when substantial portions of the ambient dust are larger than respirable. For this reason, this review is limited to measurements of the "total" dust size distribution, taken with samplers which have 100 percent penetration efficiencies for dust in the respirable size range ($\leq$10 μm).

On this basis, sixteen studies of the size distribution in coal mines have been selected (Table II-1). These studies were done in six different countries, extending back to 1960. Since the pre-1960 studies report parameters which are not generally consistent with the more modern measurements, citations before 1960 are not included.

Willeke, Whitby, Marple and Liu performed a review of coal dust size distributions with similar scope (1970). Where the studies reviewed by Willeke *et al.* were performed after 1960, we have included them in Table II-1.

In order to compare the diverse results of these studies, the data were fitted to a mass-weighted log-normal distribution dM/dD as a function of the aerodynamic diameter D. Within the log-normal

TABLE II-1: Studies of Coal Mine Dust Size Distributions

Study	Nation	Type of Mining	Sampler Location	Sampling Method	Sizing Method	Data Report
Tomb, Treaftis and Gero (1983)	USA	Continuous	Various	Total dust filter	Coulter counter	Frequency data
Burkhart, McCawley, and Wheeler (1983)	USA	Continuous, longwall	Various	Sierra 260 and 294 impactors with greased glass or stainless steel slides		MMD and GSD for 2 modes
Vitek (1977)	Czecho-slovakia	Longwall	10 m outby face, "skip bunker"	Filter in metal casing	Optical Microscope	Probit plot
White and DeNee (1972)	USA	Bituminous coal	NR	"Personal sampling filter"	Fluid redispersion and scanning electron microscope	Probit plot
Dodgson et al (1971)	UK	Various longwall mine operations	Random collier method*	Standard Thermal Percipitator	Optical microscope and Coulter Counter	β parameters
Leiteritz, Bauer and Bruckmann (1971)	FRG	Longwall, all types of coal	10 - 20 m outby tailgate	GOTHE filter	Optical Microscope	Probit plots
Landwehr and Bruckmann (1969)	FRG	NR	NR	"Siter" total dust sampler	Andreasen sedimentation	Frequency data
Breuer (1967)	FRG	Longwall and continuous mining	Return gateroad 10 - 20 m from junction	GOTHE filter	Andreasen sedimentation	Frequency plot

*: Random Collier Method - Sampler carried by investigator who remained close to a randomly selected miner from beginning to end of shift.

Table II-1 continued

Study	Nation	Type of Mining	Sampler Location	Sampling Method	Sizing Method	Data Report
Leiteritz, Einbrodt and Klosterkotter (1967)	FRG	Anthracite to "long-flaming gas coal"	NR	NR	Sedimentation	Frequency plot
Poieluiv et al (1967)	USSR	Uses different wetting methods	Near coal face	Filter	Optical microscope	Probit plot
Rogan, Rae and Walton (1967)	UK	Longwall face	Random collier method*	Standard Thermal Precipitator	Optical microscope	β parameters
Baier and Diakun (1963, 1964)	USA	Conventional and continuous, anthracite and bituminous	Breathing zone of miner at face	Membrane filter sampler	Optical microscope	Frequency data --NMD and GSD averages--NM range.
Breuer (1961)	FRG	NR	NR	Filter	Andreasen sedimentation	Frequency plot
Cartwright and Skidmore (1961)	UK	Conventional mining, anthracite and bituminous.	Intake and return air, near face when blasting	Thermal Precipirator	Optical and electron microscopes	Frequency plot
Faye and Ashford (1960)	UK	Longwall, heading and surface operations	Random collier method*	Standard Thermal Precipitator	Optical microscope	β parameters

* Random Collier Method - Sampler carried by investigator who remained close to a randomly selected miner from beginning to end of shift.

distribution, dust of total mass M contained in a given volume of air is distributed according to:

$$dM/dD = \frac{M}{\sqrt{2\pi}\, D \ln GSD} \exp\left\{ - \frac{\ln^2(D / MMD)}{2 \ln^2 GSD} \right\} \qquad \text{(II-1)}$$

where MMD = mass median diameter
GSD = geometric standard deviation.

With this model, the results of the size distribution measurement are summarized by the two parameters, MMD and GSD. Not only are these parameters used to compare size distribution measurements, but the log-normal model is conveniently used in the bias calculation.

METHODS FOR DETERMINING SIZE DISTRIBUTIONS

(1) Types of Coal Mine Dust

Coal mine dust consists of coal mixed with quartz, clay and other minerals (sometimes just called "dirt"). The dust is generated by a wide variety of mechanical processes such as shearing, drilling, blasting, etc. After the dust is suspended in the air, the particles settle out at a rate which depends on the particle size. All these factors affect the size distribution.

In order for a measurement of the size distribution to be useful in the CMDPSU bias calculation, the measurement should be taken in conditions similar to those under which the occupational health samples are taken. For the purposes of the present review, the ideal study would consist of measurements from all major types of coal mining processes, both underground and surface, found in the U.S. The samplers would be located as prescribed in the Federal dust sampling regulations.

This ideal sampling is seldom found in the literature on coal dust sizing. Most of the MSHA-required samples are personal or breathing zone samples, while most size distribution measurements in Table II-1 are made on area samples.

In the one major exception to area samples, the Pneumoconiosis Field Research study in Great Britain (Hurley et al., 1979) does include extensive breathing zone samples taken by investigators who followed randomly selected miners throughout a shift ("the Random Collier Method"). The British mines, however, use several techniques not

found in the U.S. The process of "stowing" or "filling", in which waste material is re-deposited in abandoned mine areas, is extensively measured in British studies, but is not used at all in the U.S. coal mines.

All foreign countries use some mining machines and techniques not found in the United States today. Since the type of mining can dramatically affect the dust sizes, results from foreign mining technology must be interpreted cautiously.

(2) Particle Diameters.

Many different particle diameters have been defined, depending on the method of sizing and the application of the measurement. For simplicity, this review will consider only 4 definitions of particle diameter:

D = aerodynamic diameter, the diameter of a unit density sphere with the same settling velocity in air as the particle being measured.

D_{st} = Stokes diameter, the diameter of a sphere with the same density and settling velocity as the particle being measured.

D_{ev} = equivalent volume diameter, the diameter of a sphere with the same volume as the particle being measured.

D_p = projected diameter, the diameter of the two-dimensional projection of the particle seen through a microscope.

The projected diameter itself can be defined in a vast number of ways (see Allen (1974) for examples). However, these refinements are not pursued here.

In this review, D_p, D_{ev} and D_{st} all have to be converted to aerodynamic diameter D. As summarized by Kotrappa (1972), the conversions between these diameters can be given in terms of the following quantities (where the Cunningham slip factor is set equal to 1):

K = dynamic shape factor

$$= (D_{ev} / D_{st})^2 \qquad \text{(II-2)}$$

R = ratio of projected diameter to Stokes diameter

$$= D_p / D_{st} \qquad \text{(II-3)}$$

α_v = volume shape factor

$$= \text{particle volume} / (D_p)^3 \qquad \text{(II-4)}$$

Since the particle volume is also equal to $\pi(D_{ev})^3/6$, the definition for α_v can be re-expressed as:

$$D_{ev} = D_p (6 \alpha_v / \pi)^{1/3} \qquad \text{(II-5)}$$

Finally, the relationship for converting to aerodynamic diameter is:

$$D = D_{st} \sqrt{\rho / (1 \text{ g/cm}^3)} \qquad \text{(II-6)}$$

where ρ is the particle density.

For airborne coal mine dust, the density has been reported to range from 1.3 to 1.5 g/cm^3. Since silica has ρ = 2.6 g/cm^3, dust with high silica content may have even higher densities. However, the extent of this density variation has not been reported in the literature on field samples. Following the assumption usually made in the literature (e.g., Breuer, 1967), the density ρ=1.3 g/cm^3 will be used in all calculations.

The other conversion parameters have been measured in a number of studies, which are summarized in Table II-2. The literature survey shows reasonable agreement about the mean values for $\bar{R}$ = 1.44 (std. dev = 0.10) and $\bar{\alpha_v}$ = 0.29 (std. dev = 0.08). However, serious disagreements exist over the dynamic shape factor K.

To decide on a value for K, we combine Eq. II-2, -3 and -5 to get:

$$K = \bar{R}^2 (6 \bar{\alpha_v} / \pi)^{2/3} \qquad \text{(II-7)}$$

Using the mean values for R and α_v from Table II-2 now gives K = 1.40±0.32. This outcome agrees with K reported by Robins (1954) and by Shrag and Corn (1969), but excludes the results of Kotrappa (1972), Tomb and Corn (1973) and Sansone et al. (1973).

These three measurements of K can be declared to be outliers, not only by this comparison with the R and α_v measurements, but by critiques of these studies found in the literature. On the K value measured by Kotrappa (1972), Stoeber (1972) comments : "The consistently high values of K = 1.9 for coal particles of sizes below 3 x 10^{-4} cm seem to suggest that a systematic error may be involved in the measurement....If the high values are correct, it would mean that the representative oblate spheroid at random orientation would have an axial ratio in excess of 1:20, which is not in agreement with the high value of α_v = 0.38, which indicates sphere-like shape."

According to Stoeber's theories (1972), the values of K $\leq$ 1.0 in Tomb and Corn (1973) and in Sansone, Bell and Buchino (1973) are also impossible for randomly oriented particles. Again, experimental errors are indicated. Tomb and Corn (1973) reported deagglomeration of the particles dispersed in alcohol, and calculated K from data showing random errors from 67 to 77 percent. Sansone et al. (1973) reject their own measurement of K, done by sedimentation analysis and a Coulter counter. According to their own

Table II-2: Shape Factors for Coal Dust

Parameter	Study	Mean Value	Range	Size Range (μm)
R	Robins (1954)	1.56	NR	D=3-76
	Hamilton (1954)	NR	1.12-2.05	NR
	Timbrell (1954)	1.34	NR	D=3-18
	Cartwright (1962)	NR	1.34-1.40	D=4-14
	Kotrappa (1972)	1.47	1.27-1.59	D_p=0.2-4.3
	Mean	1.44		
α_v	Robins (1954)	0.206	NR	D=3-76
	Cartwright (1962)	0.25	NR	D=4-14
	Shrag and Corn (1969)	0.303	NR	D=2-9
	Kotrappa (1972)	0.381	0.339-0.449	D_p=0.2-4.3
	Mean	0.29		
K	Robins (1954)	1.31	NR	D=3-76
	Cartwright (1962)	1.16	1.11 -1.20	D=4-14
	Shrag and Corn (1969)	1.36	NR	D=2-9
	Kotrappa (1972)	1.88	1.41 - 2.09	D_p=0.2-4.3
	Tomb and Corn (1973)	1.00*	0.05 - 2.7	D_{st}=1.2-5.8
	Sansone, Bell and Buchino (1973)	0.88	0.67 - 1.08	NR
	Mean	1.39		

Note: * Tomb and Corn (1973) report a regression $D_{ev} = 0.09 + 1.00 D_{st}$. The standard deviations in D_{ev} are 67-77 percent of the corresponding D_{st}.

critique: "It is probable that a sedimentation potential was responsible for fine particles remaining suspended after they should have settled. The implication of this with respect to size distributions obtained from sedimentation balance runs is that the observed distributions would be skewed toward the larger particle sizes. This could lower the calculated [$D_{ev}/D_{st} = K$] ratios. It is not possible, at this time to estimate the magnitude of the electroviscous effect on the values of K obtained."

In conclusion, there is consensus on the shape factors for coal dust if the K values measured by Kotrappa (1972), Tomb and Corn (1973) and Sansone et al. are excluded on the basis of their reported experimental error. In summary, we therefore combine Equations II-6, II-2 and II-5 to produce the following relationships to derive aerodynamic diameters:

$$D = D_{st}\sqrt{\rho} \qquad \text{(II-8)}$$

$$= 1.14\ D_{st}$$

$$D = (D_{ev} / R)\ (\pi / 6\ \alpha_v)^{1/3}\sqrt{\rho} \qquad \text{(II-9)}$$

$$= 0.96\ D_{ev};$$

$$D = (D_p / R)\sqrt{\rho} \qquad \text{(II-10)}$$

$$= 0.79\ D_p$$

(3) Methods of Sampling

In this review, the only sampling methods considered are those which give an unbiased sample of aerosols, especially around the respirable size range (1 - 10 μm). Therefore, we have not included two excellent studies of coal dust size distributions samples with horizontal elutriators (Corn et al., 1973; and Ogden and Rickmann, 1977).

All the British studies considered took their samples with the Standard Thermal Percipitator (STP) (Allen, p. 62-64, 1975). Studies indicate that the STP collects all particles smaller than approximately 15 μm (Watson, 1958).

Most of the other samples are taken with filters held in devices such as the 37 mm Millipore cassette. A study of these "total dust" samplers by Fairchild et al. (1981) shows that they can have biased selection properties, especially at larger particle sizes and at ambient air speeds different from the sampler's inlet velocity. The ideal sampling method is isokinetic sampling, matched to the speed and direction of the ventilation stream. We have not found any reports of isokinetic sampling for size distribution measurements conducted in coal mines.

(4) Methods of Sizing

Optical Microscope gives some version of the projected diameter D_p. Considerable work was spent on developing the accuracy of microscope counting, especially by the British Pneumoconiosis Field Research (PFR).

The PFR studies did discover a major source of error in their counts due to overlapping particles (Ashford et al., 1963). However, they concluded that this error does not affect the size distribution studies such as Fay and Ashford (1960).

Electron Microscope also measures D_p. Although the electron microscope does measure particles smaller than the optical microscope, the treatments needed for slide preparation have been reported to deagglomerate the complexes found in the large size ranges (White and De Nee, 1972). As shown by electron microscope photos (Figure II-1) copied from Kotrappa (1972) (see also De Nee, 1971), agglomerates are an important characteristic of coal mine dust. Thus, a sizing method which causes deagglomeration will give size distributions biased towards smaller diameters.

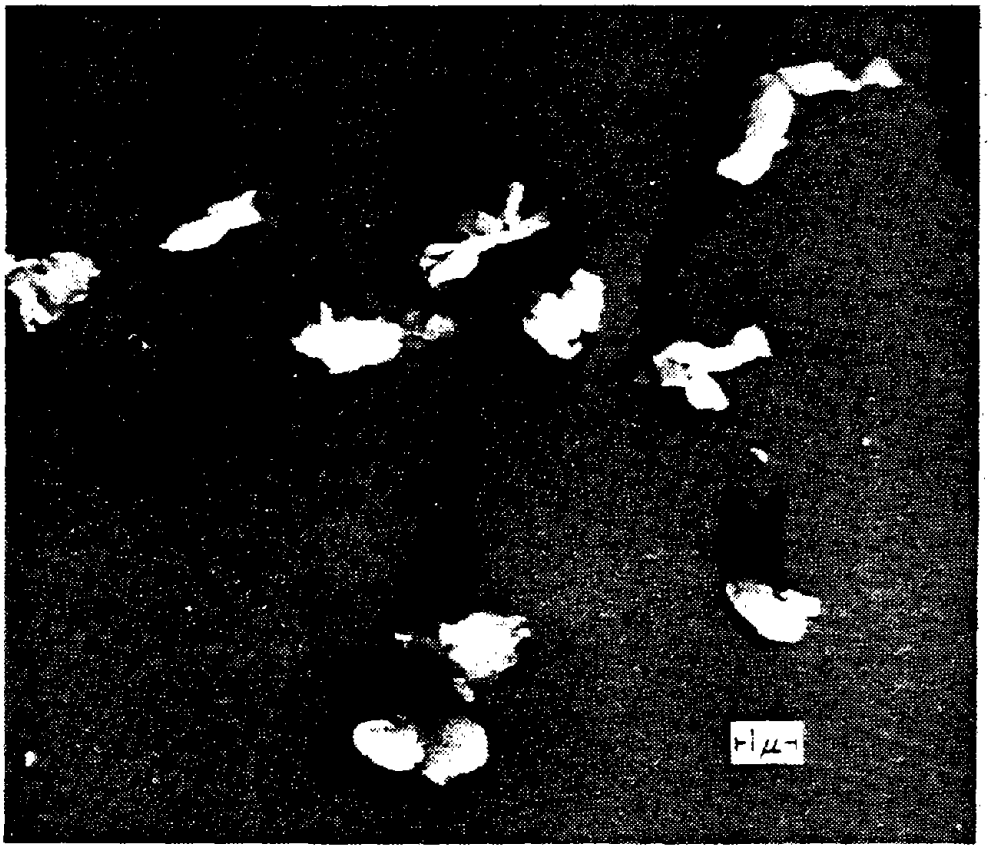
Sedimentation Methods measure Stokes diameter D_{st}. For Andreasen sedimentation analysis, the coal dust has to be suspended in a liquid. According to a self-criticism by Breuer (1967), "a great many aggregates were present in mine dust which were broken up by wet sedimentation analysis and were evaluated quite erroneously." The same criticism can be applied to all the other sedimentation analyses reviewed here.

Coulter Counter measures equivalent volume diameter D_{ev}. As documented in many places (e.g., Tomb and Corn, 1973), the liquid suspension used by the Coulter counter also tends to deagglomerate coal dust particles.

Cascade Impactor directly measures aerodynamic diameter D. Accurate results from a cascade impactor require that bounce-off of particles to lower stages be prevented. In the one cascade impactor study done in coal mines, Burkhardt, McCawley, and Wheeler (1983) carefully calibrated the impactors in the laboratory to guard against bounce-off and other biases. Because the cascade impactor measures aerodynamic diameter directly, this study contains the best current data on the size distributions in U.S. coal mines.

Figure II-1: Scanning electron micrograph of coal dust (D = 1.46 μm).

SOURCE: Kotrappa (1972)



(5) Size Distributions

The particle sizing results are reported either as the number of particles in a size range (microscope counting), the particle mass (Andreasen sedimentation and cascade impactor) or both (Coulter counter). Therefore, both number and mass distributions in particle diameter appear in the literature.

Since respirable dust is now regulated according to its mass concentration, the log-normal mass-weighted size distribution dM/dD (Eq. II-1) is the function of interest. The parameters, Mass Median Diameter (MMD) and Geometric Standard Deviation (GSD), are calculated directly from measurements by the sedimentation, cascade impactor and Coulter counter studies.

Burkhart, McCawley, and Wheeler (1983) report that the coal dust in many locations has at least two modes. The "primary" mode is attributed to the continous mining machines, while the "secondary" mode is believed to arise from re-suspended dust. In this case, the mass-weighted size distribution can be considered to be a weighted average of two log-normal distributions:

$$dM/dD = f\ dM/dD(MMD_1, GSD_1) + (1-f)\ dM/dD(MMD_2, GSD_2) \qquad \text{(II-11)}$$

where f = the mass fraction of the first mode.

However, this bimodal distribution function contains five parameters, which requires data for more particle size fractions than is usually available.

For the microscope counting studies, two alternatives were used to convert number to mass distribution. Where the raw size distribution was reported (either in a table or graph), the data were fitted to the log-normal number distribution:

$$dN/dD = \frac{N}{\sqrt{2\pi}\, D \ln GSD} \exp\left\{ - \frac{\ln^2(D / NMD)}{2 \ln^2 GSD} \right\} \qquad \text{(II-12)}$$

where N = the total number of particles in a given volume of air
NMD = the number median diameter.

According to the theory of the log-normal distribution (Allen, p. 95-97, 1974), "if the number distribution is log-normal, the surface and weight distributions are also log-normal with the same standard deviation". The mass median diameter can then be calculated by the Hatch-Choate equation:

$$\ln MMD = \ln NMD + 3 \ln^2 GSD \qquad \text{(II-13)}$$

Another approach was necessary for the PFR studies, in which microscope size data were fit to the exponential distribution:

$$dN/dD_p = \beta \exp[\beta (1 - D_p)] \qquad \text{(II-14)}$$

As noted in Table II-1, these studies only report the β parameters.

(6) Parameter Estimation

The next step in this literature review is estimating the MMD and GSD parameters for each of the coal mine dust samples reported in the various studies. Depending on the method used in the original study, the MMD and GSD are estimated by a transformation of the exponential distribution parameter β, by a probit plot analysis or by accepting the parameters derived by the original authors.

(a) From the Exponential Distribution. The bulk of the mass distribution data from British coal mines (Fay and Ashford, 1960) is presented in the form of a parameter β in an exponential model of the data (Equation II-14). In Fay and Ashford (1960), the values of β range from 0.41 - 0.81 (mean = 0.54) around longwall faces and from 0.37 - 1.12 (mean = 0.60) elsewhere undergound. The model reputedly fits coal dust distribution data well in the range $D_p \geq 1.0$ µm.

Converting the exponential distribution (Eq. II-14) from projected to aerodynamic diameters, using Equation II-10, we have:

$$dN/dD = 1.22 \beta \exp[\beta (1 - 1.22 D)] \qquad \text{(II-15)}$$

Both Eq. II-14 and II-15 are normalized so that:

$$\int_{D_p=1.0\mu m}^{\infty} dD \, (dN/dD)_{exp} = 1 \qquad \text{(II-16a)}$$

In order to compare the British data with other data considered in this review, conversion from the exponential to the log-normal distribution is necessary. This can be accomplished by selecting log-normal parameters GSD(β) and MMD(β) for each value of β so as to best fit the exponential distribution by the log-normal distribution.

The mathematical details are as follows: The log-normal number distribution dN/dD given as in Eq. II-12 is renormalized so that:

$$\int_{D_p=1.0\mu m}^{\infty} dD \, (dN/dD)_{log\text{-}normal} = 1 \qquad \text{(II-16b)}$$

The transformed parameters NMD(β) and GSD(β) are then calculated so as to minimize the function ø(β;NMD,GSD) given by:

$$\phi = \int_{D_p=1.0\mu m}^{\infty} dD\ w(D)\ [(dN/dD)_{log\text{-}normal} - (dN/dD)_{exp}]^2 \qquad (II\text{-}18)$$

The weight function w(D) above is chosen so as to best accentuate the aerodynamic diameters of interest:

$$w(D) = [\theta_{ACGIH}(D)]^2\ D^6 \qquad (II\text{-}19)$$

where θ_{ACGIH} represents the ACGIH respirable dust definition (defined below).

This choice of weight function makes the above procedure equivalent to producing an unweighted least squares fit of ACGIH-defined respirable mass distribution function, as represented by the exponential model, to that given by the log-normal model. BMRC and ACGIH respirable dust definitions are sufficiently close that an equivalent transformation is expected if the BMRC curve were used in the definition of w(D).

As the integrand in Eq. II-18 has no singularities and is close to zero at D_p = 1.0 μm and at D_p = 10.0 μm, numerical evaluation of the integral is simple. Trapeziodal integration with intervals equal to 0.01 μm gives an error estimated at less than 0.1 percent. With ø simply calculable, NMD(β) and GSD(β) are found by minimizing ø by Newton's method (Abramowitz and Segun, 1965, p. 18). MMD(β) is then calculated from NMD(β) using the Hatch-Choate equation (eq. II-13). The results are presented graphically in Figure II-2.

Note that MMD(β) and GSD(β) form a parametric representation of a uni-dimensional curve in the two-dimensional dust distribution space, (GSD, MMD). This means that if the Fay and Ashford data had been fit originally to a log-normal distribution, the resulting distribution parameters (GDS', MMD') would in general differ from the (GSD, MMD) transformed from β, and would not lie along that one-dimensional curve in the distribution space. These different parameters (GSD',MMD') would produce no problem in estimating the respirable dust fraction or bias as long as the exponential fit to the Fay and Ashford data is good within the respirable particle diameter range. In this case, the shift to the (GSD, MMD) point on the curve from the point (GSD', MMD') is simply along a path of constant respirable fraction. In other words, full knowledge of geometric standard deviation and mass median diameter is unnecessary for estimating respirable fraction or bias.

(b) From Probit Plots. The preferred method for estimating the MMD and GSD is presently the well-known probit plot analysis (e.g., Allen, 1974). In this method, the size distribution data is first plotted on log-probit paper (Figure II-3). For each particle fraction, the diameter is plotted on the logarithmic axis, and the cumulative frequency is plotted on the probit axis.

Figure II-2: Log-normal distribution parameters, mass median diameter (MMD) and geometric standard deviation (GSD), as functions of the exponential distribution parameter β.

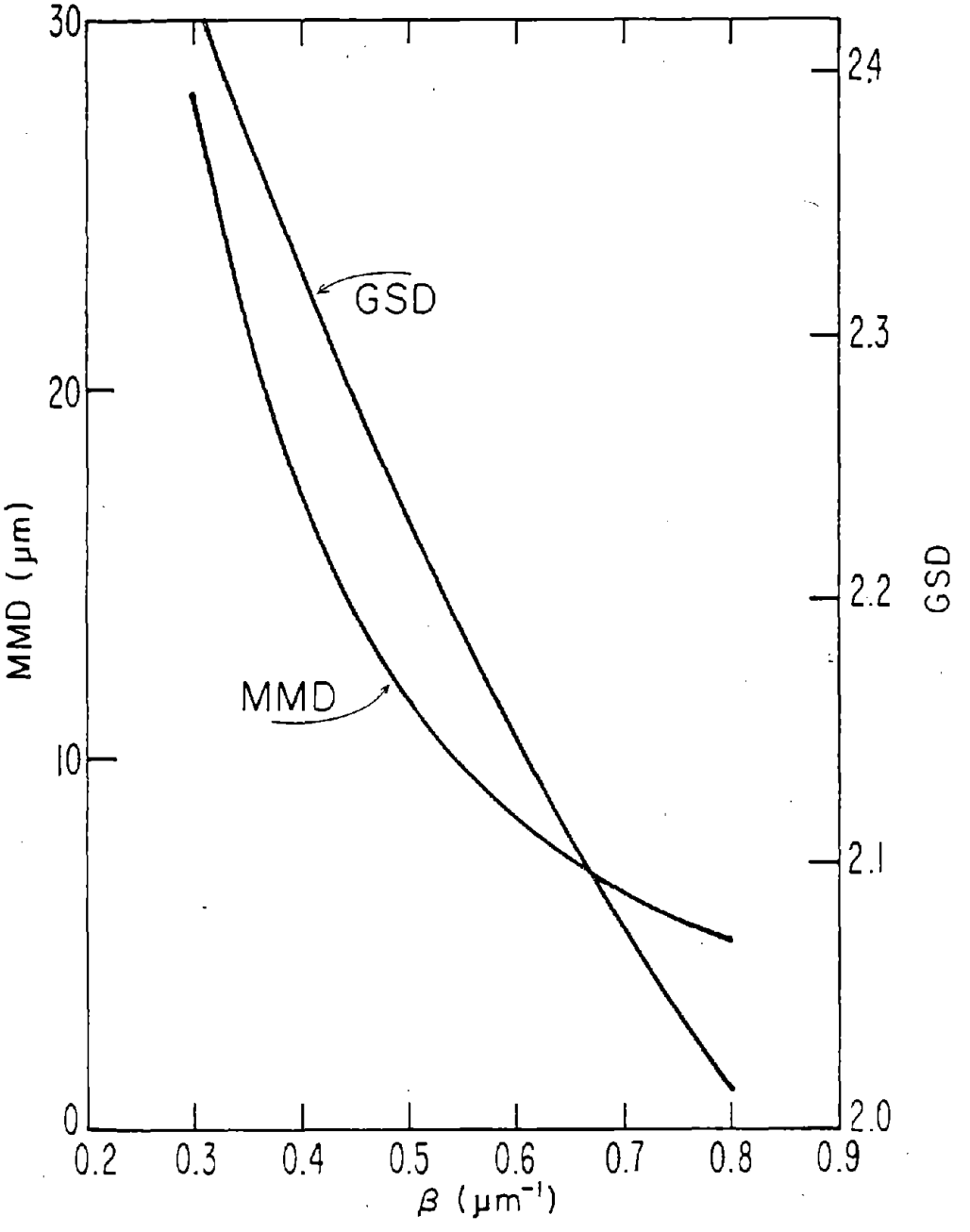
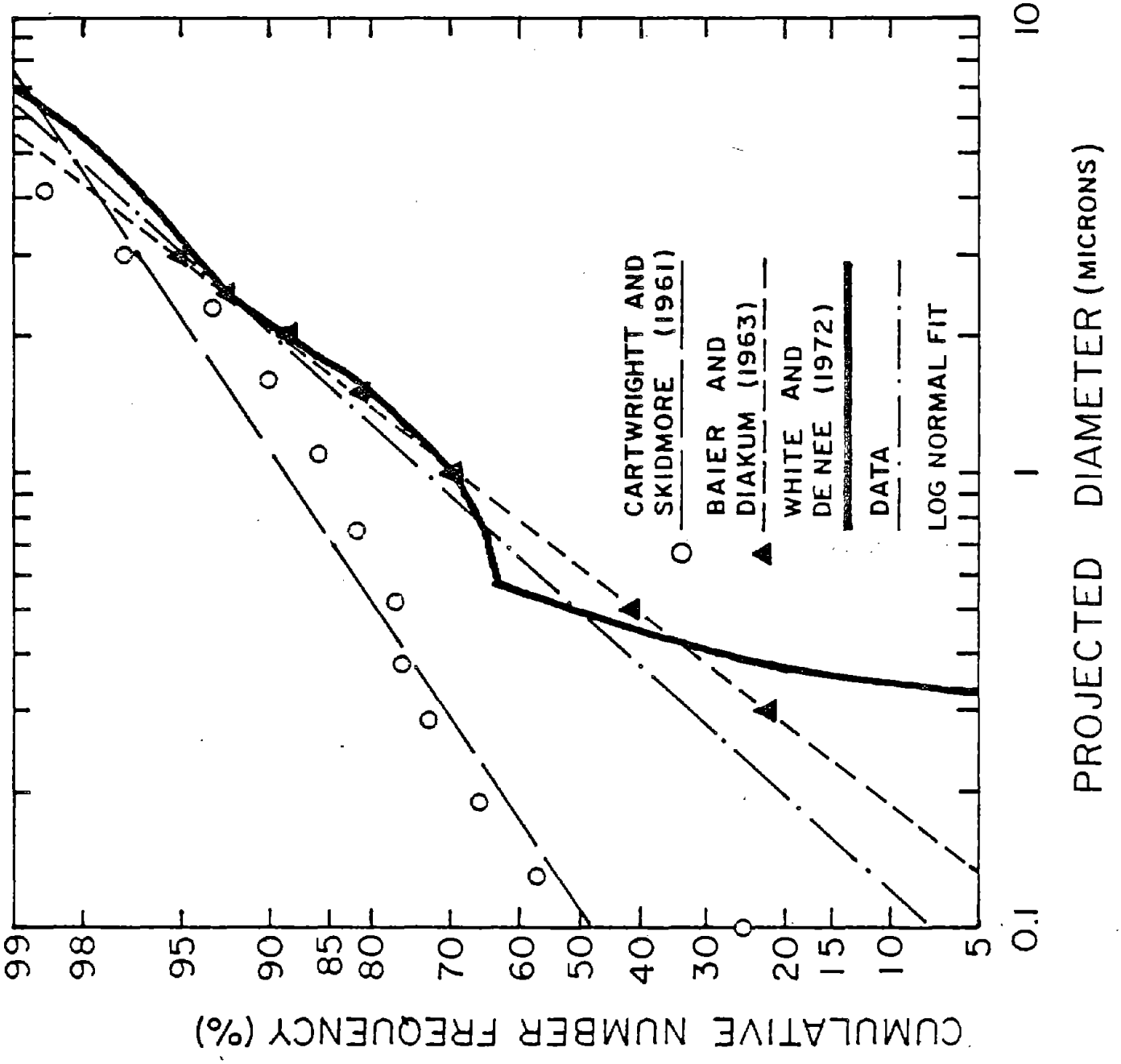


Figure II-3: Examples of probit plots for coal mine dust samples, including both the data points and the linear fit made on the assumption of a log-normal distribution.



These probit plots were reproduced in some studies, as noted in Table II-1. In other cases, we had to make probit plots from the reported sizing data. Where the sizing data were only reported as a histogram or other graph, the frequencies and diameters had to be measured from the graph in the literature, a process very susceptible to error.

The next step is to fit a straight line to the data plotted on the log-probit paper. Log-normally distributed diameters will lie along a straight line, as illustrated by the plot from Baier and Diakun (1963) in Figure II-3.

The most difficult step in this procedure is interpreting non-linear probit plots, like those shown in Figure II-3. Random deviations from linearity may be due to random experimental errors, and may effectively cancel out each other by judicious fitting of the straight line. Systematic deviations from linearity however can be caused by measurement bias or by multi-modal dust distributions (Irani and Callas, 1963).

For example, a sampler which cuts off either large or small particles will generate probit plots with a non-linear tail, which goes asymptotically towards the lnD axis. Such behaviour can be seen in the probit plot from White and De Nee (1972) in Figure II-3. As long as the cut-off does not affect the center of the plot, this behaviour can be corrected by neglecting the tail and fitting a straight line to the bulk of the data.

Even greater irregularities were sometimes encountered in the probit plots, as illustrated by the data from Cartwright and Skidmore (1961) in Figure II-3. This magnitude of irregularity is found only in the older studies, and is interpreted as systematic errors in the antiquated measurement techniques.

By hypothesizing a physical cause for the irregularities, a plausible straight line could be fit to most of the probit plots. Then, the 50-percent point on the line (D_{50}) is the median diameter, either the MMD for mass-weighted frequencies or the NMD for number weighting. If necessary, the median diameters were then converted to aerodynamic diameter using Eq. II-8, 9, or 10.

The geometric standard deviation is then given by one of the following relationships:

$$GSD = D_{84}/D_{50}$$

$$= D_{50}/D_{16}$$

$$= \sqrt{D_{84}/D_{16}} \qquad \text{(II-17)}$$

where D_{84} = 84-th percentile diameter
D_{16} = 16-th percentile diameter

Finally, NMD and MMD were derived from one another with the Hatch-Choate equation (II-13). The results of this probit analysis are all reported in Table II-3.

Table II-3: Summary of Coal Mine Dust Distributions

TABLE II-3: Summary of Coal Mine Dust Size Distribution

Study	Sample	Log-Normal Distribution Parameters NMD(μm)	MMD(μm)	GSD	Comments
1.Tomb et al. (1983)	17 locations	See Table II-5			Deagglomeration probable
2.Burkhardt et al. (1983)	Primary mode	1.2 - 1.6*	15 - 20	2.5	Median values estimated for 118 samples.
	Secondary mode	0.4 - 2.4	5 - 10	2 - 2.5	
3.Vitek (1977)	Longwall face	4.1*	8.0**	1.6	Outlier
	"Skip bunker"	1.3	16.4	2.5	Outlier
4.White and DeNee (1972)	Sewell Seam Lower Kittanning Seam	0.29**	5.41	2.7	Deagglomeration reported
		0.41	6.5	2.6	
5.Dodgson et al (1971)	"Power loading"	s=0.35-0.75	5.5-18.8	2.1-2.3	
	"Hand filling"	s=0.47-1.12	3.4-11.8	1.9-2.2	
6.Leiteritz, Bauer and Bruckmann (1971)	Coal	1.7	13.0**	2.1	
	Dirt	0.66	3.9	1.8	
7.Landwehr and Bruckmann (1969)	Two Ruhr mines	0.0781	2.2**	2.9	Deagglomeration noted. Results not log-normal.
		1.3 x 10-5	2.8	7.5	
8.Breuer (1967)	1) Longwall plough	0.911	10.2**	2.5	Deagglomeration and other inaccurancies reported.
	2) Longwall shearer	0.92	7.6	2.3	
	3) Longwall shearer	1.22	11.3	2.4	
	4) Longwall shearer	0.54	10.1	2.7	
	6) Longwall shearer	0.42	11.1	2.8	
	7) "Pneum. picks"	1.22	12.4	2.4	
	8) Longwall shearer	0.67	10.4	2.6	
	9) "Pneum. picks"	0.76	17.2	2.8	

Footnotes are at the end of the table.

Table II-3--continued

Study	Sample	Log-Normal Distribution Parameters NMD(μm)	MMD(μm)	GSD	Comments
9.Leiteritz et al (1967)	Anthracite	1.2×10^{-6} *	120**	11.9	Not log-normal, inconsistent with Ref. 6
	Gas coal	0.020	11.3	4.3	
10.Poieluiv et al (1967)+	Different wetting agents	5.61	13.4**	1.7	Not log-normal
11.Rogan, Rae and Walton(1967)	Coalfaces	σ_g=0.42-1.03	4.0-14.7	1.9-2.3	
12.Baier and Diakun (1963, 1964)	Anthracite	0.23-1.22*	3.3-18.1**	2.6	GSD from average of samples; NMD is range of all samples.
	Bituminous	0.29-0.85	0.5-1.4	1.6	
13.Breuer (1961)		0.74*	11.4**	2.6	
14.Cartwright and Skidmore (1961)+	Anthracite	.087**	4100*	6.6	Very irregular
	Bituminous	.072	15.2	3.8	
	Blasting	.029	1.2	3.1	
15.Fay and Ashford (1960)	Longwall faces	σ_g=0.41-0.81	4.6-15.3	2.0-2.3	
	Elsewhere underground	σ_g=0.37-1.12	3.4-18.6	1.9-23	

Notes: Underlined parameters denote linear probit plot, i.e. monomodal log-normal behaviour.

* Derived by Hatch-Choate equation (Eq. II-13)

** Aerodynamic diameter derived using Eqs. II-9 to II-11.

\+ Data obtained from Willeke et al. (1971).

(c) From the Literature. The probit analysis techniques described above are now used with some variations by most investigators. Therefore, the two most recent coal mine dust studies (Tomb, Treaftis, and Gero, 1983; Burkhart, McCawley and Wheeler, 1983) estimated MMD and GSD values by methods comparable to those described above. Despite minor differences in the techniques for handling non-linear probit plots, we have therefore used their reported MMD and GSD parameters from these two studies without extensive re-analysis of the sizing data.

Tomb, Treaftis, and Gero (TTG) measured size distributions on total dust samples which were suspended in an aqueous solution and run through a Coulter counter. Since the Coulter counter measures the equivalent volume diameter (D_{ev}), we convert the mass median values for D_{ev} reported by TTG to mass median aerodynamic diameters, using Eq. II-9. The average and the extremes for the resulting (GSD, MMD) pairs are reported in Table II-4.

In the most recent study, Burkhart, McCawley and Wheeler (1983) measured the distributions of aerodynamic diameters directly with a six-stage cascade impactor. However, the size distribution parameters calculated from a probit analysis cover a very wide range: MMD = 1.61 - 29.00 μm and GSD = 1.18 - 12.73. Moreover, the mean MMD of 11.46 μm is much larger than those reported by TTG.

Burkhart, McCawley and Wheeler (BMW) have explained their results in two ways. First, the cascade impactor works by very different principles than the Coulter counter used by TTG. To compare the two methods, BMW arranged for cascade impactor, Coulter counter and electron microscope analysis of samples taken side-by-side in a laboratory dust chamber.

The resulting MMD (all converted to aerodynamic diameter) are 4.2 μm for the Coulter counter, 7.2 μm for the cascade impactor and 14.8 μm for the electron microscope. From these results, the coal dust diameters measured by Coulter counter is systematically lower than cascade impactor results, as expected. However, the electron microscope result also does not agree with the other two methods, and the explanation of this outcome is not clear.

Assuming that the cascade impactor measures aerodynamic diameter more accurately, the extremely wide range of the size parameters reported by BMW still needs explanation. By plotting frequency distributions from the mean dust mass on the impactor stages (Figure II-4), BMW show that the dust at many sampling locations in a coal mine is actually bimodal. Since bimodal distributions do not have linear probit plots (Irani and Callas, 1963), the MMD and GSD values derived by assuming a mono-modal distribution may be seriously affected.

Moreover, the five parameters in a bimodal distribution function (Eq. II-11) cannot be accurately estimated from a six-stage impactor sample.
From the data they have presently collected, BMW offer a qualitative description of the coal mine dust size distributions:

Table II-4. Size Distribution Parameters from Coulter Counter Analysis of MSHA's Total Dust Samples (Mean and Extreme Values Only)

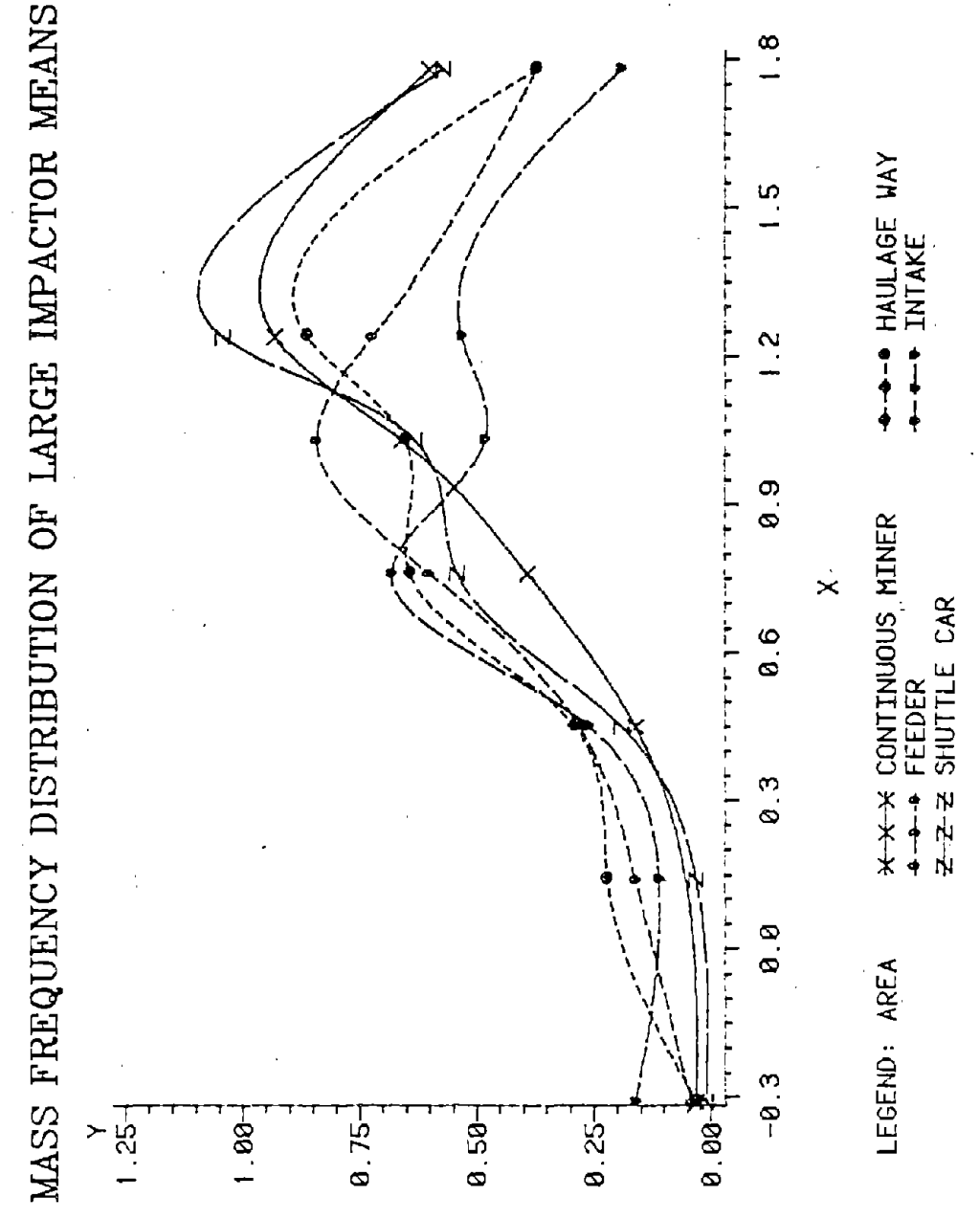
SOURCE: Tomb, Treaftis and Gero (1983)

Sampler Location	Mine	MMD (μm)	GSD
Continuous Miner	Holton Taggart	2.88	2.19
	Cedar Grove	4.13	1.95
	Loveridge*	10.08	2.92
	average	5.05	2.43
Longwall Face	Bethlehem No. 33	5.38	2.55
	Gary No. 50	6.14	2.23
	Moss No. 4	7.30	2.28
	Bethelhem No. 33	6.43	2.63
	average	6.48	2.37
Dump Point	Bethlehem No. 78	3.01	1.87
	Harman	7.70	3.01
	Harlan seam*	3.07	2.81
	average	4.92	2.45

* Note: Size distributions reported in private communication (Treaftis, 1981).

Figure II-4: Frequency Distributions for the Means of Cascade Impactor Samples from Areas in Underground Coal Mines. The frequency on the y-axis is expressed as the mean mass on an impactor stage (ΔM) divided by the total dust mass (T) and weighted by the inverse of the diameter range for that stage (Δ log D).

SOURCE: Burkhart, McCawley and Wheeler (1983)



"The primary source of the larger-size mode dust comes from the operation of the continuous miner. It should be noted that only the continuous miner shows no bi-modality. [See Figure II-4.] The single mode can be overlayed on all other size distributions and shows little difference .....

"Another point of interest is the source of the smaller-size mode noted in areas away from the face. This 'secondary' dust is likely to be the result of the action of people and machinery resuspending settled dust ....

"In certain areas of the mine, this 'secondary' source contributes as much to the mass as does the 'primary.' That is, the two modes appear to have nearly equal areas under them."

-- Burkhart, McCawley and Wheeler (1983)

Although the parameters for the bimodal distribution cannot be determined accurately for each cascade impactor sample, BMW estimate that the primary mode has MMD = 15-20 μm and GSD = 2.5, while the secondary mode has MMD = 5-10 μm and GSD = 2-2.5.

Further tests are now underway to characterize the bimodal distribution more thoroughly. At the present time, however, the BMW study is the best effort to determine the size distribition in coal mines using cascade impactors.

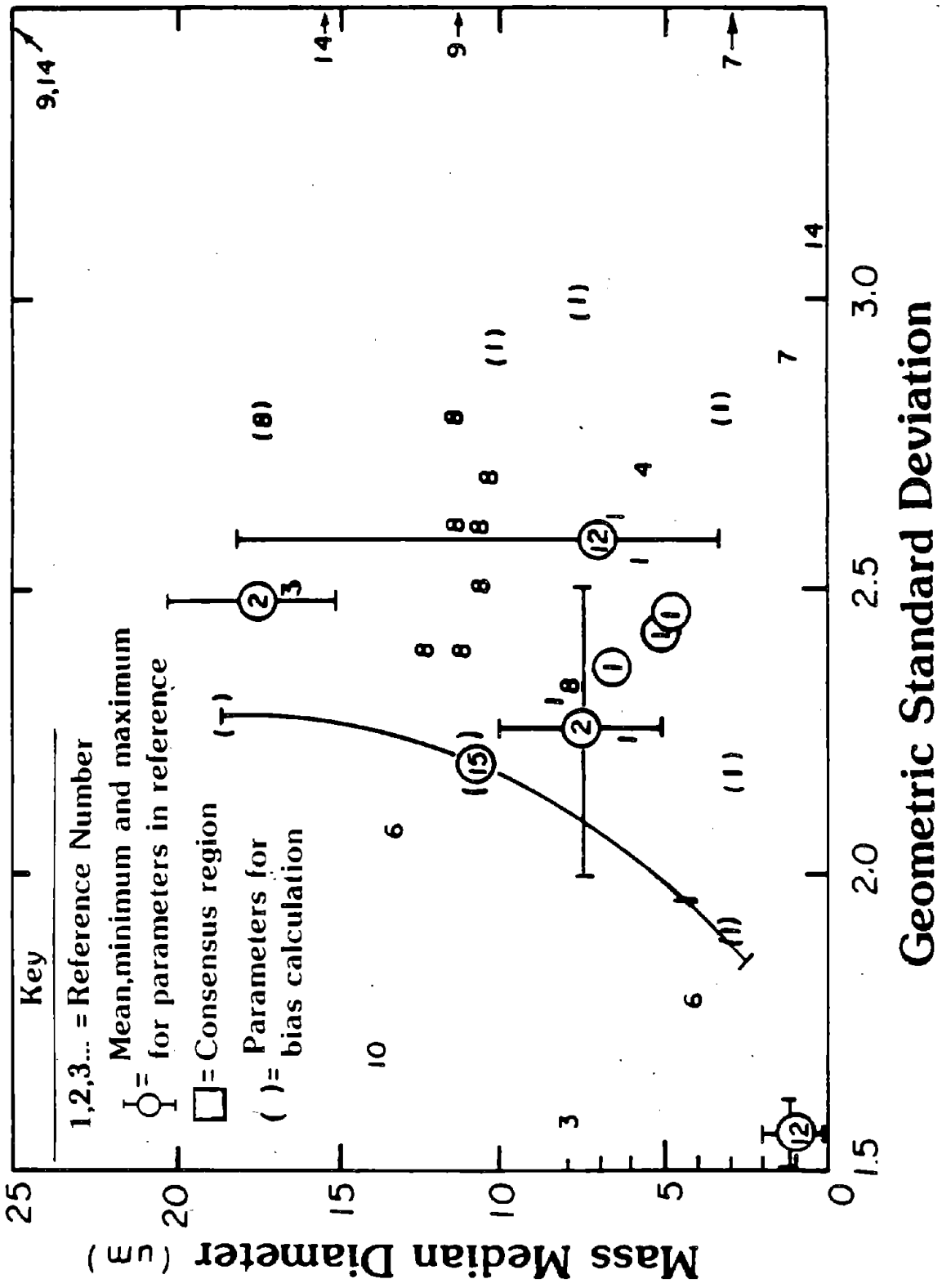
(7) The Consensus Region. In order to put limits on the accuracy of CMDPSU samples, we need to put limits on the dust size distributions expected in U.S. coal mines. In order to establish these limits, we have plotted all the sizing measurements included in this review onto the (GSD, MMD) plane (Figure II-5).

Since these measurements were taken with many different methods whose biases are generally unknown, establishing limits must rely primarily on subjective judgments of the reliability of the different studies. Based on our review of the literature, the most thorough and credible dust distribution studies have been done by the British Pulmonary Field Research (Reference 5: Dodgson et al., 1971; Ref. 11: Rogan, Rae and Walton, 1967; and Ref. 15: Fay and Ashford, 1960), by the U.S. Mine Safety and Health Administration (Ref. 1: Tomb, Treaftis and Gero, 1983) and NIOSH's Division of Respirable Disease Studies (Ref. 2: Burkhart, McCawley and Wheeler, 1983). Although there are differences among the methods used in these studies, as discussed above, their results do show a rough consensus which can be seen in Figure II-5.

Also included in this consensus region are size distributions measured by other studies (as numbered in Table II-3 and Figure II-5):

3. Vitek (1977)
4. White and DeNee (1972)
8. Breuer (1967)
12. Baier and Diakum (1964)
13. Breuer (1961)

Figure II-5: Summary of coal mine dust distribution measurements. The numbers represent (MMD,GSD) pairs measured by the corresponding study listed in Table II-3.



The studies whose results lie outside this consensus region generally have detectable flaws, such as the highly non-linear probit plots noted in Table II-3.

Thus, the limits on the dust size distributions in U.S. coal mines will be taken to be the boundaries of the consensus region, shown in Figure II-5. The measured size distributions on the margins of this region are listed in Table II-5.

DISCUSSION

These results must be treated with caution because every study in this review has deficiencies from the viewpoint of estimating CMDPSU bias. The main shortcomings are:

* density is assumed to be 1.3 g/cm^3, but can vary from this value by 10 percent or more.

* particle deagglomeration in Coulter counter, sedimentation and electron microscope analysis. The effects of deagglomeration on the log-normal parameters have never been established quantitatively. This process will certainly bias the MMD towards smaller values and may affect the GSD as well.

* sampling locations where miners do not work or mining operations not currently found in U.S. mines.

* sampling and/or analytical errors, which appear as outlier values of the MMD and/or GSD parameters or as non-linear probit plots.

* the necessity to transform the PFR data from the experimental distribution, with the attendant error possibilities.

* graphs alone were often used to present dust size measurements, so the conversion to log-normal parameters required the measurement of co-ordinates from the graphs reproduced in the literature, an error-prone process.

* the errors from fitting the log-normal distribution to cases where other distributions (e.g. bimodal curves) may have been appropriate.

* where the bimodal distribution was detected by Burkhart, McCawley and Wheeler (1983), they only had six-stage impactor data available, so the five parameters for this distribution could not be determined accurately.

The overall error from all these sources is impossible to estimate, but can clearly be large. The dynamic shape factor K alone has an error of estimate of 23 percent. In light of the potential for errors, the agreement between these varied studies is remarkable.

Table II-5 Extreme Coal Mine Dust Size Distributions Used in Bias Calculation

	Source	Sample Site	MMD (μm)	GSD
1.	Tomb, Treaftis and Gero (1983)	Dump point, Bethlehem No. 78	3.0	1.9
		Continuous miner, Holton Taggart	2.9	2.2
		Dump point, Harlan seam	3.1	2.8
		Continuous miner section, Loveridge	10.1	2.9
		Dump point, Harman	7.7	3.0
8.	Breuer (1967)	"Pneumatic pick"	17.2	2.8
15.	Fay and Ashford (1960)	Maximum, away from face	18.6	2.3
		Mean, longwall face	8.0	2.15

From this error analysis, we conclude that further size distribution studies are needed for estimating CMDPSU bias. Until such results are available, we have taken a conservative approach to setting limits on the range of size distributions which can be expected in coal mine dust. Although BMW have shown that the Coulter counter, the cascade impactor, and the electron microscope produce widely disparate size parameters from the same dust, we have included results from these three methods in the "consensus region" in Figure II-5. When more accurate size distribution measurements have been taken, we may therefore expect a smaller range of (GSD,MMD) points.

The wide variation in (GSD, MMD) values in Figure II-5 provides a major motivation behind sampler characterization at GSD = 1 (a monodisperse aerosol) as proposed in this report. Monodisperse sampler characteristics can be used to compute predicted sampler bias over a wide range of size distributions. Although there is no proof that the dust size distribution affects CMDPSU precision (as distinguished from the bias), we also require that the precision test be done with a coal dust with a size distribution representative of those found in the mines.

C. PRECISION MEASUREMENT

The random errors in CMDPSU sampling can be measured by taking replicate samples in the same dust environment. The total coefficient of variation CV_t (or relative standard deviation) from replicate samples of this kind is the measure of sampling precision used in NIOSH's statistical protocol for validating other industrial hygiene analytical methods (Busch and Taylor, 1981).

For purposes of certifying CMDPSUs, we recommend that CV_t be measured in the laboratory, using procedures as close as practical to those required for routine sampling in coal mines. Although field studies of cyclone sampling usually show poorer precision than laboratory studies (Bowman, Bartley, Breuer, and Shulman, 1982), accurate determination of CV_t in the field would be prohibitively difficult for routine CMDPSU certification. Therefore, laboratory precision tests are recommended for CMDPSU certification, just as they are used for the validation of NIOSH's other sampling and analytical methods (Gunderson and Anderson, 1980).

A number of investigators have reported the results from replicate laboratory samples with the 10 mm nylon cyclone. These studies are reviewed in depth by Bowman, Bartley, Breuer, and Shulman (1982), and their conclusions are summarized in Table II-6.

These precision tests were done under a variety of conditions: pumps in some, critical orifices in others; CMDPSUs in some, custom sampling heads in others. In light of these differences, the wide range in CV_t estimates (4 to 14 percent) is not surprising.

From reviewing this literature, the main causes of this variability in precision measurements appears to be uneven distribution of dust within the test chambers, and different sampling equipment used by the different investigators. As an example of chamber effects, the Bureau of Mines (1972) reported cyclone CV_t = 8.0 percent from tests done in their standard dust chamber, but CV_t = 5.4 percent with Lippmann's multi-cyclone holder. As an example of equipment effects, Caplan, Doemeny and Sorenson (1977) noted high variability in their replicate samples, due to faulty alignment of the vortex finder on some of the MSA units. Consequently, MSA now provides a clip with their CMDPSU to assure correct alignment. CMDPSUs made before and after this modification could, therefore, perform differently in precision tests. In light of this review, we have, therefore, sought to design a precision test which would measure the CV_t for the equipment submitted for certification, independent from the testing apparatus.

Another objective of the experimental design has been to maintain a reasonable balance between the confidence limits on CV_t and the cost of the precision test. To measure CV_t within narrow confidence limits, a large number of test runs and/or samplers is needed. However, the size of the experimental design is constrained by its costs in labor and/or sampling equipment.

Table II-6. Precision estimates for the 10 mm cyclone from replicate laboratory samples

SOURCE: Bowman, Bartley, Breuer and Shulman (1983)

Study	Degrees of freedom	Pooled CV_t (percent)	Samplers
MSHA data (1981b)	29	4.3	Bendix samplers w/ DuPont pumps
Gray and Tillery (1979)	154	7.1-4.6	Custom samplers w/critical orifices
Harris, DeSieghardt and Riva (1976)			
MSA units	38	14.5	Complete CMDPSUs
Bendix units	39	11.8	
Almich and Carson (1974)	66	4.8	Custom Samplers w/critical orifices
Bureau of Mines data (1972)	58	8.0	MSA units in dust chamber
Jacobson (1971)			
MSA units	8	6.4	Test of pulse dampeners
Bendix units	7	11.1	

A third goal for the experimental design is to measure both the differences between individual sampling units (inter-sampler variance) and run-to-run differences with the same sampler (intra-sampler variance). Unlike most gas and vapor samplers, aerosol samplers may show significant variability from unit to unit, as Lippmann and Chan (1974) found with stainless steel cyclones. Therefore, the experimental design should be capable of measuring this inter-sampler variance separate from the intra-sampler variance. This goal can be achieved by a design, which has two or more runs with the same samplers at a given concentration.

Moreover, the estimate of CV_t should combine these inter-sampler and intra-sampler variance components with equal weight. This weighting assumes that the random error in a single sample taken routinely in a mine consists of the random intra-sampler error on that occasion, plus the error characteristic of the CMDPSU chosen at random from the relatively large stock available. Such weighting is assured in the CV_t by the analysis of variance discussed below.

Finally, the experimental design will be guided by the protocol for NIOSH's validation of sampling and analytical methods (Busch and Taylor, 1981). Since this protocol has been widely applied to testing industrial hygiene sampling methods, we would prefer to retain elements of this experimental design as long as they are appropriate to CMDPSU testing. Since NIOSH's validation tests were developed primarily for gas and vapor samplers, this protocol does not consider either chamber effects or inter-sampler variance. For these reasons, the CMDPSU precision test must depart significantly from the NIOSH protocol.

EXPERIMENTAL DESIGN

The major goals for an experimental design for measuring CMDPSU precision, therefore, are:

* tight confidence limits on CV_t, while keeping down the costs of the test;

* measuring CV_t independent of the properties of the dust chamber;

* inclusion of both inter-sampler and intra-sampler variation with equal weight in the coefficient of variation;

* CV_t should have an interpretation compatible with NIOSH's protocol for validation of sampling and analytical methods.

In the original NIOSH validation tests, the experimental design had six samples taken at target concentrations equal to 0.5, one and two times the applicable health standard. All variability within the run was assigned to the imprecision of the sampling and analytical method. Therefore, CV_t was estimated by pooling the coefficients of variation from each run, as long as the values from the three runs were homogeneous. In this case, CV_t is estimated with 15 degrees of freedom (Busch and Taylor, 1981).

To adapt the design of the validation protocol to CMDPSU testing, modifications are necessary. To avoid confounding inter-sampler variations and position effects, the simple block design of the validation protocol must be expanded into a factorial design nested within the target concentration. In this expanded design, we have decided to keep the degrees of freedom at approximately 15, so that the width of the confidence limits on the CV_t estimate will be unchanged.

To achieve 15 degrees of freedom at a reasonable cost, we recommend that the number of test concentrations be reduced to two levels from the three levels used in the NIOSH protocol. To assure that the test covers a range around the health standard, we recommend that the target dust concentrations for the precision measurement be the extremes of the range covered by the NIOSH protocol, i.e., twice and one-half the standard. Since the coal mine dust standard is 2.0 mg/m^3 (assuming less than 5 percent silica), 1.0 and 4.0 mg/m^3 are the target concentrations of respirable dust. In keeping with Federal regulations (30 CFR 70 and 71), the respirable dust concentrations taken with the cyclone samplers will be converted to the equivalent taken with the MRE horizontal elutriator.

Finally, the experimental design is constrained by the test chamber's characteristics. The original work on this project was done on two chambers built for NIOSH by Los Alamos Scientific Laboratory (Fairchild et al., 1973). The larger chamber holds 8 cyclones, but showed considerable differences in the dust concentrations at the different sampling positions. The smaller chamber showed less inhomogeneity, but holds only four samplers, reducing the confidence limits on a CV_t derived from a single run. More recently, Rubow and Marple (1983) have designed a large instrument evaluation chamber which eliminates position effects by mounting as many as six samplers on a rotating table.

For testing CMDPSUs, we anticipate that the government and CMDPSU manufacturers may also use a variety of dust chambers with different capacities and different levels of dust homogeneity. To deal with this variety, we therefore recommend several experimental designs which meet the above criteria. The first set of designs (Table II-7) are for chambers with homogeneous dust concentrations at all sampler positions, i.e., no position effect in the analysis of variance. This design can be executed efficiently in chambers holding either four or five CMDPSUs. In both cases, the samplers are labeled (A, B, C . . . .), and retain their identity throughout the test. The sampler labeling, together with the nested design (i.e., at least two runs for each target concentration), enables the independent determination of inter- and intra-sampler variance.

For chambers with consistently different dust concentrations at the various sampler positions, an additional level of nesting is needed in the experimental design to eliminate this position effect from the CV_t estimate. Table II-8 shows such a design for a chamber with capacity to hold four CMDPSUs. As in Table II-7, the sequence of runs would be randomized in the actual measurement, in order to minimize the bias from any changes developing during the course of the test.

Table II-7. Experimental Designs for Chambers with Homogeneous Concentration at all Sampler Positions. (For the actual measurement, the order of the target concentrations would be randomized.)

Target Concentration (mg/cu.m.)	Run	Samplers
	Chamber Capacity = 4 CMDPSUs	
1.0	1	A B C D
	2	A B C D
	3	A B C D
4.0	1	E F G H
	2	E F G H
	3	E F G H
	Totals: 6 runs 8 CMDPSUs 24 cassettes	
	Chamber Capacity = 5 CMDPSUs	
1.0	1	A B C D E
	2	A B C D E
4.0	1	F G H I J
	2	F G H I J
	Totals 4 runs 10 CMDPSUs 20 cassettes	

Table II-8. Experimental design for the precision measurement, assuming that the sampling positions in the chamber may have different dust concentration. (In the actual measurement, the sequence of experiments and target concentrations are randomized.)

Target Concentration (mg/cu.m.)	Experiment	Run	Position 1	2	3	4
1.0	I	1	A	B	C	D
		2	A	B	C	D
	II	1	E	F	G	H
		2	E	F	G	H
4.0	I	1	I	J	K	L
		2	I	J	K	L
	II	1	M	N	O	P
		2	M	N	O	P

Totals: 8 runs
16 CMDPSUs
32 cassettes

ANALYSIS OF VARIANCE
Derived by Kenneth A. Busch (1981, 1982)

The analysis of variance (ANOVA) extracts the precision estimate from the extraneous sources of variation, such as the different target concentrations and different chamber positions. The first step in the ANOVA is to take the natural log transform of all the concentrations measured during the experimental design chosen from Tables II-7 or II-8. The ANOVA method assumes that the variance in the response variables is homogeneous over all the runs in the experimental design, and the log transform helps meet this assumption. Therefore, the response variable in the ANOVA is:

$$LNC = \ln[\ C(mg/cu.m.)\ ] \qquad \text{(II-18)}$$

For the experimental designs without a position effect (Table II-7), the following quantitites are then assumed to contribute additively to the variations in LNC :

* T_l — The deviation caused by the l-th target concentration, where l = 1, 2. This is a fixed effect with a mean square $\theta(T) = (T_1)^2 + (T_2)^2$.
* $RUN_{m(l)}$ — The deviation for the m-th run (m = 1, 2 ... M) in the l-th target concentration; a random effect with variance $(\sigma_R)^2$.
* $S_{n(l)}$ — The inter-sampler deviation for the n-th CMDPSU (n = 1,2... N) in the l-th target concentration; a random effect with variance $(\sigma_S)^2$, caused by differences in the cyclone's collection efficiencies.
* $\varepsilon_{k(lmn)}$ — The intra-sampler deviation for the k-th sample taken with the n-th CMDPSU for the m-th run in the l-th target concentration; a random effect with variance $(\sigma_\varepsilon)^2$.

Therefore, the basic model for the ANOVA is:

$$LNC_{klmn} = \mu + T_l + RUN_{m(l)} + S_{n(l)} + \varepsilon_{k(lmn)} \qquad \text{(II-19)}$$

where μ is the true over-all mean. In this model, the interaction terms like RUN*S are all assumed to be zero. This assumption is logical because variations in the samplers should have no effect on the dust concentration produced by the chamber for the different runs.

Following ANOVA theory (for example, Snedecor and Cochran, 1967), the variances predicted by this model can be related to the expected mean square terms (EMS) resulting from the ANOVA (Table II-9). Table II-9 shows that inter- and intra-sampler variances are isolated in two EMS terms:

$$EMS(S\ in\ T) = M\ (\sigma_S)^2 + (\sigma_\varepsilon)^2 \qquad \text{(II-20a)}$$

$$EMS(Error) = (\sigma_\varepsilon)^2 \qquad \text{(II-20b)}$$

However, neither EMS combines $(\sigma_S)^2$ and $(\sigma_\epsilon)^2$ with equal weighting, as is desired of a precision estimate representing what happens routinely in coal mines. The general method for obtaining the desired precision estimate is to take a linear combination of two EMS terms such that:

$$(\sigma_t)^2 = A\ EMS_a + B\ EMS_b \qquad \text{(II-21)}$$

$$= (\sigma_S)^2 + (\sigma_\epsilon)^2$$

where the coefficients A and B are chosen to make the identity correct. For the EMS terms in Eq. (II-20), the appropriate linear combination is:

$$(\sigma_t)^2 = M^{-1}\ EMS(S\ in\ T) + M^{-1}\ (M - 1)\ EMS(Error) \qquad \text{(II-22)}$$

By using Satterthwaite's approximation (see Searle, 1971, p.142), $(\sigma_t)^2$ can be treated statistically as if it were a single EMS term in the ANOVA with approximate degrees of freedom f* given by:

$$f^* = \frac{[A\ EMS_a + B\ EMS_b]^2}{(A\ EMS_a)^2/f_a + (B\ EMS_b)^2/f_b} \qquad \text{(II-23)}$$

where f_a and f_b are the degrees of freedom for the corresponding EMS.

f* is a variable depending on the actual values of the mean squares resulting from a measurement. However, the maximum and minimum values for f* can be determined from Eq. II-23. For the ANOVA in Table II-9, f* ranges from 2(N-1) to 2M(N-1). In the two experimental designs recommended in Table II-7, the maximum degrees of fredom (d.o.f.) in the precision estimate is 18 for the four-sampler chamber and 16 for the five-sampler chamber -- about the same as the 15 d.o.f. in NIOSH's validation protocol (Busch and Taylor, 1981). Note that the d.o.f. in $(\sigma_t)^2$ is less than the total generated by the design. The extra degrees have been used to compensate for the effects of the target concentrations and the runs.

The final step in the statistical analysis for these designs is to transform Eq. (II-22), which gives a variance for the natural log of the concentrations, back to an estimate of CV_t for the original concentrations. According to a theorem by Hald (1952, p. 247), a variable x which is normally distributed with constant coefficient of variation CV_x will approximately obey the relationship:

$$CV_x \cong \sigma_{\ln x} \qquad \text{(II-24)}$$

Therefore,

$$(CV_t)^2 \cong (\sigma_t)^2 \qquad \text{(II-25)}$$

A second theorem (Hald, 1952, pp. 164, 175) says that x can be regarded as normally distributed as long as CV_x is small (1/3 or less), irrespective of whether x is truly normal or lognormal. Therefore, Eq. (II-25) is a valid estimate for CV_t whether the underlying concentration distribution is actually lognormal or normal with a constant coefficient of variation.

Table II-9
Analysis of Variance for the Precision Measurement in Dust Chambers
Without a Position Effect (Table II-7)

Response Variable: LNC = ln[C(mg/m^3)]

M = Number of runs per target concentration
= 3 or 2

N = Number of samplers per run
= 4 or 5

Source of Variation	Degrees of Freedom	Expected Mean Squares
T = Target Concentration	1	$MN\Theta(T) + N(\sigma_R)^2 + M(\sigma_S)^2 + (\sigma_\epsilon)^2$
RUN in T	2(M-1)	$N(\sigma_R)^2 + (\sigma_\epsilon)^2$
S (= Samplers) in T	2(N-1)	$M(\sigma_S)^2 + (\sigma_\epsilon)^2$
Residual Error	2(M-1)(N-1)	$(\sigma_\epsilon)^2$
Total	2MN-1	

The calculation of CV_t can now be outlined. Based on the theoretical ANOVA in Table II-9, the ANOVA calculation can be performed with readily available computer programs like SAS (SAS Institute, 1979). This computation is described in more detail in Section III.A. and Appendix B. From the ANOVA calculation comes mean square values (MS), which are estimates of the expected mean squares for each source of variation. Substituting these MS values into Eqs. (II-22) and (II-25) gives the estimated coefficient of variation (distinguished by a hat):

$$(\hat{CV}_t)^2 = M^{-1}\, MS(S \text{ in } T) + M^{-1}\,(M-1)\, MS(Error) \qquad (II\text{-}26)$$

The ANOVA for the experimental design with position effects (Table II-8) is similar, but much more complex. In addition to the sources of variation defined above, LNC in this design also has variability from:

* $EXP_{j(l)}$ deviation from the j-th experiment (j = 1,2) in l-th target concentration; a random effect with variance $(\sigma_E)^2$.
* POS_n a fixed effect at each position n = 1,2,3,4 with mean square: $\theta(POS) = (\sum_n POS_n) / 3$.

The overall model is assumed to be:

$$LNC_{jklmn} = \mu + T_l + EXP_{j(l)} + RUN_{m(jl)} + POS_n + S_{n(jl)} + \epsilon_{k(jlmn)} \qquad (II\text{-}27)$$

This model neglects all the possible interaction terms, particularly POS*T, but also POS*EXP and POS*RUN. These assumptions should be checked empirically because the position effects could possibly be a function of the overall dust concentration. This question is examined in the next section.

The ANOVA for this model is outlined in Table II-10. From these results, the inter- and intra-sampler variations are isolated in the EMS terms for POS*T, POS*EXP in T and the residual error. To estimate the precision, the terms with the EMS of $2(\sigma_S)^2 + (\sigma_\epsilon)^2$ are first pooled:

$$EMS(pool) = [3\, EMS(POS*T) + 6\, EMS(POS*EXP \text{ in } T)] / 9 \qquad (II\text{-}28)$$

The d.o.f. for EMS(pool) are the sum of its component d.o.f., or 3 + 5 = 9.

In order to obtain equal weighting between inter- and intra-sampler errors, EMS(pool) is next combined with EMS(Error) in a linear combination satisfying Eq. II-21. After reversing the natural-log transform (Eq. II-25), the final precision estimate is:

$$(\hat{CV}_t)^2 = [MS(pool) + MS(Error)] / 2 \qquad (II\text{-}29)$$

$$= MS(POS*T) / 6 + MS(POS*EXP \text{ in } T) / 3 + MS(Error) / 2$$

The degrees of freedom f* for the precision estimate is calculated from Eq. (II-23), and ranges between 9 and 21.

Table II-13. Analysis of Variance for the Precision Measurement With Position Effects (Table II-8)

Response Variable: LNC = ln [C(mg/m^3)]

Source of Variation	Degrees of Freedom	Expected Mean Squares
T = Target Concentration	1	$16\theta(T) + 8(\sigma_E)^2 + 4(\sigma_R)^2 + 2(\sigma_S)^2 + (\sigma_\varepsilon)^2$
EXP in T	2	$8(\sigma_E)^2 + 4(\sigma_R)^2 + (\sigma_S)^2 + (\sigma_\varepsilon)^2$
RUN in EXP	4	$4(\sigma_R)^2 + (\sigma_\varepsilon)^2$
POS (confounded with S)	3	$8\theta(POS) + 2(\sigma_S)^2 + (\sigma_\varepsilon)^2$
POS x T	3	$2(\sigma_S)^2 + (\sigma_\varepsilon)^2$
POS x EXP in T	6	$2(\sigma_S)^2 + (\sigma_\varepsilon)^2$
Residual Error	12	$(\sigma_\varepsilon)^2$
Total	31	

Whichever design is used to measure the precision, error bounds for $\hat{CV}_t$ may be derived by noting that it is distributed approximately as if it were equal to a variance (Eq. II-25). By an elementary theorem of statistics (Snedecore and Cochran, p. 75, 1967), confidence limits on a variance estimate are given by the chi-squared distribution X^2. At the 95 percent confidence limit, the two-sided bound is therefore:

$$\hat{CV}_t \sqrt{f / (X^2)_{0.025}} \leq CV_t \leq \hat{CV}_t \sqrt{f / (X^2)_{0.975}} \qquad \text{(II-30)}$$

where f is the degrees of freedom in the $\hat{CV}_t$ estimate and in the chi-squared function. For example, the confidence limits for f = 15 are:

$$0.739\ \hat{CV}_t \leq CV_t \leq 1.547\ \hat{CV}_t \qquad \text{(II-31)}$$

CONTROL OF ERRORS

In measuring the precision, the errors which need to be controlled are those coming from the test apparatus and procedures, as opposed to those in the intrinsic operation of the CMDPSU. The principle sources of errors which have been identified in this study are random weighing errors and the run-position interactions, which have been assumed to be zero in the ANOVA. In this section, the theory behind the control and estimation of these errors will be described.

Position Effect Errors. In the statistical analysis of the precision measurement, the most serious errors arise from violations of the assumptions about position effects. In the first ANOVA (Table II-9), position effects are assumed to be negligible. In the second ANOVA (Table II-10), position effects are taken into account, but only by neglecting interactions between POS and the chamber operation variables (T, EXP and RUN). If these assumptions are not valid in practice, then the estimate of CV_t will be artificially large.

There are many empirical methods to test for position effects, using some method of measurement other than a CMDPSU. For example, we used a light-scattering dust monitor, as discussed in Part IV.A. However, any other dust measurement will have its own random errors, making the results difficult to interpret.

Therefore, the most elegant test for position effects is to simply use the experimental design for position effects (Table II-8). In the ANOVA (Table II-10), the position effects are contained only in EMS(POS). Therefore, the significance of the position effects can be determined by using the ANOVA results in an F-test (Snedecor and Cochran, 1967, p. 265):

$$F = MS(POS) / MS(POS*EXP \text{ in } T) \qquad \text{(II-32)}$$

If the probability of this F ratio with 3 and 6 d.o.f. is less than 0.05, then position effects are significant. Thus, the experimental design in Table II-7 cannot be used.

In the chambers with demonstrated position effects, we have found that the magnitude of position effects may be a function of the mean dust concentration during the run. Thus, there may exist significant run- position interactions (POS*T, POS*EXP and POS*RUN), which are variables depending on both the chamber position and the chamber operation during a run.

The most convenient manner to check for run-position interactions is to plot the residuals from the ANOVA (Figure II-6). In this example, the ANOVA residuals corrected for position effects are plotted against position. The residuals for position 1 range between ±0.009, while the residuals for position 2 range between ±0.018. Although not conclusive proof of run-position interaction, this 100 percent difference in the residuals between positions led to changes in the chamber's air flows, and a subsequent lowering of the CV_t estimate. Similar residual plots should be made with regard to the other major variables, especially the target concentration, and a qualitative check made for interactions.

To test for such interactions quantitatively, the interaction terms can be written into the ANOVA model (Eq. II-27). The POS*T interaction is treated as a fixed effect with mean square term $\theta(TP)$. POS*EXP and POS*RUN interactions are treated as random effects with variances $(\sigma_{PE})^2$ and $(\sigma_{PR})^2$ respectively. Then, the EMS terms in Table II-10 are modified:

$$EMS(POS*T) = 4\,\theta(PT) + 2(\sigma_{PE})^2 + 2(\sigma_S)^2 + (\sigma_\varepsilon)^2 \qquad (II\text{-}33a)$$

$$EMS(POS*EXP \text{ in } T) = 2(\sigma_{PE})^2 + 2(\sigma_S)^2 + (\sigma_\varepsilon)^2 \qquad (II\text{-}33b)$$

$$EMS(Error) = (\sigma_\varepsilon)^2 + (\sigma_{PR})^2 \qquad (II\text{-}33c)$$

The existence of the POS*T interaction can now be tested by the F-ratio:

$$F = MS(POS*T) \,/\, MS(POS*EXP \text{ in } T) \qquad (II\text{-}34)$$

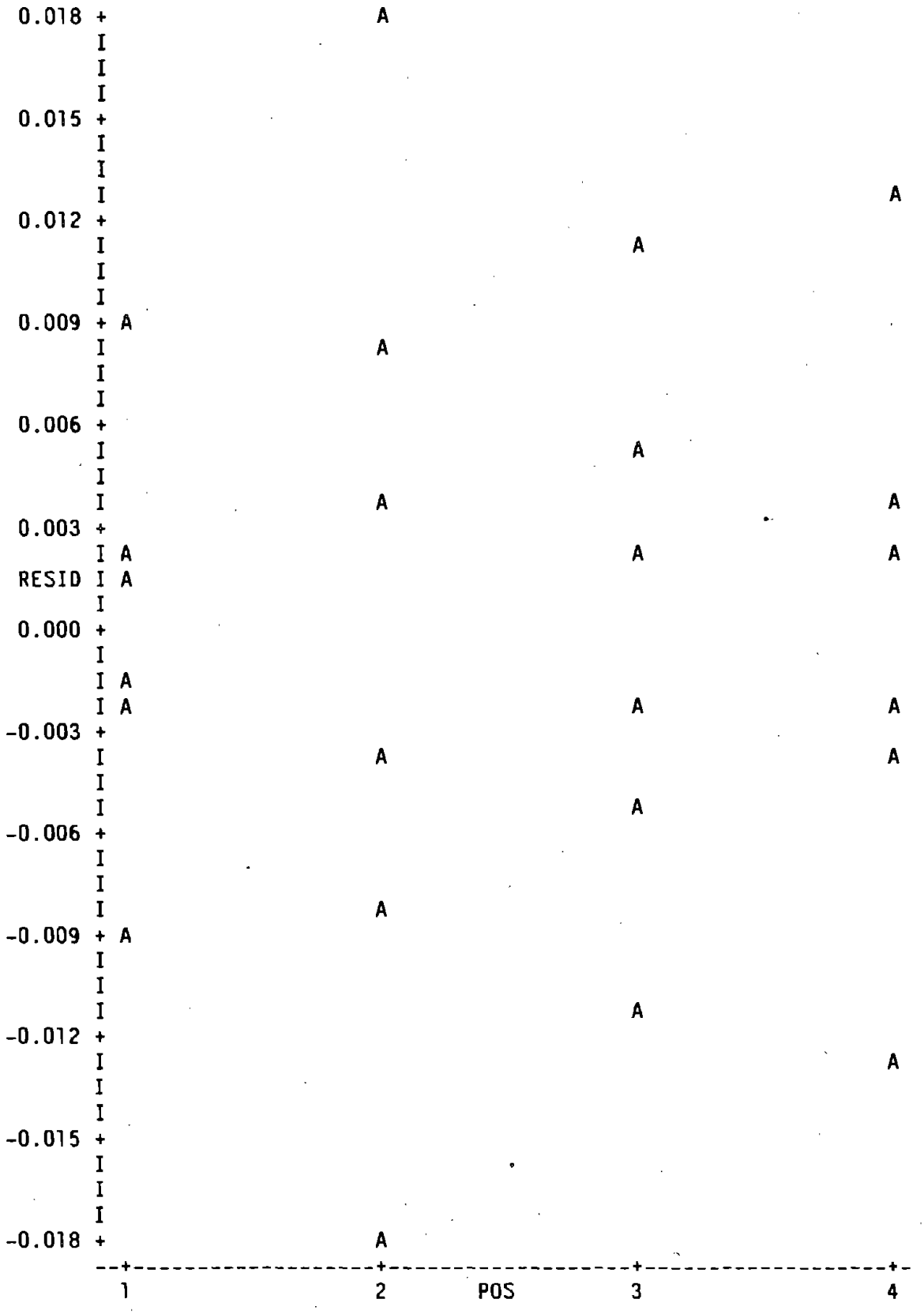
If the probability of the F-value with 3 and 6 d.o.f. is less than 0.05, then $\theta(PT)$ cannot be neglected, and the pooling step (Eq. II-28) is invalid. In this case, a precision estimate can still be obtained from:

$$(\hat{CV}_t)^2 = [MS(POS*EXP \text{ in } T) + MS(Error)] \,/\, 2 \qquad (II\text{-}35)$$

with f_* ranging from 6 to 18.

The $\hat{CV}_t$ estimate in Eq. II-35 would still contain the interaction terms $(\sigma_{PE})^2$ and $(\sigma_{PR})^2$, if they exist. The easiest test for these interactions is residual plots, such as Figure II-6. Since these interaction terms are difficult to detect and cannot be eliminated from the precision estimate without a much more extensive experimental design, every effort should be made to eliminate position effects from the dust chamber.

Figure II-6. A qualitative check for run-postion interaction by plotting ANOVA residuals (RESID) vs. sampler position (POS).



Weighing Errors. Errors in weighing the filters likewise inflate the CV_t estimate. Control of these errors has been accomplished by a consistent Quality Control program. The QC data also allows the weighing errors to be estimated, following a formalism developed by Parobeck, Tomb, Ku and Cameron (1981).

The routine filter weighings in the precision test procedure (Section III.A.) are always accompanied by weighing a standard weight and a blank, and by replicate weighings with a second operator. Both the blank and the standard weight go through 4 weighings during one run of the precision test:

	Initial	Final
Measurement	M_{I1}	M_{F1}
Quality Control	M_{I2}	M_{F2}

The initial and final weighings may occur on different days, so that the variation in blank weights contain both inter-day and intra-day variation.

From Parobeck et al. (1981), the model for such random errors is:

$$\hat{M}_{ij} = M_{true} + \delta_i + \varepsilon_{ij} \qquad \text{(II-36)}$$

where δ_i = inter-day error for day i

ε_{ij} = intra-day error for weighing j.

The inter-day errors have a constant variance $var(\delta) = (\sigma_\delta)^2$; the intra-day errors have $var(\varepsilon) = (\sigma_\varepsilon)^2$.

In routine dust sampling, the dust mass M_S is estimated from the difference between the final and initial filter weights:

$$\hat{M}_S = \hat{M}_F - \hat{M}_I$$

$$= M_S + \delta_F - \delta_I + \varepsilon_{F1} - \varepsilon_{I1} \qquad \text{(II-37)}$$

The variance of sums is given by a well-known theorem (e.g. Snedecor and Cochran, p. 190, 1967):

$$var(X_1 + X_2) = var(X_1) + var(X_2) + 2\,cov(X_1, X_2) \qquad \text{(II-38)}$$

Assuming that the inter-day and intra-day errors are uncorrelated, Eq. II-37 and II-38 give:

$$var(\hat{M}_S) = (\sigma_M)^2 = 2\,[(\sigma_\delta)^2 + (\sigma_\varepsilon)^2] \qquad \text{(II-39)}$$

This variance in the dust weight can be estimated from the quantities in the control charts described in Section III.A. From the replicate weighings on a single day, the difference d is calculated:

$$d = \hat{M}_{i2} - \hat{M}_{i1} = \varepsilon_{i2} - \varepsilon_{i1} \qquad \text{(II-40)}$$

Alternatively, we can use the range $R = /d/$. For 2 samples, the range is related to the standard deviation:

$$S_\varepsilon = \sqrt{d^2/2} = \sqrt{R^2/2} \qquad \text{(II-41)}$$

with only one degree of freedom.

Therefore, S_ε is an estimator for σ_ε. To improve the confidence limits in this estimate, the $S_{\varepsilon j}$ from all replicate weighting $j = 1,2....N$ are pooled:

$$(S_{pool})^2 = N^{-1} \sum_i (S_{\varepsilon j})^2 \qquad \text{(II-42)}$$

S_{pool} is our best estimate of the intra-day variation.

The inter-day variation can be estimated from the repeated blank weights, obtained at the final and initial sessions. This is expressed as a difference between the two means:

$$\Delta = (\hat{M}_{F1} + \hat{M}_{F2}) / 2 - (\hat{M}_{I1} + \hat{M}_{I2}) / 2 \qquad \text{(II-43)}$$

$$= \delta_F - \delta_I + (\varepsilon_{F1} + \varepsilon_{F2}) / 2 - (\varepsilon_{I1} + \varepsilon_{I2}) / 2$$

Taking the variance over all blank filters gives:

$$(S_\Delta)^2 \cong 2(\sigma_\delta)^2 + (\sigma_\varepsilon)^2 \qquad \text{(II-44)}$$

(again using Eq. II-38).

Using Satterthwaite's approximation again, the total variance for the weighing errors (Eq. II-39) can be estimated by:

$$(\sigma_M)^2 \cong (S_{pool})^2 + (S_\Delta)^2 = (S_M)^2 \qquad \text{(II-45)}$$

This expression is the recommended method for estimating the weighing errors. An example of this estimate is given in Section IV A.

To establish a Quality Control system based on these quantities, control limits have to be calculated for the quantities R and Δ. Following Parobeck et al. (1981), the control limits on R at the 99.5 percent confidence level are given by:

$$UCL_R = 3.97\ S_{pool} \qquad \text{(II-46)}$$

Likewise, the three-sigma control limits on Δ are $\pm 3\ S_\Delta$.

Finally, we note that additional Quality Control measures should be developed for the system of certifying CMDPSUs. In particular, a set of control CMDPSUs should be run through the set of certification tests periodically, and the results recorded on a control chart. This QC measure would be the most direct method for controlling the overall quality of the system.

PROPAGATION OF ERRORS

The weighing errors have an important effect of the certification tests because they are the creation of the testing lab, rather than the CMDPSUs being tested. To assess this effect, the contribution of the weighing errors to CV_t can be determined quantitatively by a propagation of errors. As in the Statistical Protocol for the NIOSH validation tests, CV_t can be decomposed into independent analytical errors CV_A and sampling errors CV_S:

$$(CV_t)^2 = (CV_A)^2 + (CV_S)^2 \qquad \text{(II-47)}$$

CV_A in respirable dust sampling is due entirely to the weighing, and is the creation of the certification lab during the precision measurement. CV_S is due to both the CMDPSU and the sampling procedure. In the precision test, CV_S is the quantity of interest, and CV_A should be relatively small.

To estimate CV_A, consider the formula for the calculation of the respirable dust concentration:

$$C = k\, M_S \,/\, Q_0\, T \qquad \text{(II-48)}$$

where k is the calibration constant for respirable dust, Q_0 is the pump flow rate specified for the CMDPSU and T is the sampling time. Following MSHA procedures, k and Q_0 are assumed, rather than measured, in the precision test. (Any errors due to k and Q_0 constitute a bias in the present sampling system, and are beyond the scope of this discussion.) However, M_S and T are measured during the precision test and are thus susceptible to random error with standard deviations σ_M and σ_T.

For a function of several variables $w = f(x, y \ldots)$, the basic theorem for the propagation of errors from x, y ... to a measurement of w is derived by Ku (1966):

$$\text{var}(W) \cong [\partial f/\partial x]^2 (s_x)^2 + [\partial f/\partial y]^2 (s_y)^2 + 2\,[\partial f/\partial x]\,[\partial f/\partial y]\,(s_{xy})^2 + \ldots \qquad \text{(II-49)}$$

where the quantities in brackets are evaluated at the means $\bar{x}, \bar{y}$ . . .

In applying this theorem to Eq. II-49, we first observe that the covariance S_{MT} was assumed to be zero because M_S and T are measured independently. Then, we get:

$$\text{var}(C) / \bar{C}^2 = (s_M / \bar{M_S})^2 + (s_T / \bar{T})^2 \qquad \text{(II-50)}$$

At this point, the term $\text{var}(C) / \bar{C}^2$ can be identified as the analytical error $(CV_A)^2$. Further, the sampling time T can be measured to within 1-2 seconds over a period of at least 6 hours, giving a relative standard deviation for the time measurement on the order of 0.01 percent, which can be safely ignored. Thus, the final estimate for the analytical error is:

$$CV_A \cong S_M / \bar{M_S} \qquad \text{(II-51)}$$

where S_M is estimated from Eq. II-45 above.

At this point, Eq. II-51 will be used to demonstrate why the manufacturer's preweights, truncated to 0.1 mg, are not adequate for the precision test. Parobeck et al. (1981) report that s_M = 0.0807 mg in the routine operation of MSHA's sampling program with filters weighed to 0.1 mg. The analytical error from using the manufacturer's weights in the precision test would be at best:

Target Concentration	Sampling Time	M_S	CV_A (percent)
1.0 mg/m^3	8 hours	0.696 mg	11.6
4.0	8	2.783	2.9

At the low concentration, CV_t would therefore be dominated by the weighing error, rather than by CMDPSU performance. For this reason, we have weighed the filters to 0.001 mg in these trials, and consideration should be given to weighing filters to at least 0.01 mg for routine certification tests.

D. CALIBRATED SAMPLING EFFICIENCY

The proposed certification accuracy test described in detail below gives a measure of the sampling unit's efficiency in sampling particles of a given size. This information is conveniently represented as a calibrated sampling efficiency $\theta_S(D)$ in terms of particle aerodynamic diameter D. This function is defined as follows.

The sampled respirable mass concentration C_S is calculated from the mass M_S sampled by the filter by:

$$C_S = kM_S/(Q_oT_o). \tag{II-52}$$

In this equation, MSHA has set the sampling time T_o equal to 8 hours (a full shift); the target flow rate Q_o and calibration constant k are to be specified by the CMDPSU manufacturer. As discussed above, MSHA neglects any random errors in the flow rate and sampling time in routine calculation of C_S from mine operators' samples. Present practice involving cyclone sampling calls for k=1.38 and Q_o = 2.0 L/min for estimating BMRC respirable dust concentration C_{BMRC}.

The quantity C_{BMRC} is given by

$$C_{BMRC} = (Q_oT_o)^{-1}\int_0^\infty dD(dM/dD)\,\theta_{BMRC}(D) \tag{II-53}$$

where dM/dD is the total mass per diameter range entering the sampler during T_o, and $\theta_{BMRC}(D)$ is the BMRC respirable dust weighting function defined as

$$\theta = 1 - (D/7.1\ \mu m)^2 \tag{II-54}$$

The ACGIH respirable dust criterion is defined pointwise as:

D (μm)	2.00	2.50	3.50	5.00	10.0
θ_{ACGIH}	0.90	0.75	0.50	0.25	0.00.

(II-55)

Least-squares fit to these five points gives the polynomial,

$$\theta_{ACGIH} = \begin{cases} 0.9, & D \leq 2\ \mu m \\ 1.6442 - 0.4189\,D + 0.01776\,D^2 \\ \quad + 0.003333\,D^3 - 0.0002564\,D^4, & 2\ \mu m \leq D \leq 10\ \mu m \\ 0.0, & D \geq 10.0\ \mu m \end{cases} \tag{II-56}$$

(Another respirable dust weighting, $\theta_{LASL-AEC}$ is identical to θ_{ACGIH} aside from $D \leq 2.0$ μm, where $\theta_{LASL-AEC} = 1.00$ (Caplan, et al., 1973).)

The bias test is intended to provide an estimate of the ratio $M_S(D)/M(D)$ of the mass $M_S(D)$ reaching the filter to the total mass $M(D)$ entering the sampling unit for particles of diameter D. Defining the calibrated sampling efficiency $\theta_S(D)$ as

$$\theta_S(D) = kM_S(D)/M(D), \tag{II-57}$$

the sampled respirable mass concentration C_S is given by

$$C_S = (Q_o T_o)^{-1} \int_o^\infty dD(dM/dD)\theta_S(D) \tag{II-58}$$

in exact analogy with Equation II-53.

The quantity $\theta_S(D)$ is useful because it allows direct comparison to $\theta_{BMRC}(D)$. As $\theta_S(D)$ and $\theta_{BMRC}(D)$ will generally differ for most diameters, there is a bias Δ (non-zero except for isolated dust distributions) between the sampled C_S and true C_R respirable mass concentrations:

$$\Delta = (C_S - C_R)/C_R$$

$$= \int_o^\infty dD(dM/dD)[\theta_S(D) - \theta_R(D)] / \int_o^\infty dD(dM/dD)\theta_R(D). \tag{II-59}$$

E. CYCLONE PARAMETER ESTIMATION

As mentioned above, certification laboratory experience with the 10 mm cyclone will be necessary for assessing the uncertainty in the laboratory's bias test for both cyclone as well as more general sampling units. Therefore, the present section is devoted exclusively to analysis of the cyclone. To this end, a mathematical model of the cyclone collection efficiency is described. In terms of the model parameters, expressions for the collected mass and uncertainty in various quantities of interest are then given for arbitrary log-normally distributed dust.

At the present time there is no quantitative physical model which adequately describes cyclone collection efficiency η defined as $\eta = (1-e_S(D))$. (Throughout this section, expressions such as η or e_S represent the physical, rather than "calibrated," collection or penetration efficiency.) However, several empirical models have appeared in literature. In Blachmann and Lippmann (1974) the collection efficiency is represented by the hyperbolic tangent of a second degree polynomial in the aerodynamic diameter. An alternative model, introduced by Held and Cooper (1979), is adopted here for the bulk of the calculation, whereas the hyperbolic tangent model is used only to a limited degree, namely for indicating that derived results are model independent. At aerodynamic diameter D, the quantity η is represented by the error function (erf):

$$\eta(D) = \int_0^D dD'(\sigma_c D')^{-1}(2\pi)^{-1/2}\exp[-0.5\ (\ln D'-\mu_c)^2/(\sigma_c)^2] \qquad \text{(II-60a)}$$

$$= 0.5 + 0.5\ \mathrm{erf}[(\ln D-\mu_c)/((2)^{1/2}\sigma_c)]. \qquad \text{(II-60)}$$

The parameters μ_c and σ_c characterize the cyclone. The quantity μ_c gives the cut-off diameter D_{cut} where $e_S(D)$ is half-maximum by:

$$D_{cut} = \exp(\mu_c). \qquad \text{(II-61)}$$

Similarly, σ_c is related to the sharpness of the cut since

$$D\ \partial\eta/\partial D = (\sigma_c)^{-1}(2\pi)^{-1/2} \quad (D = D_{cut}). \qquad \text{(II-62)}$$

The total mass M entering the cyclone is composed of botn mass M_C collected by the cyclone and the mass M_S sampled by the filter:

$$M = M_C + M_S. \qquad \text{(II-63)}$$

Using the above expression for η and the log-normal distribution for dM/dD at μ and σ, the collected mass M_C is given by:

$$M_C = \int_0^{\infty} dD\,(dM/dD)\eta(D) \qquad \text{(II-64a)}$$

$$= M \int_{-\infty}^{+\infty} dz'(\pi)^{-1/2}\exp(-z'^2) \int_{-\infty}^{\left[\sigma z' + (\mu-\mu_c)/\sqrt{2}\right]/\sigma_c} dz(\pi)^{-1/2}\exp(-z^2). \qquad \text{(II-64b)}$$

This integral may be evaluated by using the fact that the integrand is invariant under rotations of the (z,z')-coordinate system. Thus, since the line $z = [\sigma z' + (\mu-\mu_C)(2)^{-1/2}]/\sigma_C$ lies at a distance of $(\mu-\mu_C)/[2(\sigma^2+(\sigma_C)^2)]^{1/2}$ from the origin, there is a suitable rotation which brings the integral into the form

$$M_C/M = \int_{-\infty}^{(\mu-\mu_c)/\left[2\left(\sigma^2+\sigma_c^2\right)\right]^{1/2}} dz(\pi)^{-1/2}\exp(-z^2) \qquad \text{(II-65a)}$$

$$= 0.5 + 0.5\ \mathrm{erf}[(\mu-\mu_C)/(2(\sigma^2+(\sigma_C)^2))^{1/2}]. \qquad \text{(II-65b)}$$

Equation II-63 therefore gives the sampled mass in the form,

$$M_S = \int_0^{\infty} dD\,(dM/dD)e_S(d) \qquad \text{(II-66a)}$$

$$M_S/M = 0.5 - \int_0^{(\mu-\mu_c)/\left[2\left(\sigma^2+\sigma_c^2\right)\right]^{1/2}} dz(\pi)^{-1/2}\exp(-z^2) \qquad \text{(II-66b)}$$

$$= 0.5 - 0.5\ \mathrm{erf}[(\mu-\mu_C)/(2(\sigma^2+(\sigma_C)^2))^{1/2}]. \qquad \text{(II-66c)}$$

It must be emphasized at this point that the present use of log-normal size distributions is partly for mathematical convenience. The corresponding bias estimates are not, in fact, limited to the sampling of only log-normally distributed dust. Since any dust distribution can be approximated by a linear superposition of log-normal distributions, the bias also can be written as a function of the biases for log-normal distributions. For the bimodal distribution (Eq. II-11) found in coal mines, the percentage bias for the composite distribution can be shown to be bounded by the biases for the two individual distributions.

Uncertainties in various quantities of interest can now be estimated. For this purpose, the empirical model described above for the cyclone collection efficiency is assumed. The model, together with an hypothesis as to error distribution, leads to variance estimates for model parameters. The variance estimates, in turn, appear in expressions for such quantities as the variance of D_{cut} and the confidence limits on the bias between respirable and sampled mass for log-normally distributed aerosols.

As outlined above, the cyclone model parameters are estimated by linear

regression on transformed collection efficiency data. Explicitly, Equation II-50 may be expressed in the form

$$Y = \text{erf}^{-1}(2\eta-1) \cdot$$

$$= (\ln D-\mu_C)/[(2)^{1/2}\sigma_C]. \qquad \text{(II-67)}$$

After transforming measurements of η into Y using Equation II-67, linear regression in terms of $\ln D$ can be carried out. The result is an estimate $\hat{Y}$ for $\text{erf}^{-1}(2\eta-1)$ in the form

$$\hat{Y} = \bar{Y} + b(\ln D-\overline{\ln D}), \qquad \text{(II-68)}$$

where b is given by the usual regression formula,

$$b = \sum_i (\ln D_i-\overline{\ln D})(Y_i-\bar{Y}) \; / \; \sum_i (\ln D_i-\overline{\ln D})^2. \qquad \text{(II-69)}$$

In Equation II-69, the summation index i runs over the data points. As in Draper and Smith (1968) bars represent data averages and "hats" signify parameter estimates. Comparison of Equations II-67 and II-68 gives estimates for μ_C and σ_C in the form

$$\hat{\sigma}_C = (2)^{-1/2}/b \qquad \text{(II-70a)}$$

$$\hat{\mu}_C = \overline{\ln D} - \bar{Y}/b. \qquad \text{(II-70b)}$$

As mentioned above, in order to calculate parameter estimate variances, an assumption must be made as to the dominant error distribution. As in Draper and Smith (1968), the exact Y is assumed to obey

$$Y_i = \alpha + \beta \ln D_i + \varepsilon_i, \qquad \text{(II-71)}$$

where the (error free) parameters α and β are estimated as above, and ε_i is a normally distributed random variable with mean zero and (unknown) variance $(\sigma_Y)^2$. Note that the effect of errors in D_i are neglected here. Analysis along the lines of Snedecor and Cochrane, 1967, indicates that the bias in coefficient estimates due to such errors is less than 1 percent if the standard error in D_i is less than 6 percent, given the range of measured diameters. Within this assumption it is possible to show (Draper and Smith, 1968) that $\bar{Y}$ and b are uncorrelated and that their variances are given by:

$$\text{var}(\bar{Y}) = (\sigma_Y)^2/n \qquad \text{(II-72a)}$$

$$\text{var}(b) = (\sigma_Y)^2/\sum_i (\ln D_i-\overline{\ln D})^2, \qquad \text{(II-72b)}$$

where n is the number of degrees of freedom (data points).

Furthermore, $(\sigma_Y)^2$ can be estimated by $(s_Y)^2$ given by:

$$(s_Y)^2 = (n-2)^{-1} \sum_i (Y_i - \hat{Y}_i)^2. \qquad \text{(II-73)}$$

Note that $(\sigma_Y)^2$ represents the variance in the transformed data points, rather than the variance $(\sigma_\eta)^2$ in the collection efficiency itself. As $(\sigma_Y)^2$ is given approximately in terms of $(\sigma_\eta)^2$ by

$$(\sigma_Y)^2 = (\partial \mathrm{erf}^{-1}(2\eta-1)/\partial\eta)^2(\sigma_\eta)^2, \qquad \text{(II-74)}$$

an explicit expression for σ_η is available in the form,

$$\sigma_\eta = (\pi)^{-1/2}\sigma_Y \exp[-(\mathrm{erf}^{-1}(2\eta-1))^2]. \qquad \text{(II-75)}$$

Equation II-75 implies that σ_η is largest when erf^{-1} vanishes; i.e., at the cut size ($\eta = 1/2$). Thus, Equation II-75 expresses the intuitive idea that errors in collection (or transmission) efficiency measurement will be largest at particle sizes corresponding to the steepest slope in the transmission curve. Experimental fluctuations in such quantities as sampling flow rate or particle size measurement yield errors of this type.

Since Y and b are uncorrelated, it is a simple matter to estimate $\mathrm{var}(\hat{\sigma}_C)$ and $\mathrm{var}(\hat{\mu}_C)$, given the estimates of Equations II-72 for $\mathrm{var}(\bar{Y})$ and $\mathrm{var}(b)$.

$$\mathrm{var}(\hat{\sigma}_C) = (\partial\hat{\sigma}_C/\partial b)^2 \mathrm{var}(b) \qquad \text{(II-76a)}$$

$$= 2(\hat{\sigma}_C)^4 \mathrm{var}(b). \qquad \text{(II-76b)}$$

$$\mathrm{var}(\mu_C) = (\partial\hat{\mu}_C/\partial b)^2 \mathrm{var}(b) + (\partial\hat{\mu}_C/\partial\bar{Y})^2 \mathrm{var}(\bar{Y}) \qquad \text{(II-77a)}$$

$$= 2(\hat{\sigma}_C)^2[(\overline{\ln D} - \hat{\mu}_C)^2 \mathrm{var}(b) + \mathrm{var}(\bar{Y})]. \qquad \text{(II-77b)}$$

Furthermore, as D_{cut} may be estimated by

$$\hat{D}_{cut} = \exp(\hat{\mu}_C), \qquad \text{(II-78)}$$

the estimated variance in D_{cut} is given by

$$\mathrm{var}(\hat{D}_{cut}) = 2(D_{cut}\hat{\sigma}_C)^2[(\overline{\ln D} - \hat{\mu}_C)^2 \mathrm{var}(b) + \mathrm{var}(\bar{Y})]. \qquad \text{(II-79)}$$

The confidence limits for the estimated sampled mass M_S can now be determined.

$$\mathrm{var}(\hat{M}_S) = (\partial\hat{M}_S/\partial\bar{Y})^2 \mathrm{var}(\bar{Y}) + (\partial\hat{M}_S/\partial b)^2 \mathrm{var}(b). \qquad \text{(II-80)}$$

The derivatives appearing in Equation II-80 are estimable from

Equation II-66c by

$$\partial\hat{M}_S/\partial\bar{Y} = (\partial\hat{M}_S/\partial\hat{\mu}_c)(\partial\hat{\mu}_c/\partial\bar{Y}) \qquad \text{(II-81a)}$$

$$\partial\hat{M}_S/\partial b = (\partial\hat{M}_S/\partial\hat{\mu}_c)(\partial\hat{\mu}_c/\partial b) + (\partial\hat{M}_S/\partial\hat{\sigma}_c)(\partial\hat{\sigma}_c/\partial b). \qquad \text{(II-81b)}$$

The result is that

$$\partial\hat{M}_S/\partial\bar{Y} = -M\hat{\sigma}_c[\pi(\sigma^2+(\sigma_c)^2)]^{-1/2}\exp[-0.5\ (\mu-\hat{\mu}_c)^2/(\sigma^2+(\sigma_c)^2)] \qquad \text{(II-82a)}$$

$$\partial\hat{M}_S/\partial b = (\partial\hat{M}_S/\partial\bar{Y})[\hat{\mu}_c-\overline{\ln D}+(\hat{\sigma}_c)^2(\mu-\hat{\mu}_c)/(\sigma^2+(\sigma_c)^2)]. \qquad \text{(II-82b)}$$

Finally, using the estimated variance $(s_Y)^2$ for $(\sigma_Y)^2$ from Equation II-74, it is possible to specify confidence limits using a value of t. The 100 $(1-\alpha)$ percent (two-sided) confidence limits for M_S are given by

$$\hat{M}_S \pm t(n-2,1-\alpha/2)\mathrm{var}^{1/2}(\hat{M}_S)s_Y/\sigma_Y. \qquad \text{(II-83)}$$

This derivation is concluded with an illustrative calculation using the above formalism. For this purpose the monodisperse aerosol data of Caplan, et al. (1973) for Q = 1.7 L/min (no fluctuation) are used. The calibration coefficient is taken to equal unity for use with the ACGIH definition of respirable dust. For calculation of the bias, a dust distribution with MMD = 8.0 μm and GSD = 2.5 is used. In Table II-11 are listed the cyclone collection efficiency data, regression results, and confidence limit calculation.

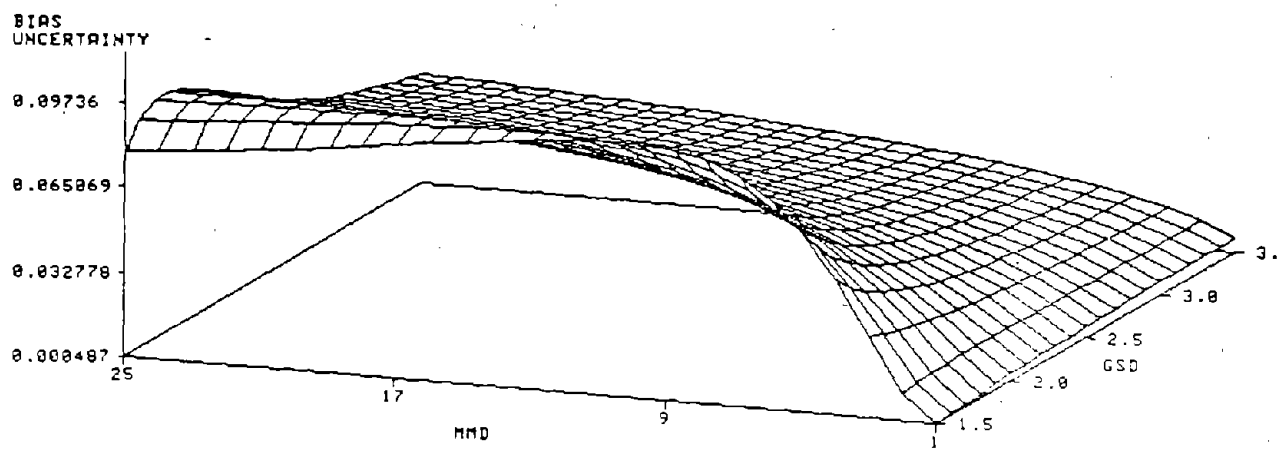
The results given in Table II-11 are limited to a single dust distribution. Computation for other distributions is easily accomplished using Equations II-80, II-82 and II-83. The result is shown in Figure II-7 where the ACGIH bias uncertainty (95 percent confidence level) is plotted for a variety of dust distributions.

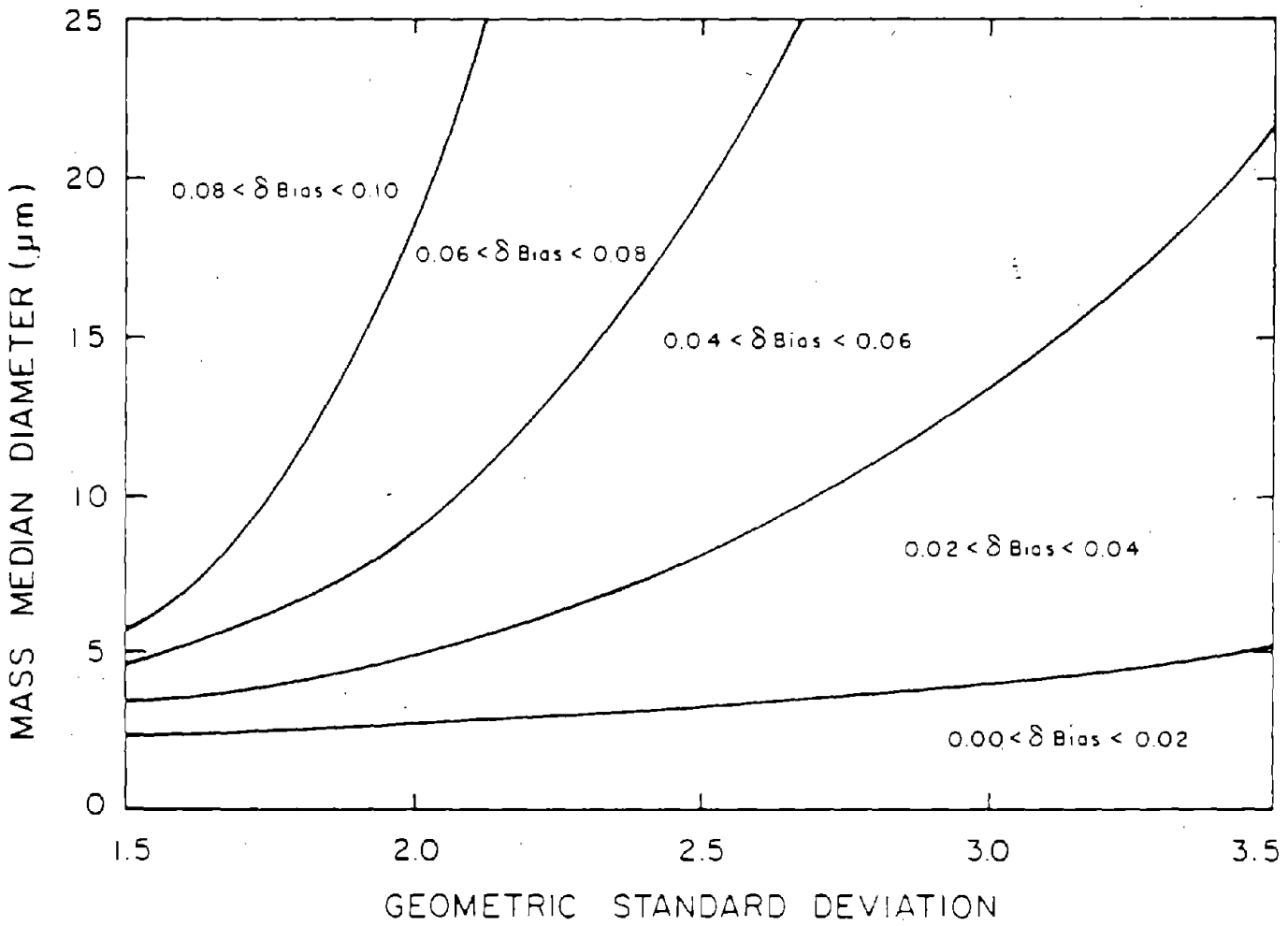
Several observations can be made concerning these results. An important point which should be brought out concerns the applicability of the hypothesis of normal error distribution. Examination of the residuals $(Y - \hat{Y})$ for the CDS data presented in Table II-11 shows no obvious trends over the diameter range covered. Thus, there exists some evidence supporting the above hypothesis, although further studies may be required.

Note, on the other hand, that the magnitude of the residual at the largest particle size considered (D = 6.0 μm) is quite large. Whether this indicates significant skewness in the collection efficiency or simply experimental error is not known. A skewed effect could conceivably accompany significant particle re-entrainment. Again, further experimentation would help settle this question.

A second point to be mentioned concerns the number n of monodisperse aerosol data points. As t(n-2, 0.975) decreases only slowly for n larger than 8,

Figure II-7. Bias uncertainty in estimating ACGIH respirable dust concentration by cyclone sampling at 1.7 L/min.





many more degrees of freedom beyond the 8 in data of Caplan, et al. (1973) would be required to significantly sharpen the confidence limits on the bias. On the other hand t(n-2, 0.975) rapidly increases with decreasing n. For example, were n chosen to equal 3, where t(n-2, 0.975) = 12.7, excessively broad confidence limits would result. Thus, evidently 8 degrees of freedom in collection efficiency measurements are satisfactory for the estimation of sampler bias. On this basis the certification accuracy test has been designed so as to include 8 measurements of the calibrated collection efficiency at diameters evenly spaced between 2.25 μm and 7.50 μm. These specific limits were selected in order to help ensure coverage of diameters over which the sampling unit's collection efficiency is expected to exhibit large variation.

TABLE II-11

Sample Analysis of Cyclone Data at Q = 1.7 L/min

D (μm)	ln D	$2\eta - 1$	$Y = \text{erf}^{-1}(2\eta-1)$	$\hat{Y}$
2.50	0.916	-0.96	-1.45	-1.427
3.00	1.10	-0.80	-0.91	-0.889
3.50	1.25	-0.44	-0.41	-0.434
4.00	1.39	-0.02	-0.02	-0.040
4.50	1.50	0.34	0.31	0.307
5.00	1.61	0.68	0.70	0.617
5.50	1.70	0.84	0.99	0.899
6.00	1.79	0.84	0.99	1.160

$\overline{\ln D} = 1.407$

Regression Results:

$\overline{Y}$	=	0.025	$(s_Y)^2$	=	0.0076
b	=	2.95	$\text{var}(\overline{Y})$	=	$(\sigma_Y)^2/8$
$\hat{\sigma}_c$	=	0.240	var(b)	=	$(\sigma_Y)^2/0.643$
$\hat{\mu}_c$	=	1.40	t(6,0.975)	=	2.45

For Dust Distribution, MMD = 8.0 μm and GSD = 2.5

$\hat{M}_S$ = 0.237 M $\qquad$ M_R = 0.209 M

$\partial\hat{M}_S/\partial\overline{Y}$ = -0.11 M

$\partial\hat{M}_S/\partial b$ = $-4.0 \cdot 10^3$ M

95 percent Confidence Limits on M_S: (0.237 ± 0.0084) M

Bias Upper Limit: 17 percent

F. BIAS COMPUTATIONS FOR GENERAL SAMPLING UNIT

As the analysis of the previous section is limited to the cyclone, considerable modification of the calculational methods is necessary for evaluating a more general sampling unit. This is simply because the above collection efficiency model will generally be inapplicable.

In order to calculate the sampled respirable mass M_S for log-normally distributed dust, the integral of Equation II-66 is evaluated numerically by interpolating between the 8 measured values of the calibrated collection efficiency $\Theta_S(D)$ using a cubic spline. At diameters less than or equal to the smallest experimental value (nominally, 2.25 μm), $\Theta_S(D)$ is constrained to be constant and continuous and is set equal to zero for diameters greater than or equal to 10 μm. The derivative $d\Theta_S/dD$ is set equal to zero at the smallest experimental diameter and at D = 10.0 μm. Trapezoidal integration with intervals equal to 0.01 μm is used to estimate the integral and gives a computational error of less than 0.1 percent.

The 95 percent confidence level values for the bias (Eq. II-83) are computed with $var(m_S)$ derived from the relationship:

$$var(M_S) = (\partial M_S/\partial TRANS)^2 var(D_{cut}). \qquad (II\text{-}84)$$

where TRANS is an incremental distance by which the collection efficiency curve is translated along the D-axis. The quantity $(\partial M_S/\partial TRANS)$ is estimated numerically from the change in the computed value of M_S upon translation of the interpolated collection efficiency curve by 0.1 μm. An estimate for $var(D_{cut})$ is taken from the collection efficiency measurements for cyclones.

In testing this calculational procedure (and the computer programs given in the Appendix), cyclone data of Caplan, et al. (1973) were used. The quantity $var(M_S)$ as calculated by Equation II-84 above was found to differ from the result given in the previous section on cyclone parameter estimation by approximately 5 percent.

G. CRITICAL STANDARD DEVIATION IN TERMS OF THE BIAS

A vital part of the CMDPSU certification scheme lies in demanding adherence to the present NIOSH accuracy criterion (Gunderson and Anderson (1980)) for the sampling of hazardous substances requiring that a sampling/analytical procedure maintain less than 25 percent error at the 95 percent confidence level. This criterion places limits on acceptable values of the bias Δ and relative standard deviation CV_t. As CV_t is defined in this document slightly differently from the above reference, the calculation of limiting CV_t is carried out in full here.

It is assumed that the dust sampling procedure gives measurements of the concentration C_S distributed with standard deviation σ about a mean value $\overline{C_S}$. The bias Δ with respect to the true respirable concentration C_R is defined as above by

$$\Delta = (\overline{C_S} - C_R)/C_R. \qquad \text{(II-85)}$$

Furthermore, the relative standard deviation CV_t is defined as

$$CV_t = \sigma/\overline{C_S}. \qquad \text{(II-86)}$$

(Compare with (Gunderson and Anderson, 1980) where C_R replaces $\overline{C_S}$.) Then for any given value of the bias the criterion of accuracy A (=0.25 in the case of the NIOSH protocol) at the 95 percent confidence level fixes the limiting value σ_{target} (or CV_{target}) as a solution of

$$0.95 = \int_{C_R(1-A)}^{C_R(1+A)} dx\ (2\pi)^{-1/2}(\sigma_{target})^{-1}\exp[-0.5\ (x-\overline{C_S})^2/(\sigma_{target})^2] \qquad \text{(II-87)}$$

which can be expressed in terms of the error function as

$$0.95 = 0.5\ \mathrm{erf}[(A-\Delta)(2)^{-1/2}(CV_{target})^{-1}/(1+\Delta)] + 0.5\ \mathrm{erf}[(A+\Delta)(2)^{-1/2}(CV_{target})^{-1}/(1+\Delta)]. \qquad \text{(II-88a)}$$

This equation can be solved iteratively using Newton's method as in the computer program given in the Appendix. For Δ near $\pm A$, however, convergence is poor. In this situation, an approximate solution is available in the form,

$$CV_{target} = (A-\mathrm{abs}(\Delta))/[2^{1/2}(1+\Delta)\mathrm{erf}^{-1}(0.90)]. \qquad \text{(II-88b)}$$

Unlike Gunderson and Anderson (1980), solutions CV_{target} are dependent on the sign of Δ (i.e., $CV_{target}(\Delta) \neq CV_{target}(-\Delta)$). Furthermore, the single-tailed, t-distributions are used to estimate 95 percent

confidence limits on the bias. Values for CV_{target} are then calculated by substituting both bias edges (limits) for Δ in Equation II-88a. The critical bias edge is then defined as that edge which gives the smaller CV_{target}. This value for CV_{target} is then compared with the upper (single-sided) 95 percent confidence level value CV_{upper}.

The quantity CV_{upper} is determined within the approximation (Hald, 1952) that the standard deviation σ_{CV} of the coefficient of variation CV_t estimated using f degrees of freedom is given approximately by

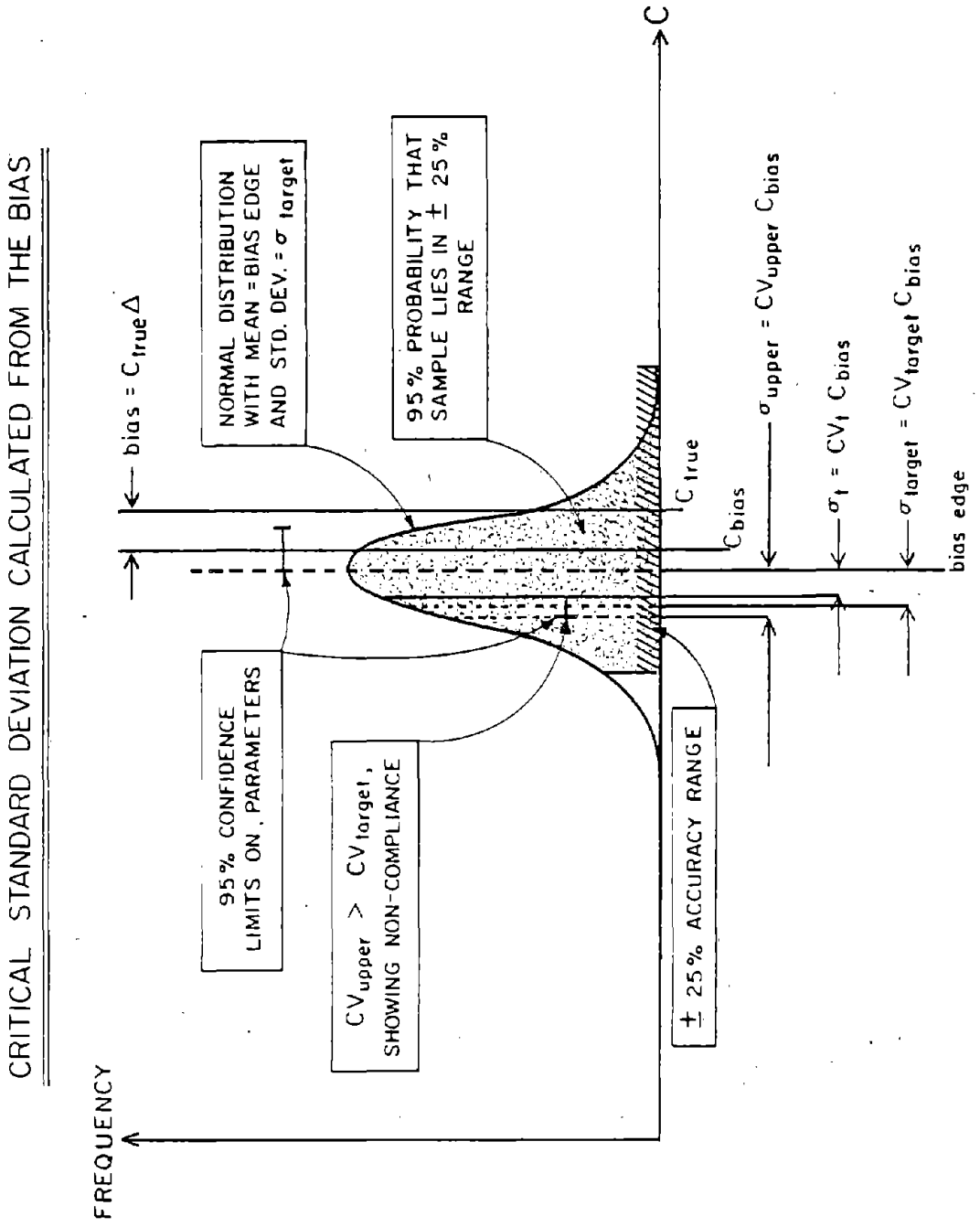
$$\sigma_{CV} \sim CV_t(2f)^{-1/2}. \qquad \text{(II-89)}$$

Then CV_{upper} is given in terms of the sample mean $\overline{CV}$ by

$$CV_{upper} = \overline{CV}[1 + f^{-1/2}\text{erf}^{-1}(0.90)]. \qquad \text{(II-90)}$$

For a pictorial impression of the numerous variables defined above, see Figure II-8.

Figure II-8. Critical Standard Deviation Calculated from the Bias



H. CALIBRATION IMPROVEMENT

As values for the estimated bias are determined for the candidate sampler over a variety of test dust distributions, calculation of an improved value for the calibration constant at the fixed flow rate under consideration is possible. Because of the manner of selecting the critical bias edge as described in the previous paragraphs, CV_{target} is not an analytic function of the calibration constant. Thus, Newton's method is not immediately applicable.

Instead, systematic search for an improved calibration constant is carried out as follows. During the initial compliance calculation, the two test dust distributions giving extreme values (maximum and minimum) of the bias edge are determined. The bias for each is then shifted by $\delta\Delta$ by changing the calibration constant by a factor F:

$$\delta\Delta = (F-1)(1+\Delta). \qquad \text{(II-91)}$$

Critical edges are determined for both dust distributions over a set of trial values F. The value $F_{optimal}$ is then found for which the smallest CV_{target} for the two distributions is maximum over the trial values of F.

III. EXPERIMENTAL PROCEDURES

The procedures in this section are recommended for the routine accuracy testing of CMDPSUs for the purposes of certification. These recommended test procedures have been developed after the trials of the accuracy tests reported in Section IV were analyzed and reviewed by NIOSH's Testing and Certification Branch. The differences in procedure and the reasons for the changes will be discussed in Section IV.

These procedures assume that the Federal coal mine dust sampling regulations will have been modified as recommended in Section V. Without the modifications in the sampling regulations, the accuracy test procedures recommended here could still be used, but would result in the presently certified CMDPSUs exceeding the ±25 percent accuracy limits set by NIOSH for the validation of other sampling and analytic methods.

A. PRECISION TEST

The purpose of this test is to measure CMDPSU precision, using procedures approximating those prescribed for use in coal mines. At this time, the coal mine dust sampling procedures in Federal regulations (30 CFR 70 and 71) are followed as closely as would be possible in a laboratory routinely testing CMDPSUs for certification. In testing novel samplers, the manufacturers instructions would be followed in sampler operation.

The dust chamber and the experimental procedures are designed for personal samplers only. Area or machine mounted samplers would need a larger chamber and possibly a different experimental design.

APPARATUS:

1. Sampler Calibration System

   a. Coal dust chamber. In the present study, the chamber (Figures III-1 thru III-3) was adapted from the Standard System of Fairchild, Tillery and Ettinger (1977). This chamber had significant position effects. The design of Rubow and Marple (1983), on the other hand, does not have significant concentration differences due to the effects of sampler position.

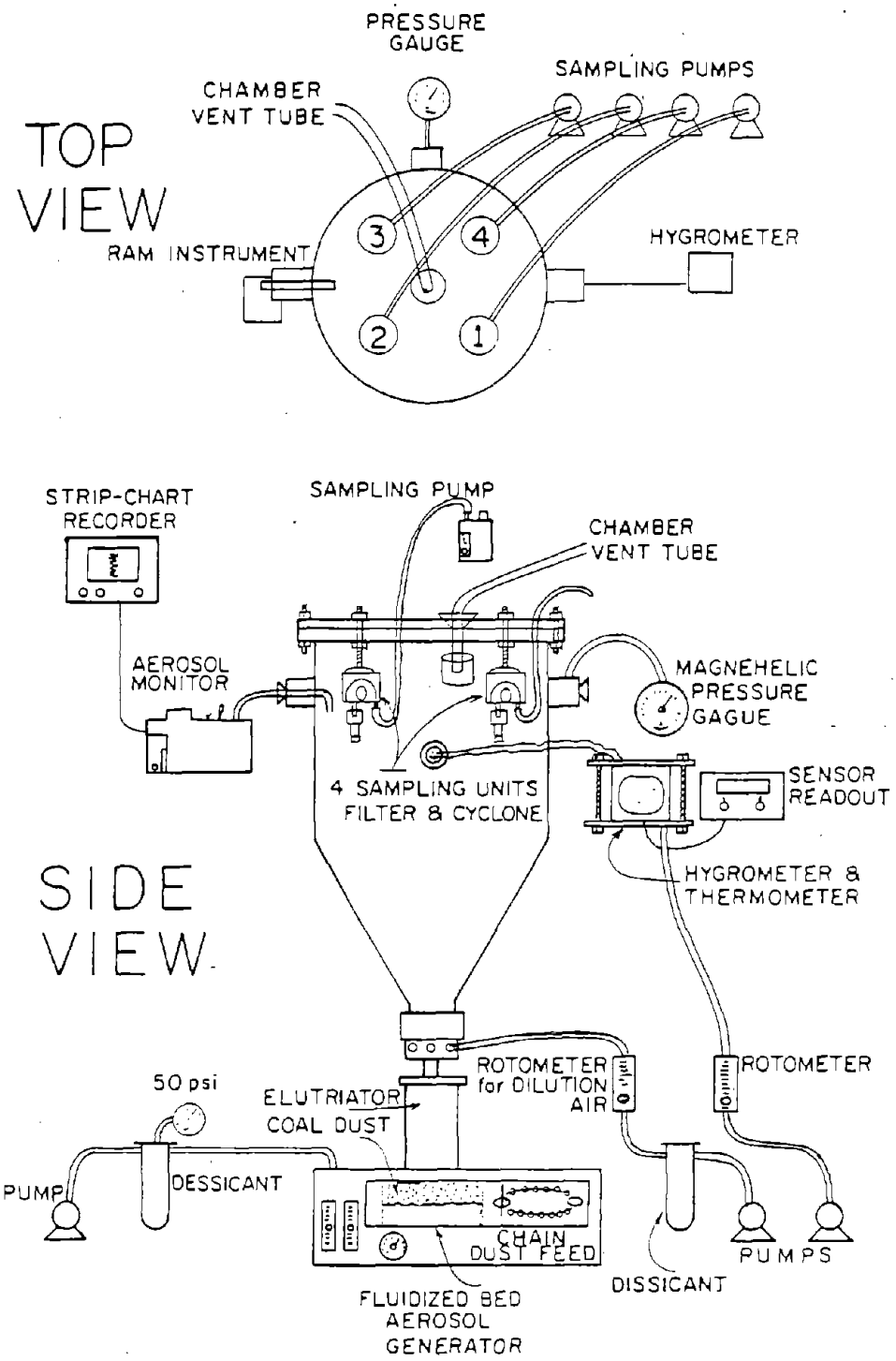
   b. TSI Fluidized Bed Aerosol Generator (Model 3400). To give a broader range of aerosol sizes, the cylcone should be removed from the elutriator.

   c. Charge neutralizer.

   d. Source of clean, dry air.

   e. Coal dust whose MMD and GSD lie within the shaded area in Figure II-5. For preparation of the dust, see the comments by Fairchild, Tillery and Ettinger (1977).

Figure III-1: Dust Chamber for the Precision Test



Precision Test Apparatus

Figure III-2: Precision test chamber, showing (top to bottom) the hygrometer, strip chart recorder for the RAM, sampling pumps, dust chamber, Respirable Aerosol Monitor, air flow controls, and fluidized bed aerosol generator.

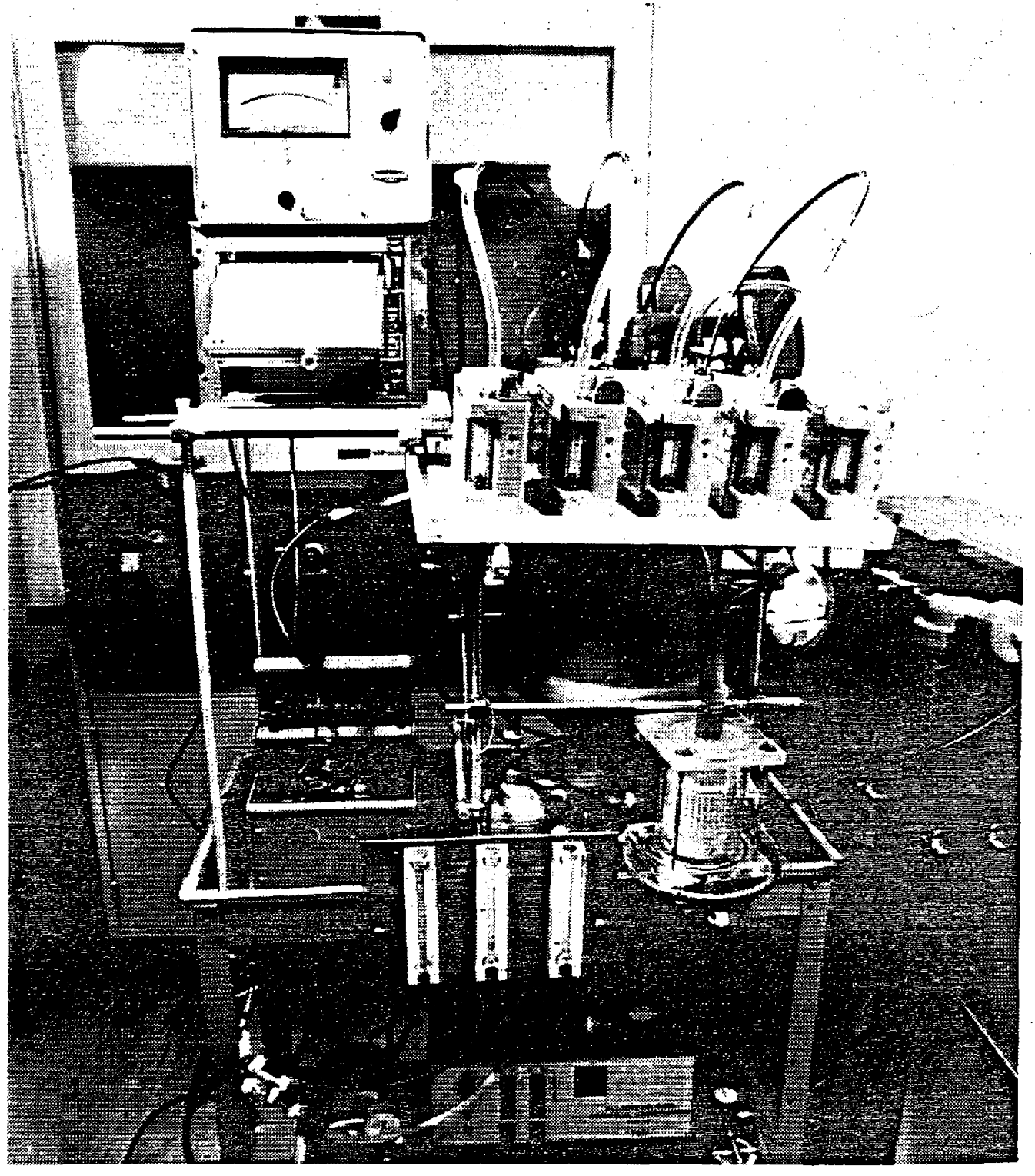
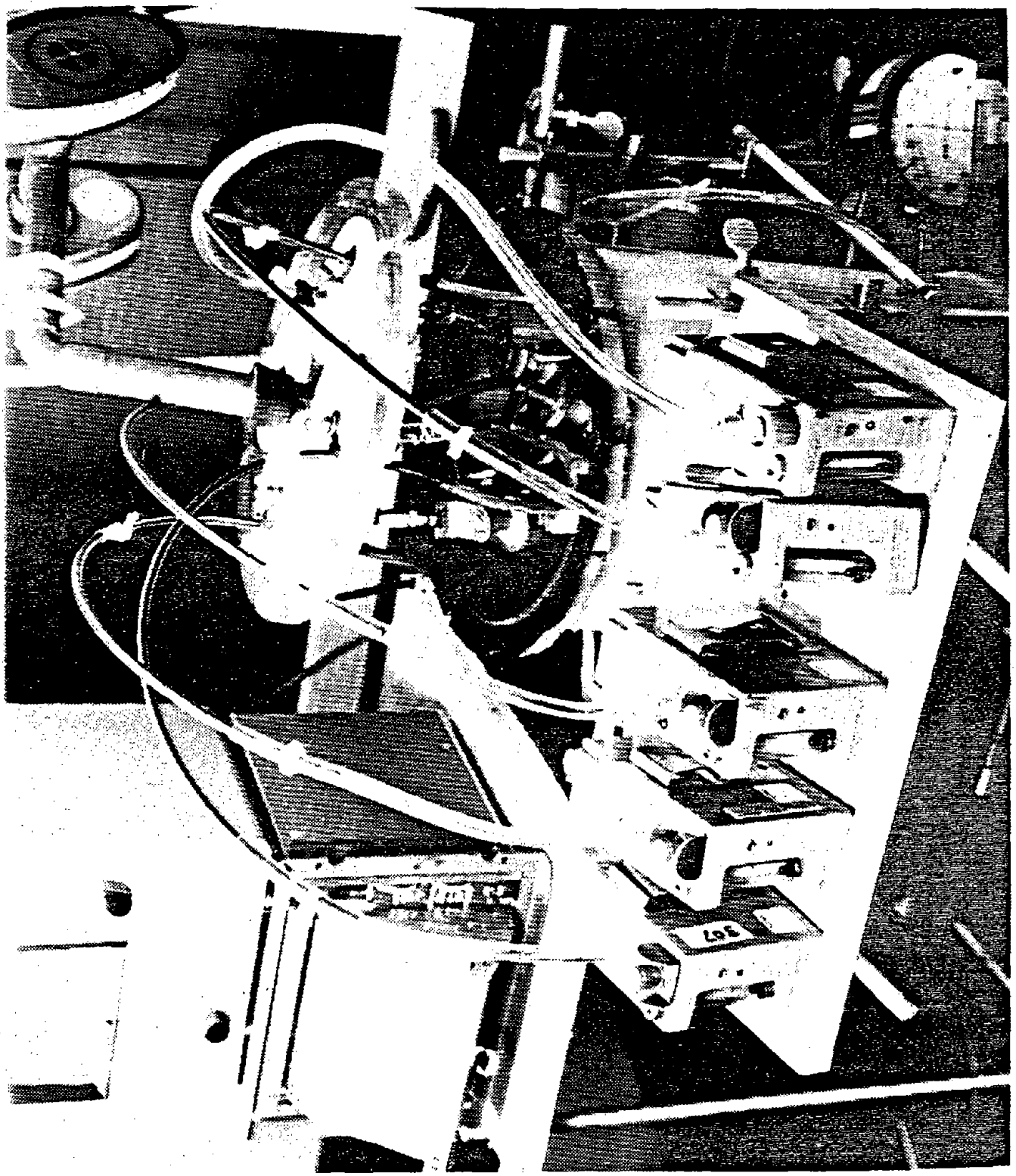


Figure III-3: Coal dust chamber with lid lifted to show MSA samplers in position for testing.



2. Monitoring Instruments

   a. A dust photometer, such as the GCA's Respirable Aerosol Monitor (RAM) or the Sibata P-5.

   b. Strip-chart recorder for monitor output.

   c. Electric hygrometer.

   d. Pressure gauge for chamber.

   e. Stop clock.

3. Weighing Equipment

   a. Balance with read-out to 1 microgram.

   b. Balance room with constant humidity (50 percent ± 5 percent) and temperature (±2°C). Alternatively, the balance can be enclosed in a glove box attached to a saturated solution of calcium nitrate for humidity control (see Figure III-4).

   c. Thermometer and hygrometer for monitoring water vapor concentration in balance room air.

   d. Tweezers grounded to balance.

   e. Polonium Static neutralizer (Nuclear Products Co. Model 2 U500). Replace after 9 months of age.

   f. Reference weights (National Bureau of Standards Class M).

4. Coal Mine Dust Personal Sampling Units (quantities depend on the experimental design, as discussed below).

   a. Complete sampler units (pump, sampling head, and connectors), as provided by the manufacturer. All components of the CMDPSU are labeled A,B,C, etc. (see Figure III-5), so they can be kept together during the test.

   b. Sealed filter cassettes whose filter capsules have been pre-weighed to 0.01 mg (if available). At least a dozen extra cassettes should be available for preliminary testing and repeats of flawed runs.

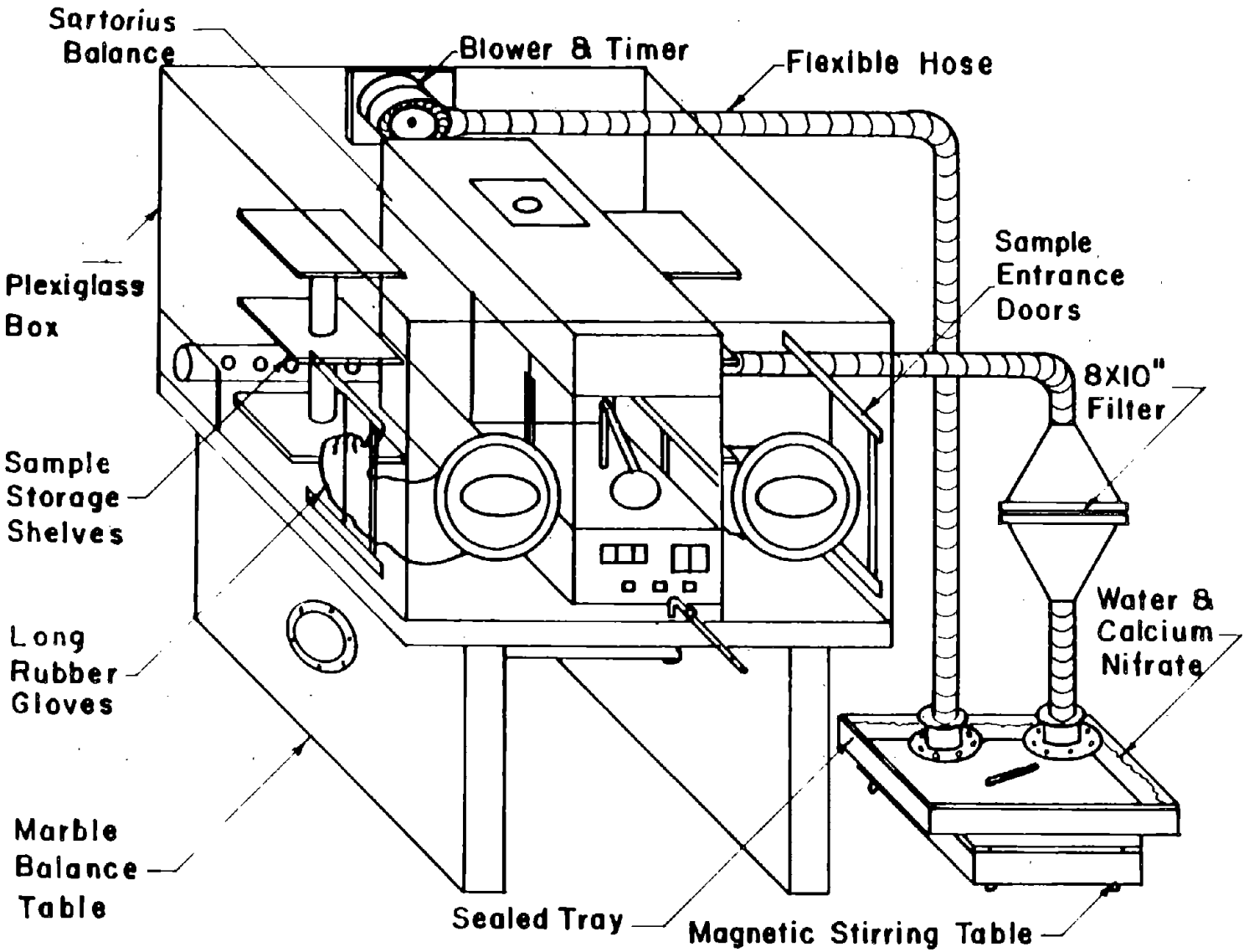
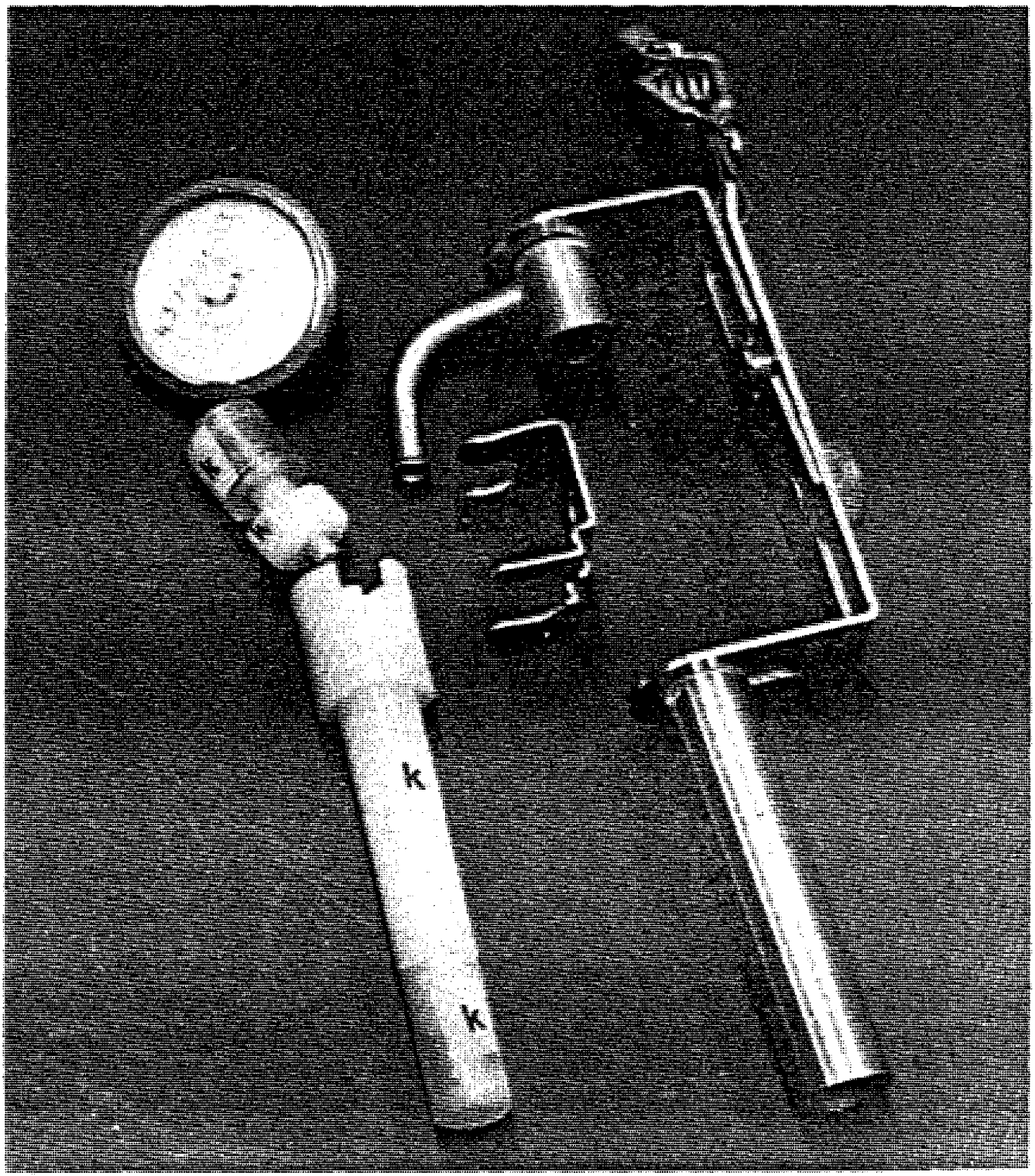


Figure III-4: Glove box to control humidity for the microbalance.

Figure III-5: MSA sampler head (disassembled). All parts (pump included) are labeled so that the unit can be kept intact througout the experimental design.



PROCEDURE:

1. Experimental design

Designs for the precision measurement are discussed fully in Section II C. The design to be used depends on the properties of the dust chamber, particularly its sampler capacity and the homogeneity of dust concentrations at the sampler positions. The three experimental designs recommended in Section II C have the following requirements:

Design	Sampler Capacity	Position Effects	No. of Runs	No. of CMDPSUs	No. of Cassettes	Degrees of Freedom
Table II-7 (top)	4	No	6	8	24	6 - 18
Table II-7 (bottom)	5	No	4	10	20	8 - 16
Table II-8	4	Yes	8	16	32	9 - 21

For all the designs, the actual order of the runs should be randomized in order to prevent the development of biases during the course of the test. For this purpose, the order for the runs in Tables II-7 and II-8 can taken from a table of random numbers. As an example, randomization of the design in Table II-8 would give the following:

RANDOMIZED EXPERIMENTAL DESIGN

Order of runs	Target Conc. (mg./cu.m.)	Sampler Positions				Run labels	
		1	2	3	4	EXP	RUN
i	4.0	I	J	K	L	I	1
ii	1.0	A	B	C	D	I	1
iii	1.0	E	F	G	H	II	2
iv	4.0	I	J	K	L	I	2
v	4.0	M	N	O	P	II	2
vi	1.0	E	F	G	H	II	1
vii	4.0	M	N	O	P	II	1
viii	1.0	A	B	C	D	I	2

Note: All targets are MRE-equivalent respirable dust concentrations.

2. Run Procedure

   a. Weigh filter capsules and blanks (Part 3). If manufacturer has weighed filter capsules to 0.01 mg, the initial weighing is replaced by the manufacturer's weight.

   b. Quality control weighings (Part 4).

   c. Insert filters into capsules and seal. Assemble sampler heads and check for leaks (Part 6).

   d. Prepare chamber and monitors for run (Sections 5 and 7).

   e. Start dust generator. After the dust concentration stabilizes in the target range, turn on samplers and start clock.

   f. Monitor dust concentration during run, adjusting dust generator and diluter to keep concentration in target range.

   g. Adjust the pumps according to manufacturer's directions to maintain 2.0 L/min flow rate. If the pump flow rate varies more than ±5 percent in consistency tests (conducted previously), then the pumps may also be adjusted one-half hour after the start and before the end of the run. Record pump flow rate at half-hour intervals throughout the run.

   h. Run samplers for 7 hours. (This sampling time is a compromise between the 8-hour samples required of CMDPSUs in regulatory applications and the administrative constraints in running the testing laboratory.) . At the end of run, turn off clock and pumps in same order as used in Part 2.d.

   i. Weigh filters (Part 3) and do QC weighing (Part 4).

   j. Collect results on data sheets for statistical analysis (Parts 8 and 9).

3. Weighing Procedure. Generally, the weighing procedures described by Jacobson and Parobeck (1971) are recommended. In a few cases, modifications are suggested where they appear to improve weighing accuracy.

   a. Filter Conditioning

      Place the filters in a container with the same atmosphere as the balance, but shielded from dust and disturbances. Let the filters equilibrate with the balance chamber air for at least one hour before weighing.

b. Balance Calibration We believe in calibrating the microbalance, rather than adjusting it mechanically for changes in sensitivity. This procedure was devised for Sartorius Model 2405 Microbalance. Balances which weigh to 0.01 mg would not appear to need calibration for every batch of filters, as recommended here.

1. Record humidity, temperature and pressure in balance room. If humidity in particular has exceeded the range from 45-55 percent, it must be controlled before proceeding.

2. Release balance and set scale to zero. If the zero knob is insufficient, bring the zero within range by adjusting the balance's front feet. Acheive the final zero with the adjustment knob. If internal adjustments are needed to reach zero, get help from a professional.

3. Check balance sensitivity against a 10 mg reference weight which has been kept in a weighing jar for this purpose.

   a. place 10 mg reference weight on pan.

   b. dial up the 10 mg counter-weight on the balance (reading of 0.01 grams) and release. Record scale reading (S_0) on data sheet.

   c. set counter-weights to 0.00 gr. and release. Record scale reading (S_1).

   d. calculate the calibration factor c:

$$c = 10{,}000 \text{ ug} / (S_1 - S_0) \qquad \text{(III-1)}$$

4. The masses for the filters are then calibrated according to the model:

$$M = \text{true mass of object being weighed} = M_{cw} + c\,S \qquad \text{(III-2)}$$

where M_{cw} = nominal mass of counter-weights (covers 1 g to 10 mg range on the microbalance)

S = Scale reading (1 mg - 1 ug range)

c. Order of Weighing

1. Balance calibration

2. 200 mg NBS Class M standard weight

3. Blank filter capsule (treated exactly the same as the sample filters for the entire test)

4. Sample filters

5. Quality control procedure (see Part 4 below)

d. Weighing Filters

1. Adjust zero before each weighing

2. Eliminate static on filter

a. For MSA capsules with aluminum shields, place filter in pan, using tweezers grounded to the balance with a wire and alligator clips.

b. Bendix cassettes - hold filter in front of the static neutralizer before placing on balance pan.

3. Weigh filter to 1 ug (or 10 ug if the filters are preweighed to that level by the manufacturer).

4. Use tweezers to put filter back in cassette.

e. Records (see Weighing Data Sheets in Appendix D)

1. Before each weighing session, record the temperature, and relative humidity.

2. From the calibration, record the scale readings S_1 and S_0 (defined in part b.3.b. and c.). Also note anything unusual about the balance (e.g. the need to adjust balance feet to reach zero).

3. For each capsule, record the filter ID number, the manufacturer's weight and the uncorrected test weight.

4. Quality Control Procedures

a. Have a different operator do the QC weights. To minimize the likihood of a humidity change, do the QC weighings immediately after the test weight.

b. Calibrate the balance (See 3.b.)

c. Weigh the standard weight and the blank filter.

d. Record all the QC weighings on the Weighing Data Sheet and record the results of the QC calculations on the Quality Control charts (Tables III-1 to III-3). Following the theory in Section II C, the QC calculations are performed as follows:

1) Balance Calibration. As described in Part 3.b., the balance calibration gives the scale readings both with the balance's counter-weight (S_0) and without (S_1). For the QC calculation, take the difference $S_1 - S_0$ (whose reciprocal is proportional to the balance calibration factor c). On the control chart (Figure III-1), the range and the average of this difference is recorded for both the initial weighing session and the QC session. Although control limits have not been determined for the calibration weights, this data is useful in determining the need for balance maintenance.

1) Standard weight. In a single weighing session, the 200 mg standard weight is weighed first by the analyst (X_1) and then by the QC operator (X_2). These masses are first corrected (if this applies to the balance used) and then averaged:

$$\bar{X} = (X_1 + X_2) / 2 \qquad \text{(III-3)}$$

The average weight is then graphed on the control chart (Figure III-2). Exceeding the control limits for the standard weight usually implies problems with the weighing procedures, the balance or the calibration.

2) Replicate Weights. The corrected weights for both the 200 mg standard weight and the blank filter are used to calculate the range R between initial and QC results:

$$R = X_1 - X_2 \qquad \text{(III-4)}$$

These ranges are entered in the control chart (Figure III-1), and compared against the control limits (Eq. II-46). If R exceeds the control limits , the entire set of filters are re-weighed by the QC operator.

Unless the humidity had changed between the initial and QC weighings, an R out-of-control indicates deviations in weighing procedures and/or balance behavior. When this occurs, both operators should observe each other to standardize their technique.

Table III-1: Quality Control Chart for 10 mg reference weight used to calibrate the microbalance.

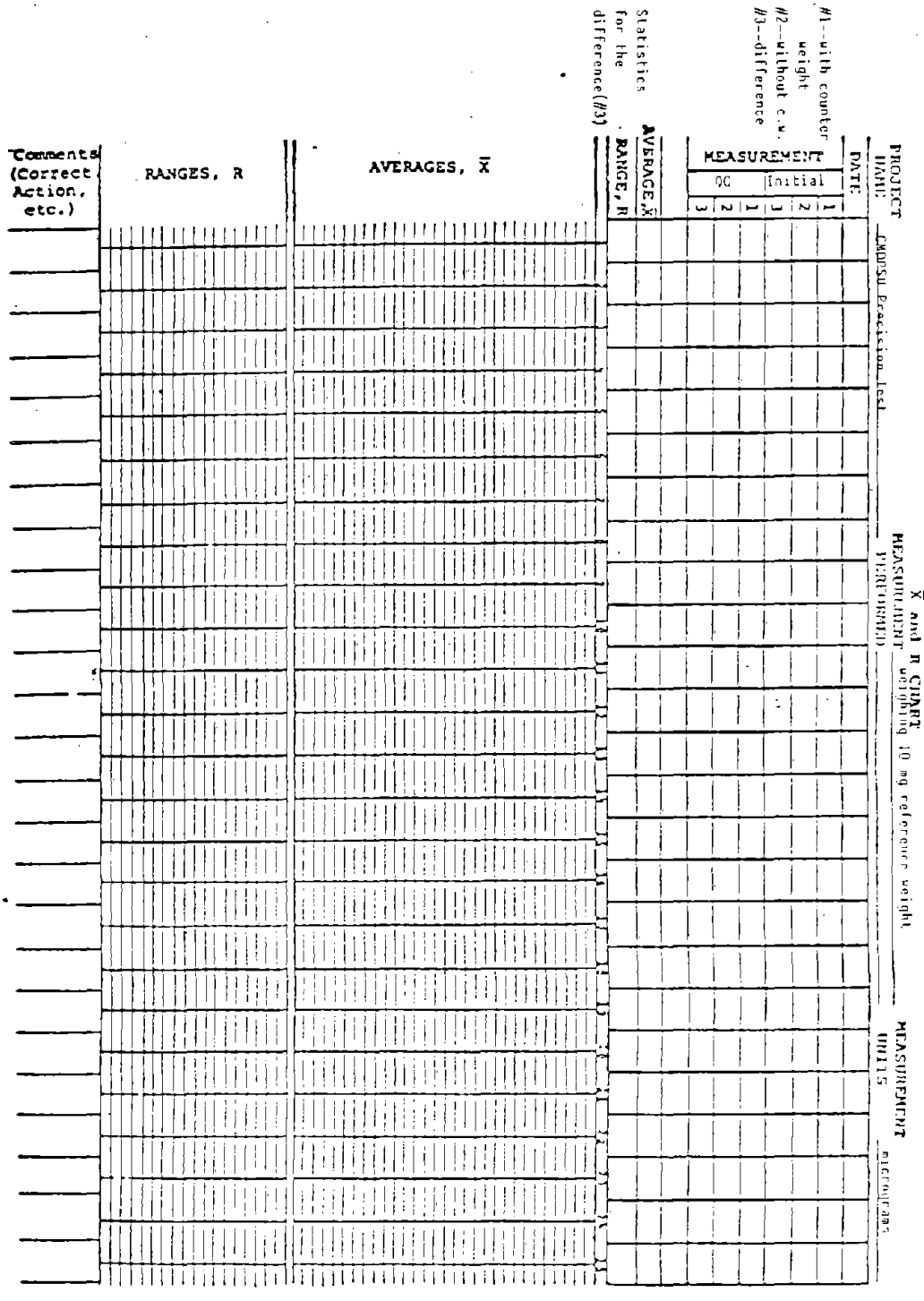


Table III-2: Quality Control Chart for Replicate Weights. Sample calculation shown in the first two columns.

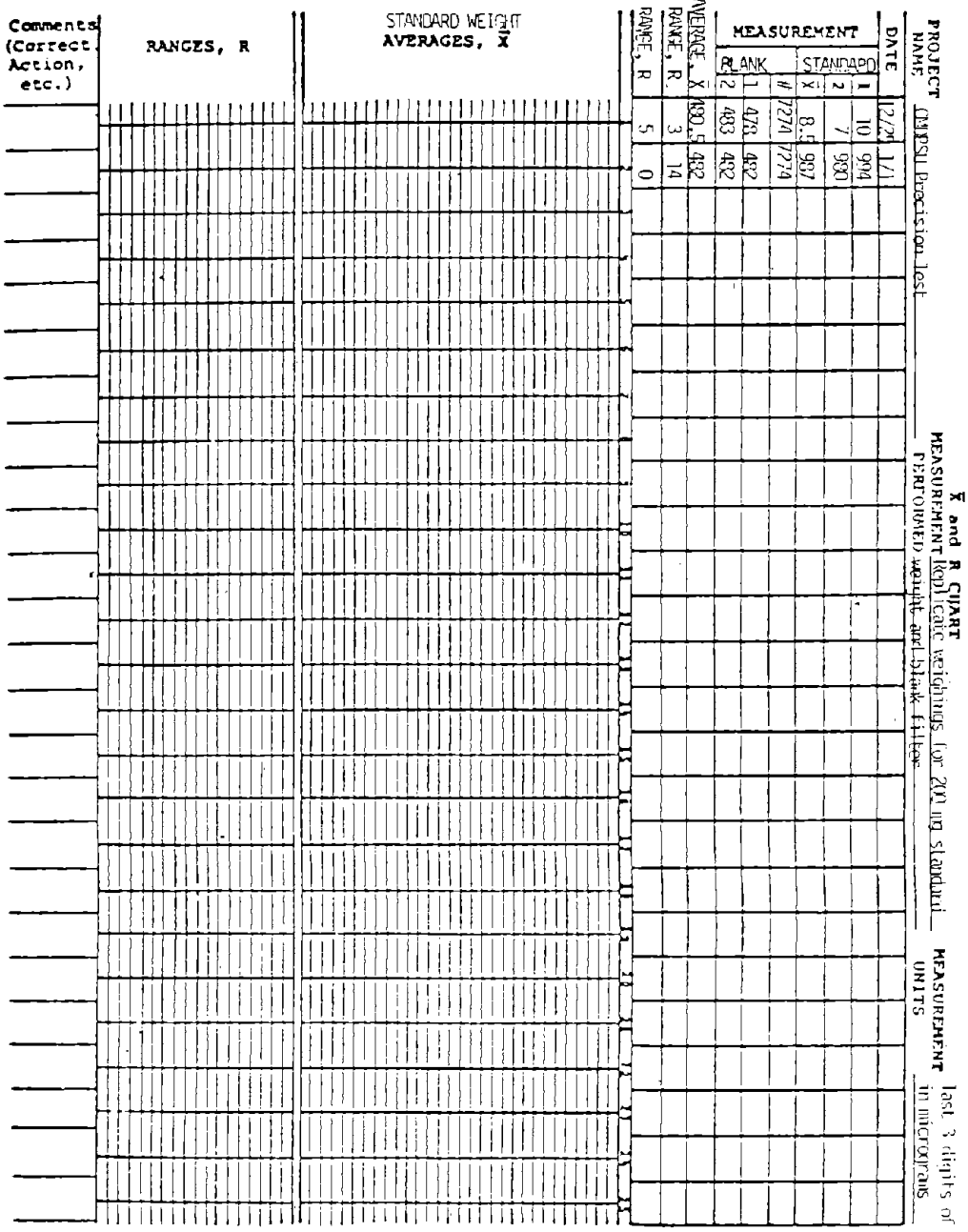
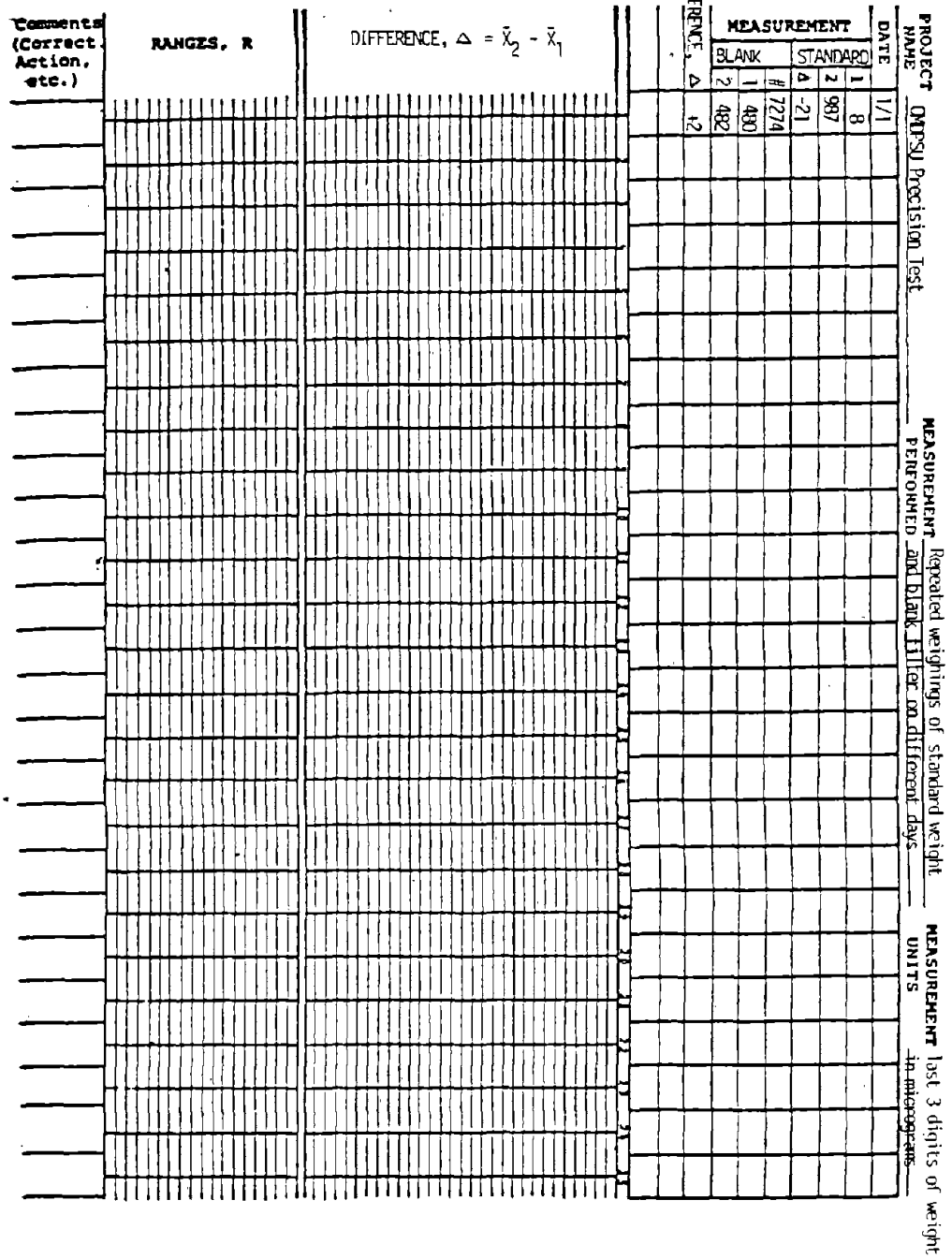


Table III-3: Quality Control Chart for Repeated Weights. Sample calculation shown in first column.



PROJECT NAME OMPSU Precision Test

MEASUREMENT PERFORMED Repeated weighings of standard weight and blank filter on different days

MEASUREMENT UNITS last 3 digits of weight ~~in micrograms~~

DATE	MEASUREMENT			DIFFERENCE, Δ
	BLANK		STANDARD	
	2	1	1	2
1/1	482	480	8	+2
		# 7274	987	
			-21	

DIFFERENCE, $\Delta = \bar{X}_2 - \bar{X}_1$

RANGES, R

Comments (Correct Action, etc.)

3) Repeated Weighings. Weighings on different days give a measure of day-to-day variations in balance performance. This effect can be estimated from the repeated weighings of the standard weight and of the blank filter (provided that both initial and final weighings are done by the certification laboratory).

To control for this effect, the difference of weights from successive sessions are charted on the control chart for repeated weighings (Figure III-3). Record the average weight ($\bar{X}$) for the 200 mg standard in a single session. Then calculate the difference with the average weight from the previous session:

$$\Delta = \bar{X}_i - \bar{X}_{i-1} \qquad \text{(III-5)}$$

Plot this difference against the control limits $\pm 3s_\Delta$. The average weights for the blank filter can also be charted in this way if the certification lab takes the filter preweights.

An out-of-control situation in the repeated weights usually indicates a significant change in the humidity of the balance room. Where the filter capsules are weighed initially and sealed into the cassettes by the certification laboratory, this control chart for the blank filter also indicates weight gain (or loss) due to the filter handling processes. When Δ is out of control, the entire run in the precision experiment must be repeated.

f. Additional QC Measures. The calculations in this test were done twice by different people as a QC measure. Other QC measures would be desirable, such as control charts for the difference between manufacturer's and certification weights on blank filters, for the average pump flow rates, and for the over-all precision (CV_t) of some standard set of CMDPSUs. DPSE could assist in developing these systems if desired. For further suggestions, see "Quality Assurance Handbook for Air Pollution Measurement Systems" (EPA; 1976,1977).

5. Preparation of Dust Chamber

a. Assure that the air supply to the dust generator is clean and dry.

b. Check level of coal dust in the generator and fill if needed.

c. Turn on air supplies and the dust generator.

d. Keeping all the settings on the fluidized bed generator constant, adjust chamber dilution air so that the dust monitor shows concentrations corresponding to the target for respirable dust. Note that all the target concentrations are expressed as their equivalent to the MRE horizontal elutriator. (See calculation method in Part 8.d. below.)

The GCA RAM set up to sample from a probe (i.e. without a cyclone pre-selector) did not give readings corresponding directly to the respirable dust concentration. In the trial measurements, we found the following monitor values corresponded to the MRE-equivalent target concentrations:

Respirable Dust Target	RAM Read-out
1 mg/cu.m.(MRE equivalent)	1-2 mg/cu.m.
4 mg/cu.m.	7-8 mg/cu.m.

e. Take out ports of chamber and install samplers. (See Part 6 below). Adjust CMDPSU position so that all inlets are symmetrical around the circumference. (See Fig. III-6).

f. When dust concentration stabilizes, sampling can begin. In the smaller chambers, the dust concentration may have to be adjusted to meet the target after samplers are turned on,.

6. Preparation of CMDPSU's

a. Calibrate the sampling pumps to 2.0 L/min. For the flow rate callibration, either use a bubble meter (MSHA Informational Report No. 1121), or a flow meter which has been calibrated against the bubble meter.

b. Charge the sampling pumps.

c. Clean cyclones with soap and water. Use alcohol to clear out stubborn coal dust, but wash with clean water. Let dry thoroughly.

d. Use filter cassettes preweighed by the manufacturer to 0.01 mg. Mark the cassettes with the sampler ID (A,B,C...), taken from the experimental design. Then, attach the cassette to the corresponding sampler head.

e. Check the assembled sampling head for leaks, using the apparatus in Figure III-7. With the pinch clamp open, run the sampling pump until the pressure reading on the manometer reaches 4 inches of water. Then, close the pinch clamp and time how long the pressure holds at this level. A satisfactory seal on the sampler should hold the four-inch vacuum for at least one minute.

Figure III-6: To view of coal dust chamber, showing the symmetric placement of sampler inlets.

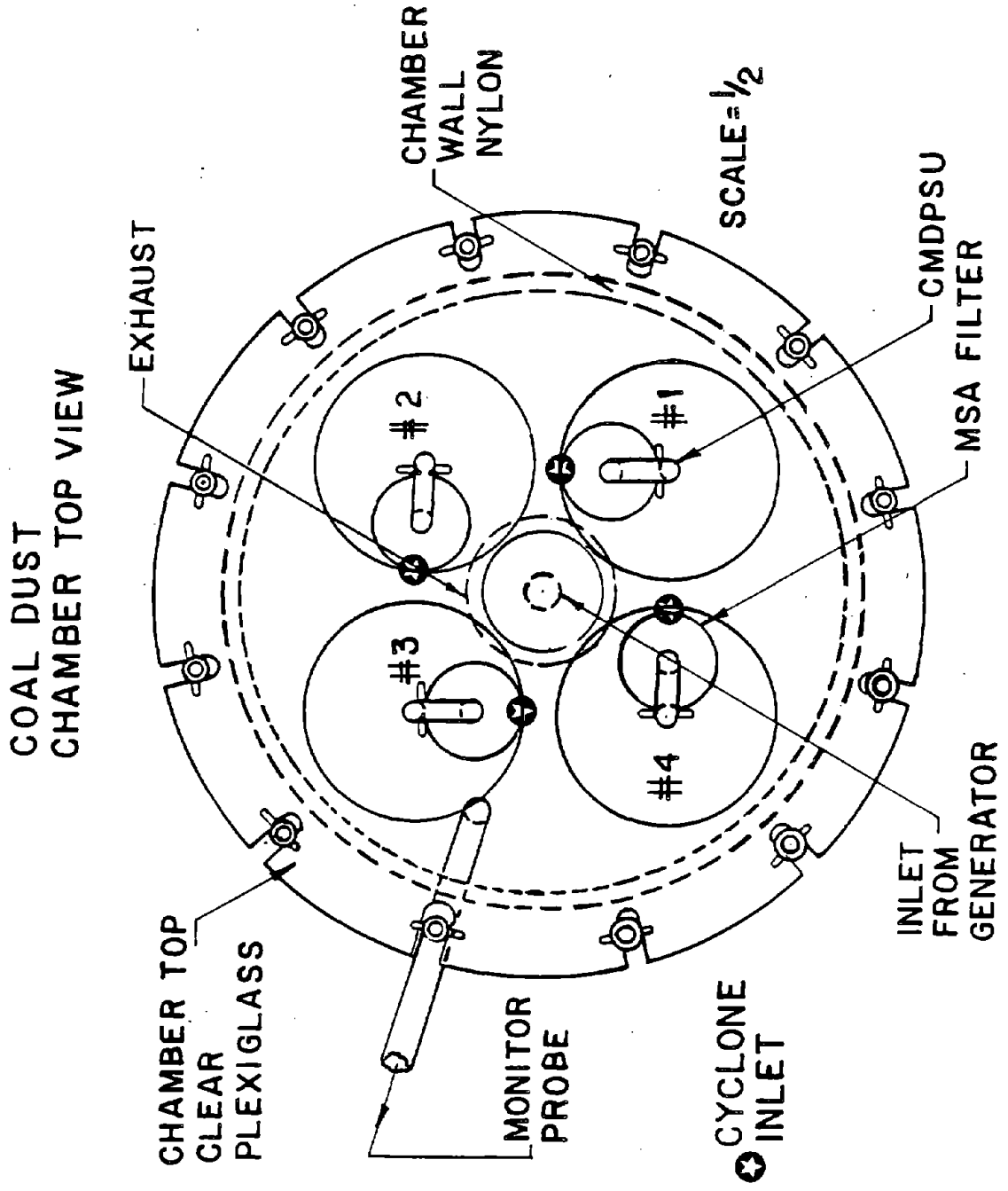
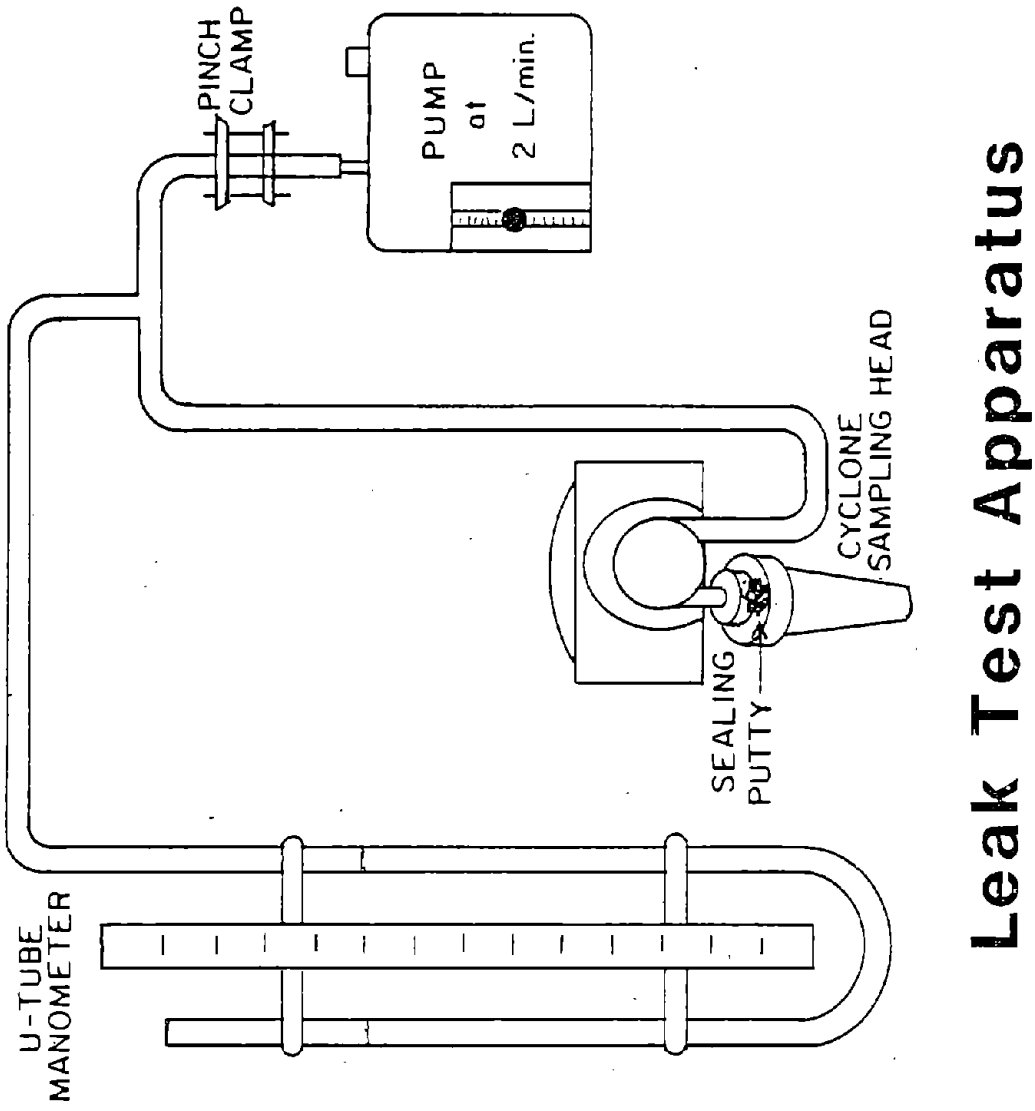


Figure III-7: Apparatus for the leak test on the sampler head.



f. Before the coal dust is started to run through the chamber (see Part 5), install the samplers in the chamber according to the experimental design.

g. Hook up the pumps to the sampler heads.

h. Zero clock and start timing first. THEN START SAMPLING.

i. Immediately, adjust the pump rotameters so that the ball touches the top of the calibration mark. If necessary, repeat the adjustment one-half hour into the run and one-half hour before the run is finished.

j. Before all adjustments, record the rotameter setting. Do this also at 1/2 hr. intervals throughout the run.

k. At end of run, stop clock and record time. STOP SAMPLERS IN SAME ORDER AS THE START.

l. After run, remove units and disassemble. Check each CMDPSU for signs of leakage or other unusual behaviour (e.g. the leakage of dust at the junction between the Bendix sampling head and the cassette). Plug cassettes and take to the balance room for weighing (Part 3).

m. Check the calibration of at least one pump after each run. If the flow rate is outside the range of 1.9-2.1 L/min, check the calibration of all pumps.

7. Dust Monitor

Monitoring the dust levels is an important check for malfunctions in the dust generator, the most common source of errors in most dust chambers. If necessary, the monitor readings may also be a guide for adjusting the dilution settings to maintain the dust concentration near the target. The other chamber monitors, like the hygrometer, provide environmental data, which could prove useful in documenting and diagnosing sampler failures.

a. Charge the battery on portable monitors, like the RAM.

b. Calibrate the zero and span on the monitor, according to the manufacturer's directions.

c. Adjust the sampling rates from the chamber into the monitors. By performing an air flow balance (see Section IV A), make sure that the monitors are not drawing excessive air from the chamber flows.

d. Turn on the strip chart recorder attached to the dust monitor. Record temperature, humidity and all chamber parameters on the chart at the start of the run.

e. If the chamber needs it, use the monitor readings to adjust the dust generator to achieve the target concentration. Mark these adjustments on the strip chart.

f. Record any major changes in chamber and environmental parameters on the strip chart. After the run, file the strip chart with the data sheets.

g. Clean the dust monitor and the sampling probe after every run.

8. Calculations (These calculations are conveniently done on a programmable pocket calculator.)

a. For weighings with a microbalance, calculate a calibration factor (Eq. III-1), and the corrected weight (Eq. III-2) for the initial weight (M_i) and the final weight (M_f). Enter results on Weighing Data Sheet and Final Data Sheet (Appendix D).

b. On the Pump Flow Rate data sheet, calculate the following statistics:

*mean flow rate $\bar{Q}$ (L/min)

*per cent deviation $\delta_Q = [\bar{Q} / 2.0 \text{ L/min} - 1.0] \times 100$ percent

*standard deviation s_Q

*coefficient of variation $CV_Q = s_Q / \bar{Q}$

c. Enter the sampling time T for each run.

d. Calculate the MRE equivalent dust concentrations:

$$C(\text{mg/cu.m.}) = \frac{k \times [M_f(\text{mg}) - M_i(\text{mg})] \times 1000 \text{ L/cu.m.}}{T(\text{min}) \times 2.0 \text{ L/min}} \qquad \text{(III-5)}$$

where k is the calibration factor for respirable dust. Currently, MSHA's value for k is 1.38.

9. Statistical Analysis

The theory for the statistical analysis is developed in Section II C. The actual calculation is performed easily on a computer statistics package such as SAS (SAS Institute, 1979). (The ANOVA computation is also feasible on a desk calculator. See a statistician or a text, such as Snedecor and Cochran, 1967.) The SAS program is listed in Appendix B. The steps in this computer analysis are as follows.

A. Enter Data.

Enter the concentrations from the Final Data Sheet into a computer DATA file, along with the labels for the treatment variables, such as T and RUN. See the example in Figure III-4.

B. Program the analysis of variance.

For the experimental design chosen, locate the appropriate analysis of variance model (Tables II-9 or II-10). Then, program that ANOVA with the computer statistical package availible. SAS commands for the analysis of variance are listed in Appendix B.

C. Calculate CV_t

When the analysis of variance is done by SAS, the output (Tables III-5 and III-6) gives both the mean squares needed to calculate CV_t and the results of significance tests for the different sources of variance. Locate the parameters necessary for the precision calculation, as shown in Tables III-5 and III-6.

In the SAS output, the mean square (MS) for the residual error is given directly, but the mean square terms for other sources of variation must be derived from the appropriate sums of squares (SS). As shown in Tables III-5 and III-6, the SS is just the degrees of freedom (DF) times the MS. Since the formulas for calculating CV_t (Eqs. II-26, II-29 and II-35) are all derived in terms of the MS, these CV_t formulas can be modified to use directly the SS values in the SAS output, as shown below:

i. Design without position effects.

$$\hat{CV}_t = \sqrt{SS(S \text{ in } T) / 2M(N-1) + (M-1)\ MS(Error) / M} \qquad \text{(III-7a)}$$

where M = number of runs per target concentration = 3 or 2
N = number of samplers per run = 4 or 5

ii. Design with position effects. In the ANOVA output for position effects (Table III-6), the significance of the T*POS interaction is shown by an F-test (Eq. II-34). A small value for this probability (no more than 0.05) shows that this interaction is significant, and requires that the second formula (below) be used in calculating CV_t:

a. Negligible T*POS interaction. (PR > 0.05)

$$\hat{CV}_t = \sqrt{[SS(POS*T) + SS(POS*EXP \text{ in } T)] / 18 + MS(Error) / 2} \qquad \text{(III-7b)}$$

b. Significant T*POS interaction. (PR $\leq$ 0.05)

$$\hat{CV}_t = \sqrt{SS(POS*EXP \text{ in } T) / 12 + MS(Error) / 2} \qquad \text{(III-7c)}$$

Table III-4. Example of a data file (fictitious numbers) for the analysis of variance of the experimental design with position effects.

Card Record	SAS input names				
	T	EXP	RUN	POS	CONC
1	1	1	1	1	1.265
2	1	1	1	2	1.269
3	1	1	1	3	1.261
4	1	1	1	4	1.330
5	1	1	2	1	0.754
6	1	1	2	2	0.804
7	1	1	2	3	0.794
8	1	1	2	4	0.794
9	1	2	1	1	0.725
10	1	2	1	2	0.737
11	1	2	1	3	0.724
12	1	2	1	4	0.741
13	1	2	2	1	1.201
14	1	2	2	2	1.297
15	1	2	2	3	1.258
16	1	2	2	4	1.280
17	4	1	1	1	2.661
18	4	1	1	2	3.668
19	4	1	1	3	3.908
20	4	1	1	4	3.290
21	4	1	2	1	3.661
22	4	1	2	2	3.943
23	4	1	2	3	3.814
24	4	1	2	4	3.109
25	4	2	1	1	5.096
26	4	2	1	2	6.218
27	4	2	1	3	5.992
28	4	2	1	4	4.179
29	4	2	2	1	3.755
30	4	2	2	2	5.097
31	4	2	2	3	5.842
32	4	2	2	4	3.155

Table III-5. SAS output from the analysis of variance for the experimental design without position effects. Important terms for the precision calculation are underlined, and their identity is given below.

GENERAL LINEAR MODELS PROCEDURE

CLASS LEVEL INFORMATION

CLASS	LEVELS	VALUES
T	2	1 4
S	10	A B C D E F G H I J
RUN	2	1 2

DEPENDENT VARIABLE: LNC

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE
MODEL	11	9.81118359	0.89192578
ERROR	8	0.01944442	0.00243055*
CORRECTED TOTAL	19	9.83062802	

MODEL F =	366.96		PR > F = 0.0001
R-SQUARE	C.V.	ROOT MSE	LNC MEAN
0.998022	5.6327	0.04930064	0.87526112

SOURCE	DF	TYPE I SS	F VALUE	PR > F
T	1	9.33284069	3839.80	0.0001
RUN(T)	2	0.45916141	94.46	0.0001
S(T)	8	0.01918149**	0.99	0.5074

IDENTITY OF IMPORTANT TERMS:
* MS(Error)
** SS(S in T) = 8 MS(S in T)

Table III-6. SAS output from the analysis of variance for the experimental design with position effects. Important terms for the precision calculation are underlined, and their identity is given below.

GENERAL LINEAR MODELS PROCEDURE
CLASS LEVEL INFORMATION

CLASS	LEVELS	VALUES
T	2	1 4
EXP	2	1 2
POS	4	1 2 3 4
RUN	2	1 2

NUMBER OF OBSERVATIONS IN DATA SET = 32

DEPENDENT VARIABLE: LNC

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE
MODEL	19	18.24203773	0.96010725
ERROR	12	0.07010831	0.00584236*
CORRECTED TOTAL	31	18.31214604	

MODEL F =	164.34		PR > F = 0.0001

R-SQUARE	C.V.	STD DEV	LNC MEAN
0.996171	11.0099	0.07643533	0.69424275

SOURCE	DF	TYPE III SS	F VALUE	PR > F
T	1	16.27134234	2785.06	0.0001
EXP(T)	2	0.41996235	35.94	0.0001
RUN(EXP T)	4	1.15868942	49.58	0.0001
POS	3	0.16953609	9.67	0.0016
POS*T	3	0.16804582**	9.59	0.0016
EXP*POS(T)	6	0.05446172+	1.55	0.2427

TESTS OF HYPOTHESES USING THE TYPE III MS FOR EXP*POS(T) AS AN ERROR TERM

SOURCE	DF	TYPE III SS	F VALUE	PR > F
POS	3	0.16953609	6.23	0.0284++
T*POS	3	0.16804582	6.17	0.0290++

IDENTITY OF IMPORTANT TERMS:

* MS(Error)

** SS(POS*T) = 3 MS(POS*T)

\+ SS(EXP*POS in T) = 6 MS(EXP*POS in T)

++ Probability of error in the F-tests for significance

D. Additional quality controls

The additional tests included in the SAS program provide checks on the chamber performance and the statistical assumptions used in the analysis. These checks are:

i. Significance test for POS. In the ANOVA output with the position effects (Table III-6), the significance of the POS term is shown by an F-test (Eq. II-32). A large value for this probability (no less than 0.100) is an indication that the experimental design without position effects may be used.

ii. Duncan's Multiple Range Test by Position. This test, shown in Table III-7, indicates the positions where significant fixed differences in the mean concentration occur. In the example shown here, positions 2 and 3 have significantly higher concentrations than positions 1 and 4. Although this factor is removed by the ANOVA, its existence raises the possibilty of run-position interactions, which are assumed to be zero. Therefore, Duncan's test may help diagnose the causes of inhomogeneous concentrations within the dust chamber.

iii. POS*T Interaction. In the ANOVA with position effects, the best formula for CV_t (Eq. III-7b) assumes that the POS*T interaction is negligible. Another way to detect this interaction besides the F-test is to plot the ANOVA's residuals (i.e. observed value minus the value predicted by the ANOVA model). The residuals are presumably random samples from a normal distribution with mean 0 and variance $(\sigma_\varepsilon)^2$. The example plot (Figure III-8) shows clearly that residuals are distributed randomly for T=1 mg/m^3 but the range for T=1 mg/m^3 exhibits a dependency on postion. Therefore, Eq. III-7c should be used in the precision calculation, and more efforts should be made to reduce position effects in the chamber.

iv. POS*EXP Interaction. The POS*EXP interaction is assumed to be negligible in the ANOVA with position effects. Again, plots of the residuals give a qualitative check on this assumption. The example in Figure III-9 shows that EXP=2 has clear position effects -- large differences in position 1 down to little difference in position 4 -- while EXP=1 has relatively consistent residuals at all positions. This interaction is unavoidably included in the CV_t estimate, and should be eliminated by alterations in the chamber.

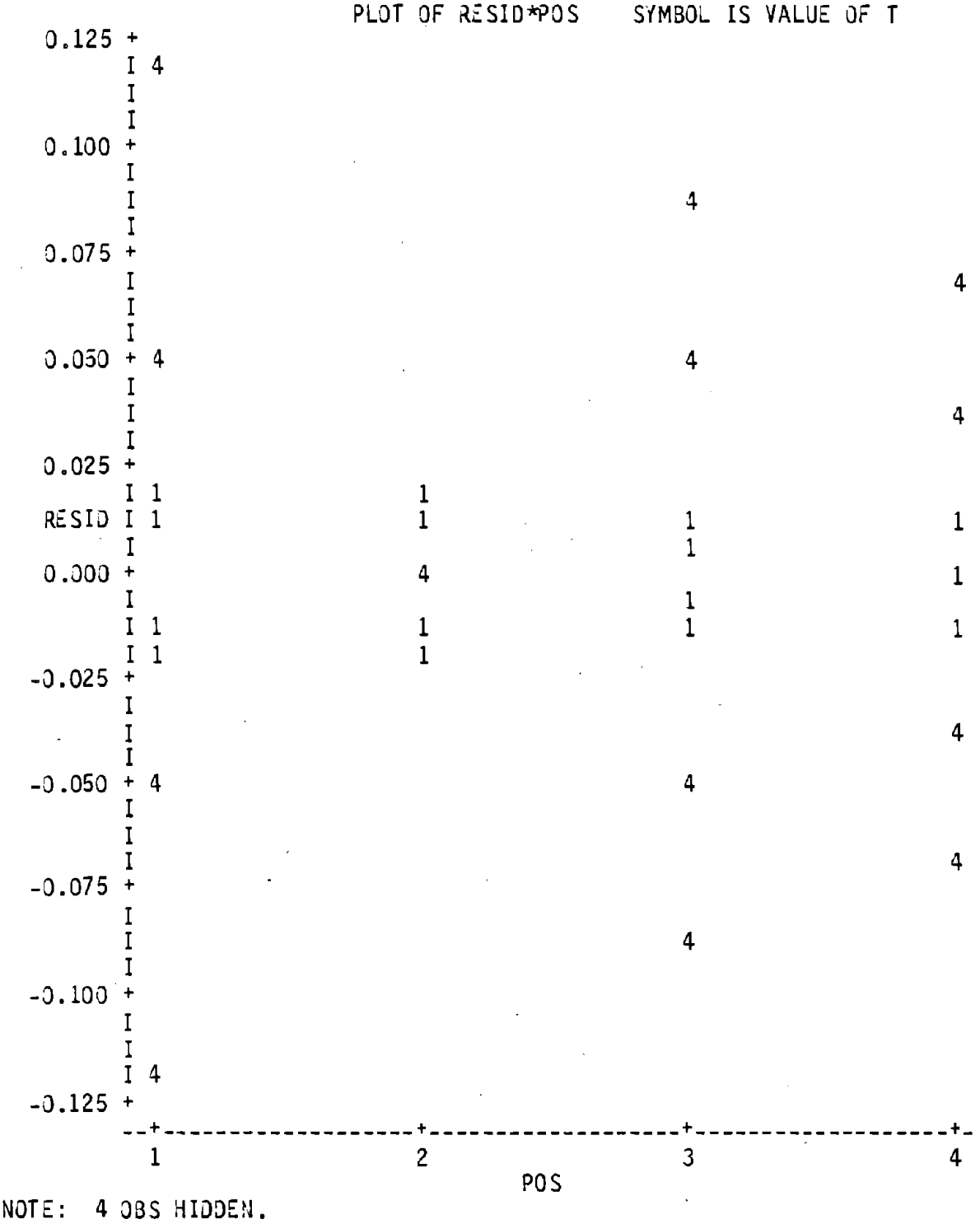
Table III-7. SAS output for Duncan's Multiple Range Test, applied to the mean concentration by position. This example shows a significant difference from the concentrations at positions 1 and 4 to those at positions 2 and 3.

GENERAL LINEAR MODELS PROCEDURE

DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE: LNC
NOTE: THIS TEST CONTROLS ERROR RATES AT DIFFERENT LEVELS
DEPENDING ON THE NUMBER OF MEANS BETWEEN EACH PAIR
BEING COMPARED. ITS OPERATING CHARACTERISTICS SOMEWHAT
RESEMBLE FISHER'S UNPROTECTED LSD TEST.
ALPHA=0.05 DF=12 MSE=.0058424
MEANS WITH THE SAME LETTER ARE NOT SIGNIFICANTLY DIFFERENT.

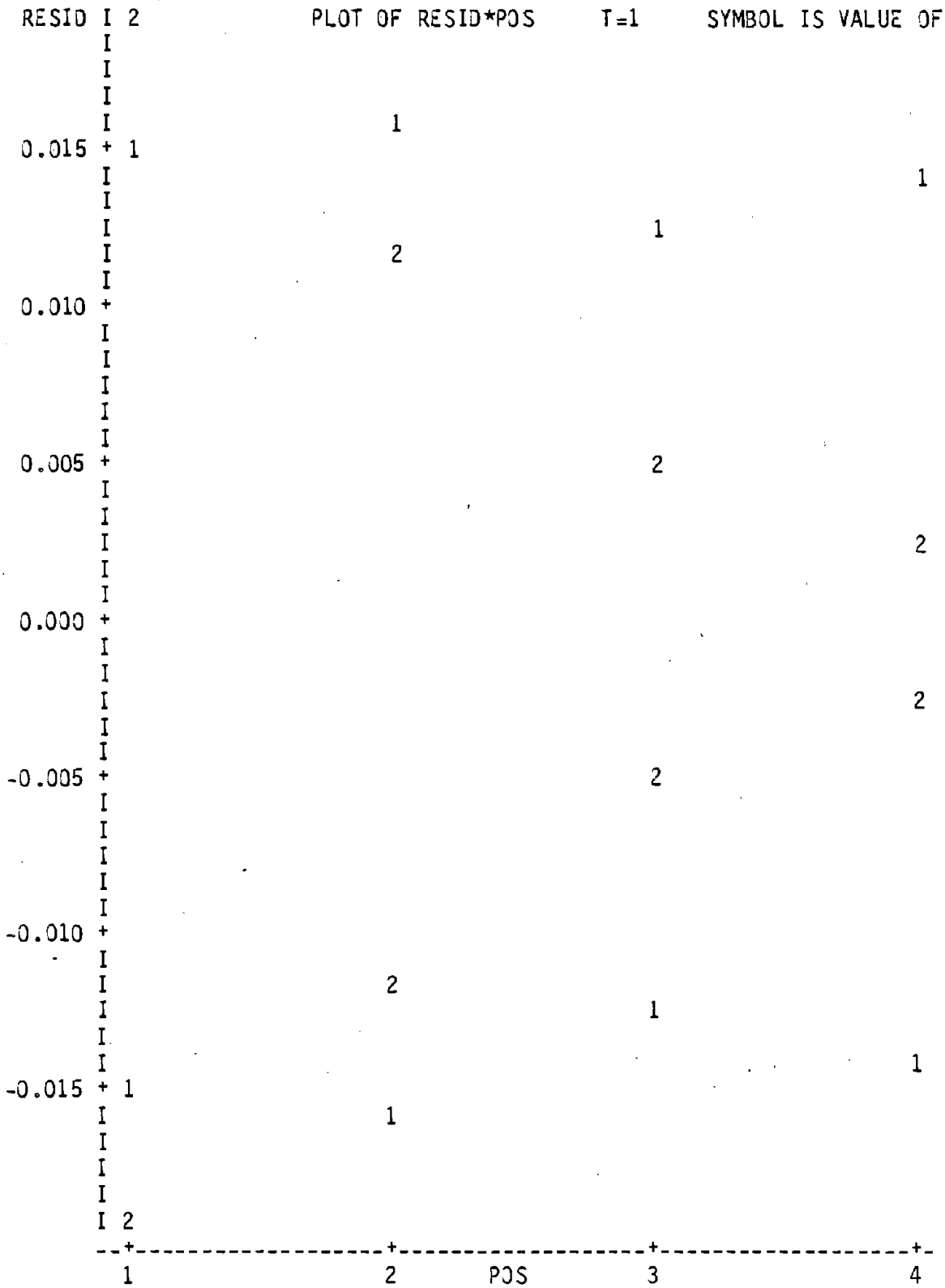
DUNCAN GROUPING	MEAN	N	POS
A	0.77062	8	3
A			
A	0.76283	8	2
B	0.63028	8	1
B			
B	0.61323	8	4

Figure III-8. SAS plot of ANOVA residuals (fictious data) to show POS*T interaction.



NOTE: 4 OBS HIDDEN.

Figure III-9. SAS residual plot (fictitious data), showing POS*EXP interaction at T=1 mg/cu.m.



B. COLLECTION EFFICIENCY MEASUREMENT

Certification testing requires measurement of the collection efficiency of the complete sampling unit (i.e., including the sampling unit's pump) operated according to the manufacturer's specifications. As mentioned in Section II, the measurements are to be taken at aerodynamic diameters equal to 2.25, 3.00, 3.75, 4.50, 5.25, 6.00, 6.75 and 7.50 micrometers. This section describes the experiment for affecting these measurements.

Detailed descriptions of the labortory equipment as well as the procedures to be followed are given below. However, a brief outline of the methods to be used is as follows. The apparatus used to test these samplers uses a chamber in which the sampling head assembly is suspended. Plastic sampling unit tubing is replaced by a similar tube which incorporates a feed-through through the chamber wall so that the pump can be attached outside the chamber. A standard test aerosol of potassium hydrogen phthalate (KHP) doped with uranine (sodium fluorescein) is then generated in dry air, deionized, and passed through the chamber. The test aerosol generator is a Berglund-Liu vibrating orifice aerosol generator which produces a monodisperse aerosol of a size determined by the frequency of the orifice, the flow rate of the liquid feed, and the concentration of the solid in the volatile liquid. The amount of aerosol collected on the sampler surfaces and on the filter is determined by dissolution of the KHP/uranine in a disodium phosphate buffer (7.05 g/L in water) and fluorometric measurement of the uranine. Several points at the above particle sizes are taken to determine the penetration curve of the sampling unit.

APPARATUS

A diagram of the entire apparatus is given in Figure III-10. A more detailed description of the various parts of the setup is given below, broken down into the various subsections as follows: test aerosol generator, including air supply, liquid feed and flowmeter, frequency generator and counter, and deionizer; test chamber; continuous particle size monitor; and analytical equipment including additional quality control checks.

Test Aerosol Generator. A Berglund-Liu vibrating orifice aerosol generator (Thermo Systems Inc. model 50A) with a 20-μm orifice is used to generate the monodisperse aerosol. The generated aerosol is charge neutralized with a 10 mCi ^{85}Kr deionizer (TSI model 3054). From the original calibration data, the deionizer source is estimated to have dropped to about 7 mCi.

An Exact model 126 function generator is used to supply a 7.2 V rms sine wave signal of appropriate frequency to the Berglund-Liu. The frequency used falls in the 40 to 90 kHz region, such that a single particle size is observed. The frequency is monitored with a digital counter (Eldorado model 1420).

The KHP/uranine stock solution (14.992 g/L KHP, 0.299 g/L uranine in 50 percent H_2O/50 percent isopropanol) is diluted with 50 percent H_2O/50 percent isopropanol to an appropriate concentration for the desired

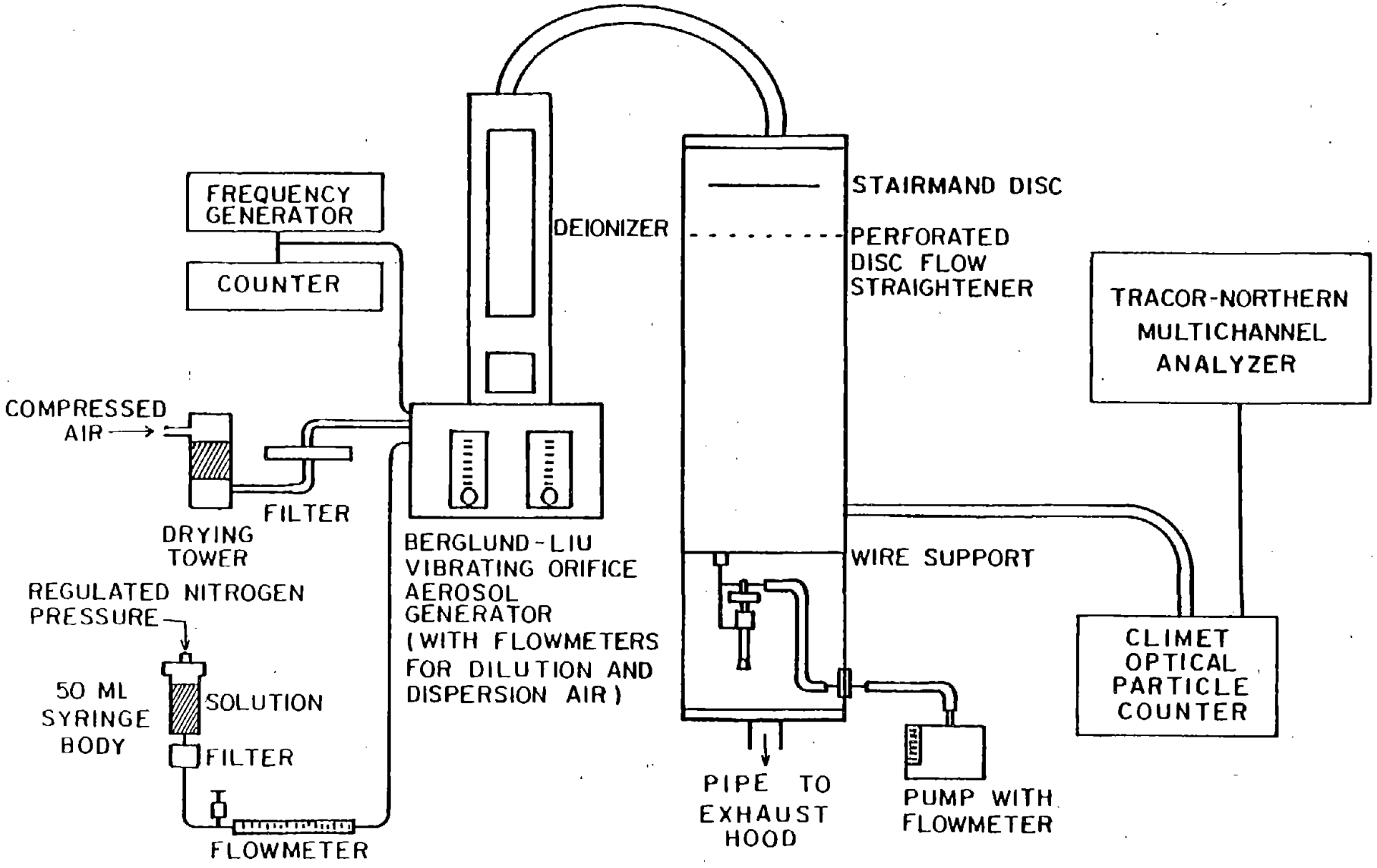


Figure III-10. Apparatus for the collection efficiency measurement.

particle size. The diluted solution is held in a glass 50 ml syringe body with a custom made adapter in place of the plunger. The adapter is connected via tubing to a cylinder of nitrogen gas with a two-stage regulator to maintain a steady pressure of about 20 psi. A toggle valve on a T-connector is put in the nitrogen line to allow the pressure to be released in the syringe body when fluid needs to be added. The pressure is monitored with a Dynisco model DR 482 pressure monitor. The liquid feed in the syringe passes through a filter and then through a flowmeter. The flowmeter consists of a 1 ml syringe connected to a T-fitting in the line so that a bubble can be injected in the flowing liquid and then a serological 0.200 ml pipette body (graduated every 0.010 ml). The bubble often causes the Berglund-Liu to stop generating, so the flow is usually taken only at the end of the run.

Test Chamber. The plate covering the top of the test chamber is made of 1.27 cm thick Plexiglass with a 6.35 cm. long, 3.18 cm. ID Plexiglass tube cemented into the center. This plate is attached to the outlet of the deionizer by a 0.57 m. length of 3.18 cm. ID flexible hose. The chamber is constructed from a large Plexiglass tube of 13.7 cm ID, 0.4 cm wall thickness and 138 cm long. The tube is mounted vertically with the aerosol inlet in the center of the top. A 9 cm. diameter Stairmand disc is held by three 1.2 cm. wide tabs bolted to the wall at 28 cm from the top and a perforated disc flow straightener at 42 cm from the top; the flow straighener has 0.16 cm. diameter holes in a 0.12 cm. thick plate the same diameter as the inside of the chamber and held by four tabs bolted to the wall. The three sampler heads are suspended from a wire support at 104 cm from the top so that the sampling inlets are located 112 cm from the top. The wire support hangs from three 2.5 cm. 10-32 machine screws through the chamber wall. A removable cap at the bottom allows the excess air flow to exit through a flexible hose to a laboratory hood. At 42 cm from the bottom a 1/4" tube inserted through the wall leads to the Climet optical particle counter. The feedthroughs connecting the sampling heads to their pumps are located 5 cm from the bottom of the chamber. The samplers hang vertically in the chamber.

Continuous Particle Size Monitor. A Climet model 208 optical particle counter sampling at 7.0 liters per minute (0.25 cfm) is used to monitor the particle size distribution in the chamber, although any optical particle counter of similar type (such as Royco) would work as well. The Climet output is connected to a Tracor Northern TN-1710-8K multichannel analyzer with a TN-1710-2 5 MHz ADC input. This allows the particle size distribution to be continuously monitored to check for proper Berglund-Liu operation. A TN-1710-7 Autoprogram Module permits repetitive sampling and display of the distribution without requiring operation by laboratory personnel.

Analytical Equipment. Filters to be analyzed are immersed in the buffer solution in a 50 ml beaker which is placed in an ultrasonic bath (150 W) for 1 minute. Sampler surfaces are washed down with the buffer solution into 30 ml beakers; a plastic wash bottle was used for the buffer solution.

The fluorescent intensity of the solutions is measured with a Turner Model 110 fluorometer or Varian Fluorichrom fluorometer (model 430020-02) using a Keithley model 192 digital voltmeter (or equivalent) to read out the signal. Solutions are drawn into the fluorometer cell with a 10 ml syringe attached to the outlet tubing via flexible tubing for use with the Varian fluorometer. The excitation filter is a 430 nm interference filter and a Corning 5-58 color glass filter. The emission filter is a Corning 3-72 and two layers of "straw" acetate filters. Response of the fluorometer is about 70 V per g/L of uranine using high lamp intensity and high photomultiplier voltage. The Turner fluorometer requires a 47B primary filter and 2A-12 secondary filter.

Checks on sphericity and uniformity of the aerosol can be accomplished by impacting the particulate on a microscope cover slip with a Marple impactor of appropriate orifice size and a 2 Lpm personal sampling pump (MSA model G). This sample, taken at the end of the run, can then be observed microscopically.

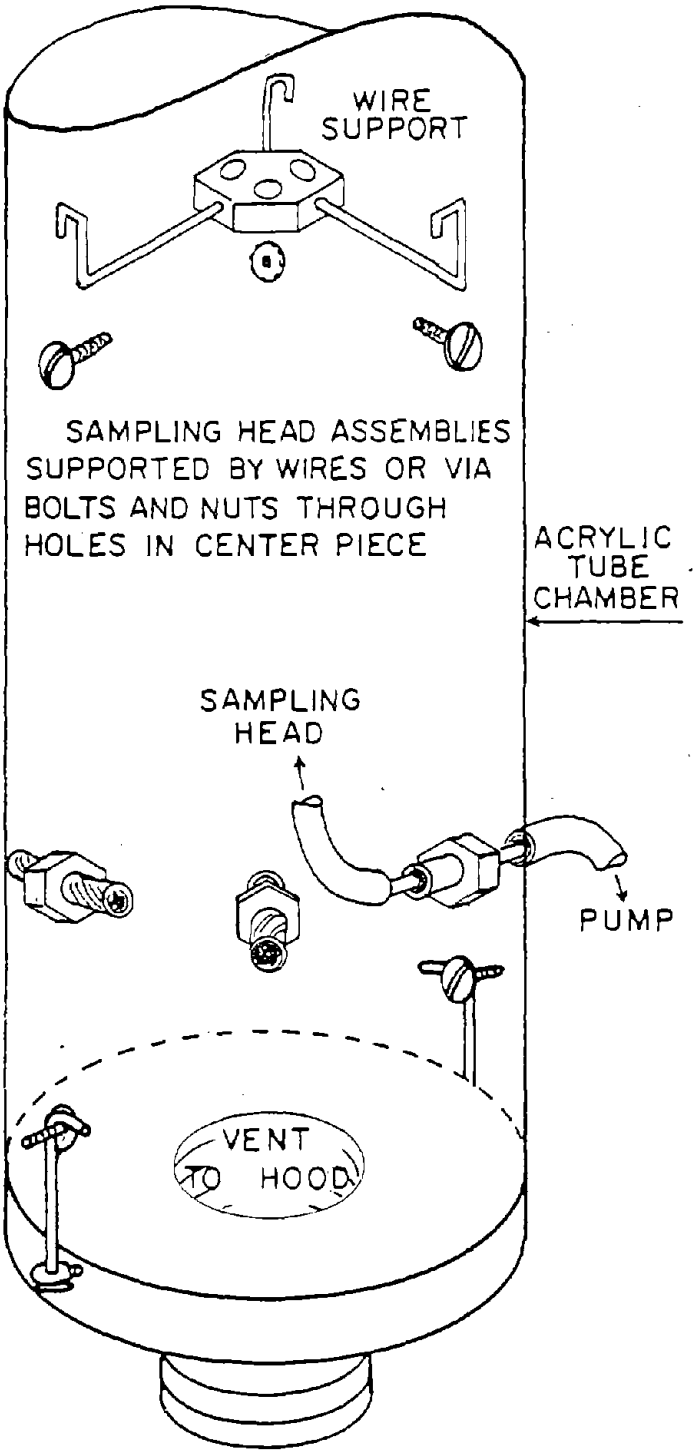
PROCEDURE

Three sampling head assemblies are connected by clips, bolts, or some other technique to the wire support (see Figure III-11), which is supported on three bolts which penetrate the chamber wall. Connections are made from the sampling heads to the wall feedthroughs with 1/4" ID flexible plastic tubing. A connection with the same type of tubing is made from the exterior of the feedthrough to the personal sampling pump such that the total length of tubing including the feedthrough is about the same as the normal tubing length and type used in actual sampling. The feedthroughs are Jaco nylon 1/4" tubing connectors screwed from the outside into drilled and tapped holes in the chamber walls. The connector is drilled so an approximately 10 cm piece of 1/4" OD hard plastic tubing can pass through and be held in place by one compression nut on the outside of the chamber. The tubing connections are made leak tight at the feedthrough by a wire tie, by Teflon tape, by flaring the tubing ends, or some other technique which will not constrict the tubing.

The liquid feed system is flushed with distilled water (to remove any cyrstalline KHP in the syringe filter), with 50 percent isopropanol/water, and finally with the diluted stock solution to be used. The system is flushed by adding the appropriate liquid to the syringe body by a small funnel through the tubing connector used to hook up the nitrogen tank. The syringe body is then pressurized and the liquid allowed to flush through the system and out the relief valve on the outlet of the Berglund-Liu.

The syringe body is then filled with 30 to 50 ml of the diluted stock solution and pressurized. The relief valve on the Berglund-Liu is closed. Care must be used to avoid bubbles in the liquid feed line because they will cause the Berglund-Liu to quit generating. During the flushing procedure and this loading and pressurizing procedure, the orifice should be in place but without the dispersion air cap. At this point, the fluid will begin to flow through the orifice as pressure builds up; the fluid must be periodically removed with a towel as it builds up. When the pressure exceeds about 15 psi, a stream will form from the orifice following removal of the standing liquid. An operating pressure of about 20 psi was found to work well.

Figure III-11. Details of the Sampling Compartment for the Collection Efficiency Measurement.



A clogged or partially clogged orifice, the usual cause of failure to function, can often be remedied by trying to force distilled, filtered water back through the orifice with a plastic wash bottle. It is usually necessary to release pressure and remove the orifice holder to do this.

With the function generator on, the stream is checked for a single particle size by blowing the dispersion air across the stream with the small tube supplied by TSI. A single stream, bent at an angle from the original stream should be observed by forward scattering of a more or less collimated light beam aimed at the stream. Adjust the frequency until this is the case (60 to 85 kHz is recommended). At the end of the run the pumps are shut off and the liquid flow into the Berglund-Liu is checked and recorded by injecting a small bubble of air which fills the diameter of the flowmeter (and is at least as long as the diameter) into the flowing stream. The bubble is timed with a stop watch for 0.200 ml.

The aerodynamic diameter D of the particles generated by the Berglund-Liu is given by:

$$D = \rho^{1/2} (6 Q C / 3.14 f \rho)^{1/3} \qquad \text{(III-8)}$$

where ρ is the density of the particle (1.636 for KHP), Q is the measured liquid flow rate in liters/sec, C is the nonvolatile concentration in g/L (15.29 g/L for the stock solution times ml of stock divided by total final volume), and f is the frequency in hertz.

The sampling heads are removed from the chamber and the filters and samplers are removed from the holders. Care must be used to maintain sample identity by proper labelling in the following process.

The filter is removed from the cassette and separated from its aluminum foil or plastic capsule. The filter is placed in a 50 ml beaker and the interior of the aluminum foil or plastic capsule is rinsed off into the beaker using a few ml of the disodium phosphate buffer (approx. 7.05 g/L). The filter is thoroughly wetted by the solution using a glass rod or disposable pipette. The beaker is placed in an ultrasonic bath for 1 minute, and then is decanted into a 25 ml volumetric flask. Because the filter is almost the same diameter as the beaker, some liquid is usually trapped under it; this may be removed by tilting the beaker upright and decanting again. The beaker and filter are rinsed thoroughly into the flask with three more portions of buffer, thoroughly wetting the filter and decanting as before. The flask is then brought up to the line with buffer. A blank filter should also be run, especially if air was drawn through all three sampling heads rather than leaving one of them as a blank.

In a series of test runs, two active sampling heads and one blank were used; a total dust filter with an inlet diameter of 0.5 cm. rather than the cyclone's 0.2 cm. square inlet was also taken using the third feedthrough left unused by the blank. Both the cyclones and the filters of the blanks were always very near zero (within the typical variation of the fluorometer), and the average

of the sum of the sampler fluorescence signal (cyclone plus filter) to the total dust filter fluorescence signal was 1.06 ± 0.08. On this basis, it is recommended that three active sampling heads be used with an external blank filter if prior checks of the operator and chamber show the blanks to be insignificant. A total filter sample may be run if desired, but these results suggest that there is no significant effect due to the inlet geometry of the cyclone, a conclusion reached in more extensive studies in the literature (Breslin and Stein, 1975; Pickett and Sansone, 1973). If leakage through the filter is suspected, this should be checked thoroughly in a separate series of experiments using for comparison a filter of known high efficiency; inclusion of an additional filter in the sampling line may alter the pulsation effects of the pump/sampler system under test.

The sampler is now dismantled to facilitate extraction of collected uranine. Each sampler section with active collecting surfaces is rinsed twice with small amounts of buffer from a plastic wash bottle with a fine jet. All the rinsings are collected in a 30 ml beaker. The contents of the beaker are transferred to a 25 ml volumetric flask and the beaker rinsed into the flask with buffer. The flask is then brought up to the mark with buffer. A blank may also be run.

The flasks are thoroughly mixed and the fluoresence is measured by drawing a volume of about 1 to 2 ml through the fluorometer and then taking and recording a reading. Two readings on each flask may be useful for avoiding errors due to reading or bubbles in the fluorometer cell. The zero of the fluorometer should be set with the buffer solution drawn into the cell.

The penetration, or transmission, of the sampling head with the pump used is given by the fluorescence signal of the filter divided by the total fluorescence signal (sum of filter and cyclone values) with appropriate blank corrections. These values should be determined at eight evenly spaced points over the range of 2.25 to 7.50 micrometers aerodynamic diameter. Under conditions of insignificant inlet effects and no filter leakage (as discussed above) this penetration measurement is equivalent to the collection efficiency.

RECOMMENDED IMPROVEMENTS

The port for attachment of the Climet optical particle counter should be at the bottom of the chamber, perhaps in the removable end plate. The Climet's intake flow rate is quite high and this may perturb the flow past the samplers, although there was no evidence of this in the data taken. The Climet does not need to sample continuously, and could be placed in calibration mode, where no sample is drawn, for periods between checks of the particle distribution.

A larger diameter cylinder for the chamber would permit more samplers to be tested at one time, if desired. In particular a chamber of about 20 cm. diameter would allow a neater arrangement of three Bendix units in the chamber.

C. SAMPLING UNIT ACCEPTANCE CRITERIA

The experimental procedure detailed in Sections III-A and III-B above provide data characterizing both random and systematic errors of the candidate sampling unit. These data are required for calculation along the lines of Section II for reaching a decision as to certification.

Explicitly, the precision experiment (Section III-A) yields replicate dust concentrations which are the input necessary for the computation of the program of Appendix B. The result of the calculation is a pooled relative standard deviation CV_t. For a sample computer run, see Section IV-A.

The bias experiment (Section III-B) provides 3 sets of monodisperse data at (nominal) aerodynamic diameters equal to 2.25, 3.00, 3.75, 4.50, 5.25, 6.00, 6.75, and 7.50 micrometers. For the specific case of the 10 mm cyclone, each set of uncalibrated data gives a value for $var(D_{cut})$ by using the program of Appendix A. Of the three numbers so obtained, the intermediate value is to be used as input to the certification decision calculation for both cyclone and general sampling unit certification. In order to represent the certification laboratory's accuracy reasonably, it is recommended that the cyclone experimentation giving the estimate of $var(D_{cut})$ be conducted within a year of the candidate sampling unit certification testing. The eight particle size bias results are combined as described in Section II-F.

The certification decision is carried out using the program of Appendix C. Required input is CV_t, $var(D_{cut})$, and any one of the three sets of monodisperse data. (If one of the three sets appears to indicate malfunction of the corresponding sampling unit, these data would not be used in the calculation.) Upon input of these numbers, computation of sampler compliance is carried out at the test dust distributions indicated in Section II-B. Each dust distribution for which 25 percent accuracy at the 95 percent confidence limit is not attained is indicated. After this computation, an attempt may be made to improve the calibration constant (see Section II-G). Subsequently, calculations at the test dust distributions are again carried out, now using recalibrated data. The candidate sampling unit is to be considered not certified (at the relevant calibration constant) if the unit is out of compliance at any one of the test dust distributions.

IV. SAMPLE RESULTS AND DISCUSSION

A complete run-through of the precision and bias test was performed on MSA Model G Sampling Units. The precision test was also tried on the Bendix CMDPSUs, but encountered difficulties. The results are presented here as an example of the accuracy test methods, showing how these methods can be adapted to routine CMDPSU testing.

A. PRECISION TEST

The method recommended for the precision test (Section III.A.) evolved over several attempts to achieve the CV_t values reported for the 10 mm cyclone in the literature (Table II-6). The first attempt at the precision test had a CV_t averaging twenty percent. In subsequent measurements, the following improvements were adopted:

* replace the Wright Dust Feeder with a fluidized bed aerosol generator;

* replace the large Standard Coal Aerosol Test System (8 sampler capacity) with the smaller Standard Instrument Calibration System (4 sampler capacity);

* install a 200 mCi titanium tritide charge neutralizer;

* increase the dilution air into the dust chamber to assure that no sampler was depleting the dust concentration around the inlet;

* control the humidity in the balance environment with a glove box in equilibrium with a saturated calcium nitrate solution (Figure III-4);

* replace the calibration procedures using the microbalance's mechanism with mathematical calibration against a standard weight;

* develop a quality control system for the weighing procedure, using replicate weighings of the filter blanks;

* develop methods for re-sealing the CMDPSU's filter cassettes without creating leaks or artificial weight gains;

* develop a nested experimental design to account for both position effects and inter-sampler variability.

With these improvements, the final precision measurement on the MSA model of the CMDPSU gave a CV_t estimate of 2.1 percent, which is the lowest value reported to date.

Review of these results within NIOSH led to further changes in the precision test method. The experimental designs recommended in Sections II.C. and III.A. have been completely revised from the design used in the trial runs. In addition, the procedures for re-sealing the filter cassettes were found to be a potential source of large errors, and have

been dropped in favor of using the cassettes sealed by the manufacturer. Since this new procedure means that the manufacturer's pre-weight must be used in the precision test, we recommend that manufacturers pre-weigh the filter capsules to the full 0.01 mg sensitivity of their balances, rather than truncating the weights to 0.1 mg as they currently do.

The recommended experimental procedures in Section II.A. have yet to be tested in full. The results reported here are from the final trial precision test performed on the MSA model of the CMDPSUs, as well as the difficulties encountered with precision testing the Bendix model. This test consisted of six runs in the four-sampler chamber, using a total of 12 CMDPSUs. The target concentration was 1.0 mg/m^3 throughout this test.

APPARATUS

The dust chamber used in the final precision measurement is the modified version of the four-sampler chamber made by Fairchild, Tillery and Ettinger (1977), shown in Figures III-1, -2, -3, and -6. As shown in Figure III-6, the dust chamber holds four CMDPSUs in a symmetric array about the dust inlet.

The primary modification to the original chamber was replacing the Wright dust feeder with the TSI Fluidized Bed Aerosol Generator (Model 4300). The fluidized bed generator was found to be more reliable in operation, and could reproduce the dust concentation better. The cyclone pre-selector on the dust generator was removed to broaden the dust sizes to which the samplers were exposed during the precision test. Another addition to the original chamber was the GCA Respirable Aerosol Monitor (RAM) for following the dust concentration during a run.

EXPERIMENTAL DESIGN

The experimental design used in this trial run of the precision measurement is given in Table IV-1. This design is similar to the one recommended for dust chambers with position effects (Table III-8). The differences are that the entire design is executed at a single target concentration, but with three experiments for the given target. In the trial measurement reported here, the target concentration was 1 mg/m^3. As with the other designs recommended in Section II.C., the order of runs is randomized to prevent biases from developing in the course of the measurement.

WEIGHING ACCURACY

The accuracy in the filter weights was monitored and controlled by the procedures outlined in Section III.A. The most recent QC charts are duplicated in Figure IV-1 and Figure IV-2.

In these charts, the primary difference from the procedures in Section III is the use of a "long-term blank" filter. Unlike the blanks prepared for each run, the long-term blank was not sealed a cassette, but was stored permanently in the glove box. The long-term blank, especially for the MSA

Table IV-1. Experimental Design for the Trial Precision Test. Samplers are labeled A through L.

Experiment	Runs in Experiment	Positions 1	2	3	4	Order of Runs
1	1	A	B	C	D	I
	2	A	B	C	D	III
2	1	E	F	G	H	II
	2	E	F	G	H	VI
3	1	I	J	K	L	IV
	2	I	J	K	L	V

Figure IV-1. Control Chart for Replicate Weighings on the MSA Filter Capsules. Out-of-control situations are circled.

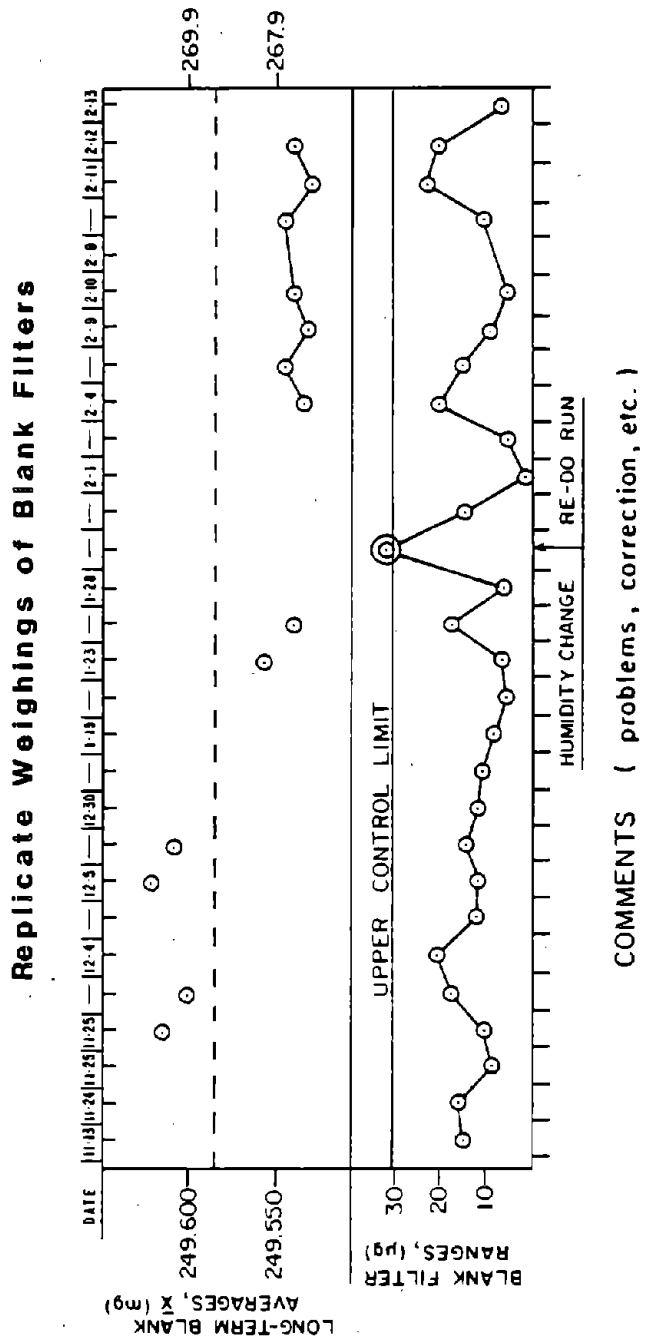
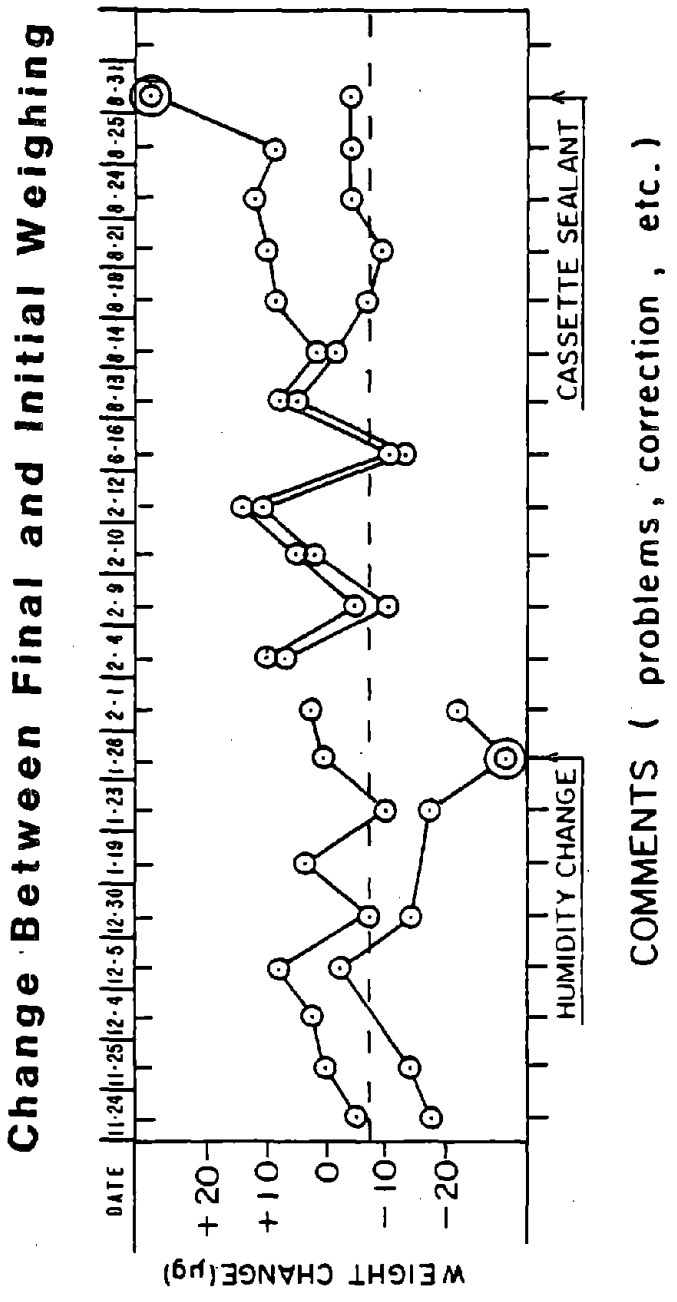


Figure IV-2. Control Chart for Repeated Weighings of the MSA Filter Capsules. Out-of-control situations are circled.



filters, was not a satisfactory control on the consistency of the balance calibration, because the constant handling of the filter occasionally caused sizable gains or losses. In Figure IV-1 for example, long-term blank 2686 dropped 0.053 mg in weight between days 12/5 and 1/23.

Therefore, the QC procedures in Section III.A.4 recommend a 200 mg NBS standard weight. In addition to greater stability under handling, an NBS-calibrated weight will make the weighing accuracy traceable to the NBS primary standards. Traceability will be especially important when manufacturer's preweights are used in the precision test.

The main application of these control charts at the time of the precision tests was to control the within-day reproducibility of the weighings, as measured by the range R between the initial and audit weights on a single day. On the one occasion (day 1/28 in Figure IV-3) when the range exceeded the upper control limit, the $Ca(NO_3)_2$ solution in the humidity control system was found to be depleted, so the entire run was repeated.

The over-all accuracy of the filter weights can be computed from the QC data, as derived in Section II.C. The results for the MSA filter weights are given in Table IV-2, and have been sub-divided into the periods before and after the mathematical calibration of the balance was instituted. From these statistics can be estimated S_M, the standard deviation in the dust weight M_S. Using Eq. II-45, the weighing precision with mathematical calibration for the MSA filters is:

$$S_M = \sqrt{(S_{pool})^2 + (S_\Delta)^2} = 12.9\ \mu g \qquad \text{(IV-1)}$$

The second use for the statistics in Table IV-2 is to check for biases in the weighing procedures. Bias due to changes in the operators and/or the balance would show up in the difference between the replicate weighings (d). Bias due to the filter treatment shows up in the difference in the repeated weighings (Δ). Using a two-tailed t-test, both means $\bar{d}$ and $\bar{\Delta}$ during the mechanical calibration period are significantly different from zero at the 95 percent confidence level, indicating biases. With the mathematical calibration, both means are negligible at the 90 percent confidence level. Thus, both forms of weighing bias were reduced by the mathematical calibration procedures recommended in Section III.A.4.

Finally, these results can be used to calculate control limits for future weighing of MSA filters with DPSE's microbalance. Using Equations II-46, the upper control limit on the range R of replicate weighinbs is 30.6 μg. The control limits on the difference Δ in repeated weights are $\pm$30.9 μg.

Table IV-2. Accuracy Statistics for the Weighing of MSA Filter Capsules. Weight parameters are given in micrograms, and are defined in Section II C

		Mechanical Balance Calibration	Mathematical Balance Calibration	Total
Replicate Weight Statistics	n	15	44	59
	$\overline{d}$	+5.0 µg	+1.5 µg	+2.4 µg
	$\overline{R}$	9.7 µg	8.8 µg	9.0 µg
	S_{pool}	8.2 µg	7.5 µg	7.7 µg
Repeated Weight Statistics	n	8	22	30
	$\overline{\Delta}$	10.1 µg	-2.6 µg	-4.6 µg
	S_{Δ}	10.2 µg	10.3 µg	10.7 µg

DUST CHAMBER CHARACTERIZATION

For the precision measurement, the important properties to monitor in the dust chamber are the air flows and the dust concentration over time and over sampling positions. Less important to the outcome are the dust size distribution and chamber velocities. Since these latter quantities potentially affect CMDPSU bias, they are reported for completeness, but their measurement is not recommended for routine certification testing.

A typical air flow balance is given in Table IV-3. The input air flows are measured with rotameters and checked with a bubble meter. The chamber had been tested for leaks, and completely sealed except for the single exhaust port. The important thing about the air flow balance is that the air inputs are substantially larger than the outflows, assuring that no sampler is depleting the dust around the inlet. This rule was not always followed. In the early trial runs of the precision test, a negative net air flow was allowed to develop, and the resulting CV_t was sometimes as large as 20 percent. Therefore, the net air balance is an important quantity to monitor in minimizing the chamber's effects on the sampler precision measurement.

The Respirable Aerosol Monitor (RAM) was used to monitor the dust concentration during a run. Since the RAM's read-out did not equal either total dust or respirable dust concentration, we treat the RAM output as a relative indication of dust concentration.

A strip chart recording for a 5 hour run is shown in Figure IV-3, with adjustments of the fluidized bed aerosol generator marked on the chart. Over short periods (~ 1/2 hour), the dust concentration varied by only ~10 percent, but longer-term drift in the dust concentration required adjustments, which were hard to make precisely. Therefore, the dust concentration could not be controlled exactly in this test. We recommend adjusting the concentration as seldom as possible, keeping the RAM reading within 2 mg/m^3 of the target concentration.

Despite these variations, the fluidized bed generator gave a more stable dust concentration and is more reliable than the Wright Dust Feeder and other generators we have tried.

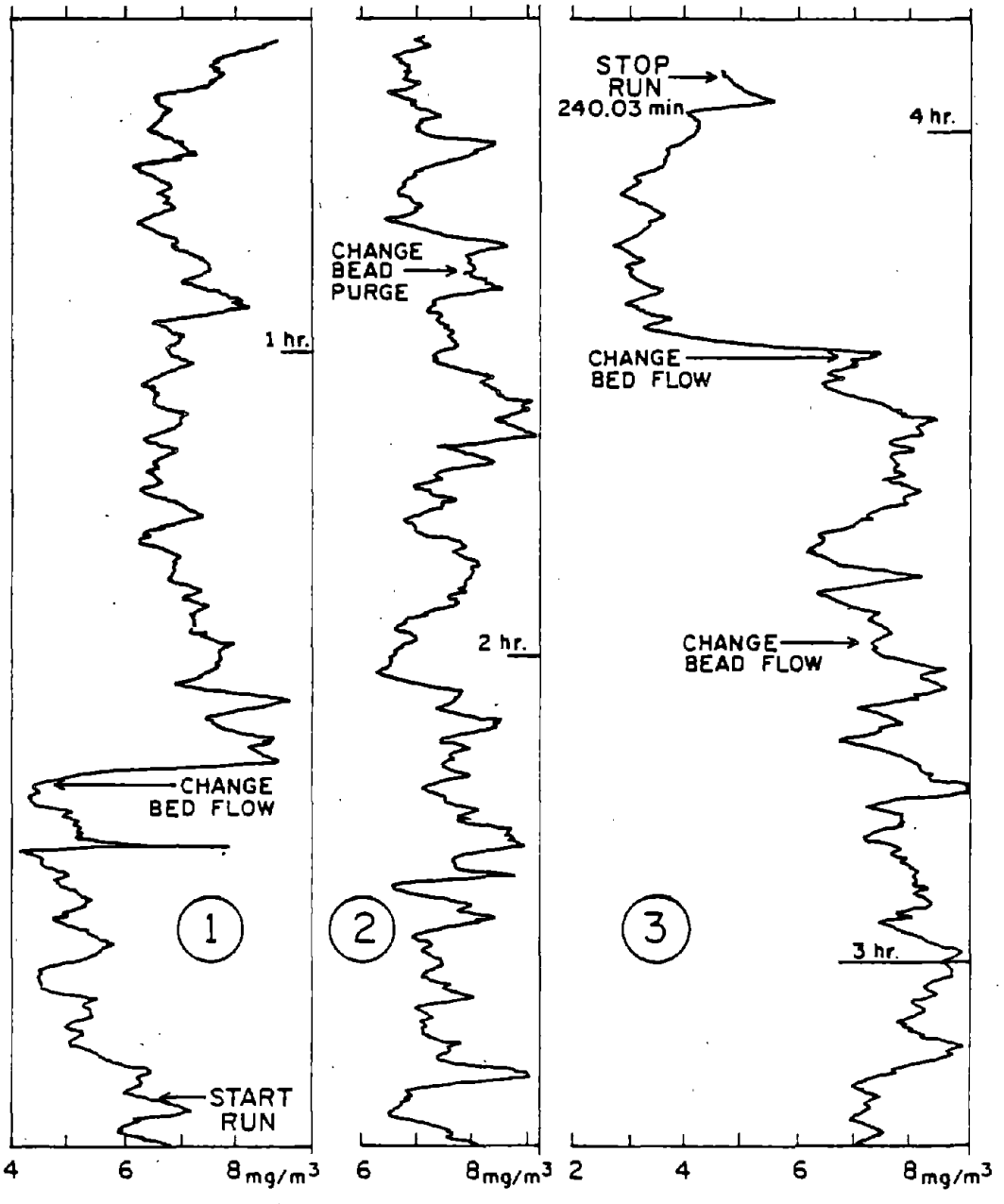
The RAM was also used to test the dust concentrations at the sampler inlets by use of a 10-inch-long probe made of copper tubing. Sample results from such a test give the following concentrations (averaged over 1/2 hour sampling periods):

Position	Mean Concentration
3	6.3 mg/m^3
2	6.5
3	6.5
4	7.1
2	7.0

Table IV-3. Air Flows in the Dust Chamber. Typical values for a run whose average CMDPSU concentration is 6.20 mg/m^3

SOURCE		FLOW RATE (L/min.) Rotameter	Bubblemeter
	AIR IN		
Fluidized Bed Aerosol Generator			5.9
Dilution Air		20.0	21.5
Total In			27.4
	AIR OUT		
4 CMDPSUs	4 x 2.0 =	8.0	
RAM		2.2	
Hygrometer		3.0	2.85
Total Out			13.05
Net Air for Exhaust			+14.3

Figure IV-3. Dust Concentration over Time during a Run. Strip chart recording made by the Respirable Aerosol Monitor (RAM). Average cyclone measurement was 6.20 mg/m^3 (MRE equivalent).



The variation due to drift in the concentration monitored by the RAM makes such a test difficult to interpret. We have found that the fluctuations less than 1 mg/m^3 between positions (as shown here) lead to little position effect in the over-all precision experiment, while ranges over 2 mg/m^3 should be avoided.

The air velocity in the dust chamber is primarily a function of the amount of input air. When enough dilution air is added to assure a positive net influx, the velocity in the chamber fluctuates between 0-30 ft/min. This range of velocities is too low to have significant inlet effects.

The dust size distribution was determined both by counting on a filter with a Scanning Electron Microscope and by the new Aerodynamic Particle Sizer (Baron, 1983). For the SEM count, a sample was taken on a Nucleopore filter in an open-faced cassette. The dust particles on the filter were sized and counted with NIOSH's SEM-image analysis system operating at 400X. The conversion from projected diameter to aerodynamic diameter used only the density of coal, but assumed a shape factor of one (see Eq. II-10). The APS was used to sample dust directly from the Fluidized Bed Aerosol Generator's vertical elutriator. The probit plots of the number size distributions are shown in Figure IV-4. The mass median diameters are determined by the Hatch-Choate equation (Eq. II-13).

In Figure IV-4, not only do the median diameters disagree, but the geometric standard deviations are different as well. This discrepancy might indicate some bias due to the different sampling methods. Because the SEM samples were taken directly from the chamber with less chance of sampling bias, we hold this method to be more dependable. The SEM dust size parameters:

$$\text{MMD} \simeq 6.34\ \mu\text{m}$$
$$\text{GSD} = 2.16$$

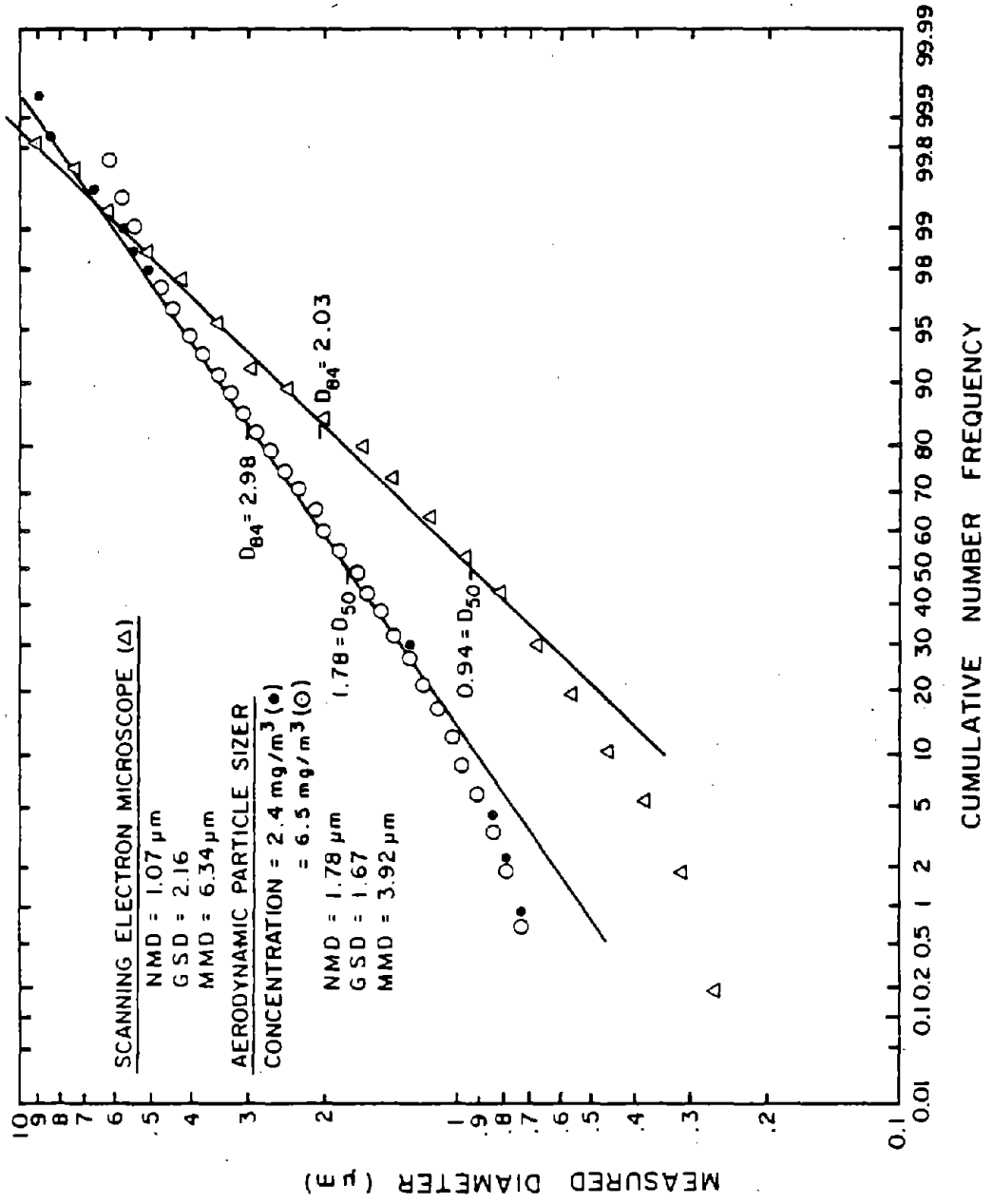
lie within the range of coal mine dust distributions shown in Figure II-5.

CMDPSU PERFORMANCE

The precision tests were all done with old MSA and Bendix CMDPSUs obtained from various branches of NIOSH and MSHA. The pumps were repaired (when necessary) and calibrated by DPSE's Maintenance and Calibration Laboratory. On the sampling heads, all O-rings were inspected and replaced where necessary. On the MSA units, the sampling heads (see Figure III-5) had plastic collars, rather than metal ones.

The only adaptation required in the CMDPSUs was re-assembling and sealing the filter cassette after the filter capsule had been pre-weighed on the microbalance. This step was deemed necessary in order to improve weighing precision over the 0.1 mg sensitivity provided by the manufacturers pre-weight.

Figure IV-4. Number Distributions of the Coal Dust from the Fluidized Bed Aerosol Generator.



The method of re-sealing the cassettes had to meet two conditions. The sealing method could not affect the capsule's weight, nor could it allow air or dust leaks noticeably greater than found in cassettes sealed by the manufacturer. The weight stability was tested by comparing the weight changes of filters which had been sealed in the cassettes with the weight of control filters stored in the balance's glove box. Additional checks were provided during the precision measurements by Quality Control charts for the blank filters which had been sealed in cassettes (Sect. III.A.4). The leaks in the cassettes were tested by the method described in Section III.A.6.e.

On the basis of these tests, the MSA cassettes were sealed with a cloth-backed label tape. The shrinkable cellulose bands, often used to seal filter cassettes, caused an increase in the weight of MSA's aluminum-shielded filter capsules.

The Bendix cassettes, after much trial and error, were sealed with a combination of Silastic cement and the cellulose shrinkable bands (see Figure IV-5). This combination held -14" of H_2O negative pressure in the leak test, while other sealing systems were not leak-tight. This combination did not affect the weight stability of the Bendix filters, except for one blank filter which gained 0.03 mg after being sealed in the cassette. This anomalous weight gain may have been due to the transfer of some Silastic glue to the filter capsule during cassette assembly.

The final problem with the Bendix filters was attaching the capsule to the cassette. Bendix's previous capsule design allowed leaks around the hole where the cassette nib is inserted (Figure IV-6). Due to this fault, Bendix redesigned the capsules.

Another problem with the Bendix sampling head is dust accumulation at the junction between sampler head and cassette (Figure IV-7) in half of the samples. Due to the other problems with the Bendix precision tests, we cannot say whether this occasional dust loss affects CV_t significantly. However, the problem bears watching, and could be eliminated by a better fit between the sampler head, O-ring and cassette.

To minimize leaks occurring at this junction, we also ordered unassembled cassettes and capsules directly from Bendix, rather than disassemble the pre-weighed cassettes usually sold by Bendix. Despite these precautions, attaching the capsule to the cassette was still difficult in the Bendix design, and there is no guarantee that the cassettes assembled for the certification test are as leak-tight as those assembled by the manufacturer.

Considering all the problems with assembling and sealing the Bendix cassettes, routine certification testing should be done with cassettes assembled and sealed by the manufacturer. The manufacturer should also weigh the filter capsules to at least 0.01 mg in order to make the precision test meaningful at the 1.0 mg/m^3 concentration,.

Figure IV-5. Seal devised for the Bendix filter cassette. Under the cellulose shrinkable band shown here is a seal of Silastic cement.

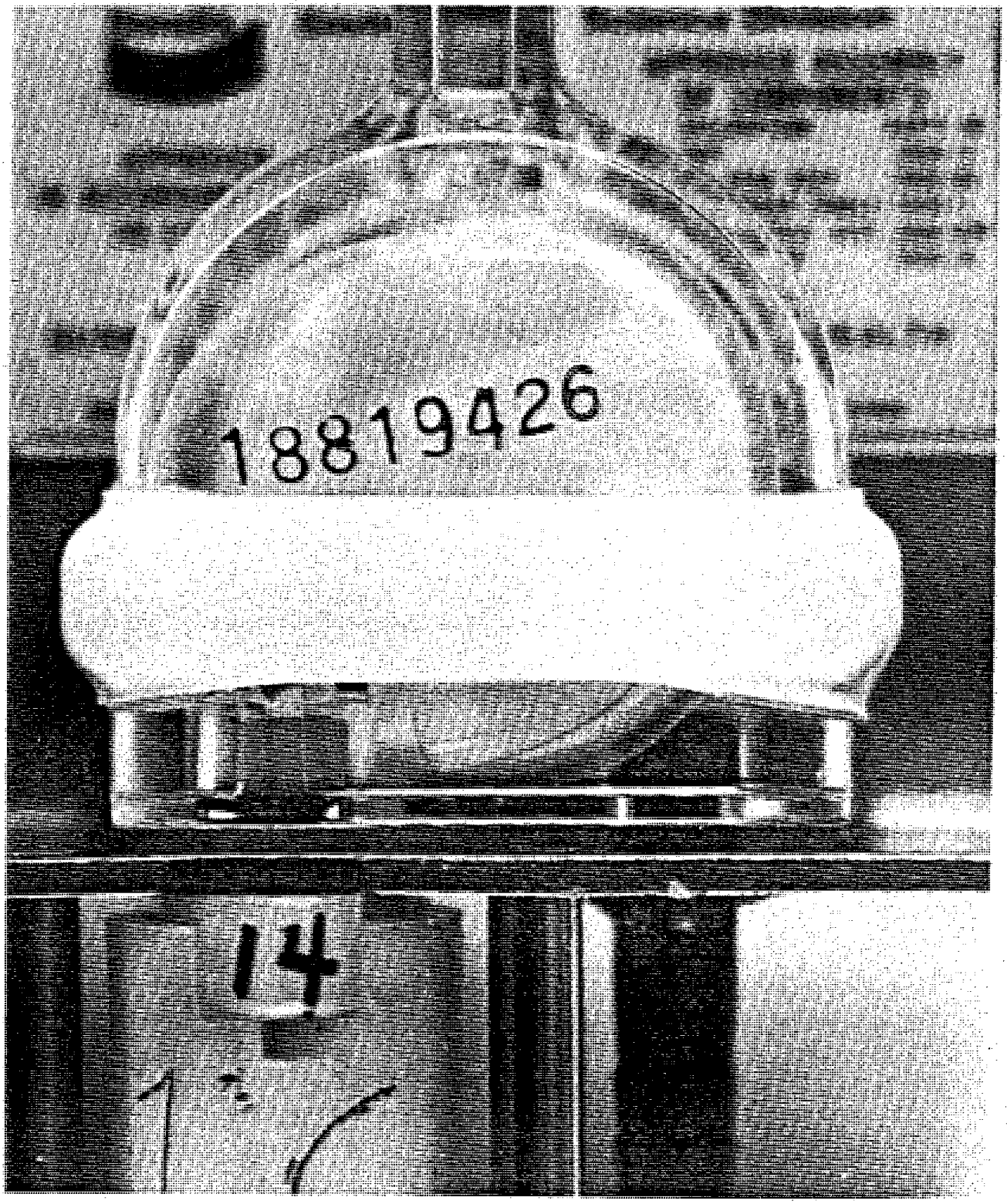


Figure IV-6. Bendix filter capsule, showing the hole through which the cassette's nib (in background) is inserted. In 1981, leaks were discovered at the junction between the hole and the nib, and Bendix has redesigned their capsules.

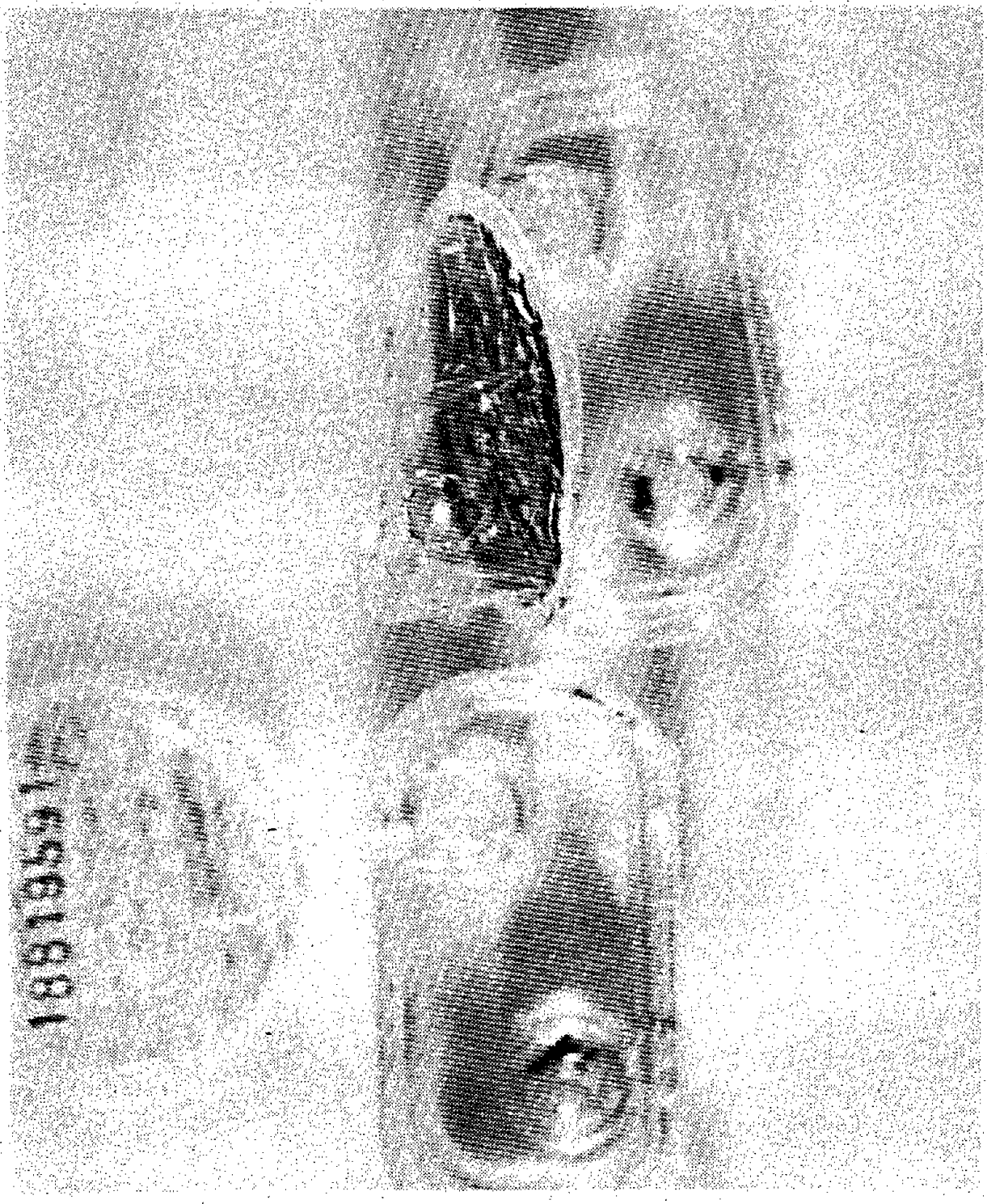
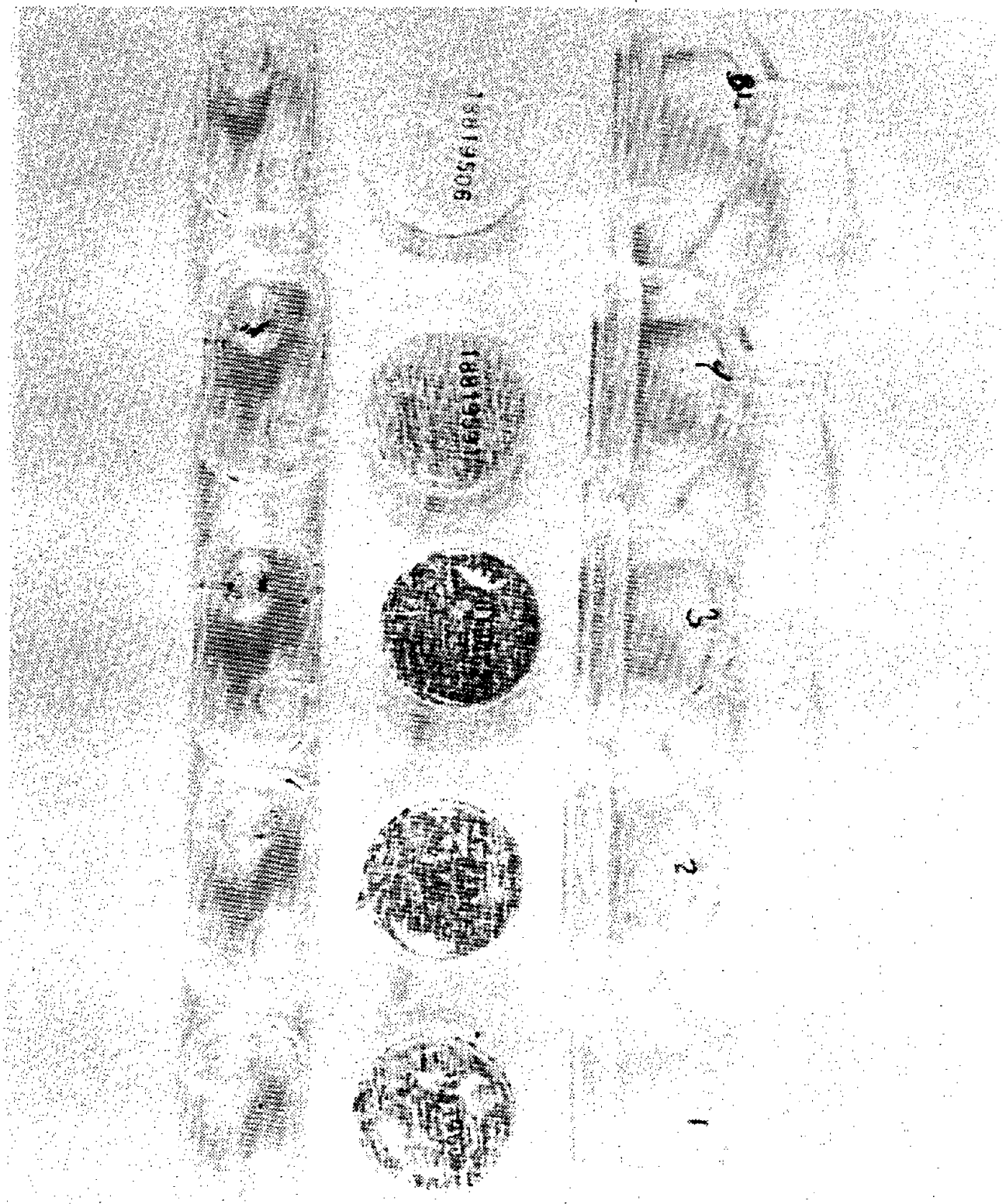


Figure IV-7. Bendix filter cassettes after a test run, showing dust accumulation in the wells of the second and third cassettes (from left) where the sampler head is joined. This accumulation was found in half of the samples taken.



Looking at the pump performance, the MSA Model G pump worked very well. Over 48 samples, the pump flow rates, as measured by the rotameters during the run, averaged 1.99 L/min, with an inter-pump coefficient of variation equal to 1.11 percent. The pooled intra-run CV in the flow rates was 1.57 percent. No MSA pump, which had been successfully repaired and calibrated, failed during a 5 hour run. In the certification testing, such statistics may be used to demonstrate compliance with pump reliability criteria, such as 30 CFR 74.3(a)(13).

The Bendix Micronair pumps, on the other hand, were unreliable and difficult to adjust accurately. During one test design with 24 samples, Micronaire pumps failed to hold their charge for a five-hour run, and had to be replaced. These pumps had been used previously in the field, but had been repaired before the precision test run, just as the MSA pumps were treated. The failures in the Micronair pumps could perhaps have been eliminated if new batteries or pumps were used.

How to treat the dust concentrations from these defective pumps would be a serious question if they had failed during actual certification testing. In NIOSH's present accuracy test (McCawley and Roder, 1975), this question has not come up because the CMDPSUs are powered by an external voltage source. The reliability of the pumps and their batteries is tested independently (McCawley and Roder, 1976).

We strongly oppose the use of an external power supply in the precision test. Since the pump batteries obviously affect the measurement of the dust concentration through the air flow rate, the batteries' performance is obviously part of the CMDPSU's overall precision. Therefore, we recommend that the CMDPSUs in the precision test be powered by the manufacturer's batteries, charged and maintained according to their instructions.

If the pumps still fail during the precision test, we recommend that the run be repeated once after the failed pump is repaired according to the manufacturer's instructions. Our rationale is that pump failure is easily detected during field samples, and the routine practice is to take another sample the next day. In calculating CV_t, the results of the second run should be used. However, the pump failure should be prominently noted in the public report on the certification decision for the information of CMDPSU manufacturers and purchasers.

PRECISION DATA AND STATISTICAL ANALYSIS

The data from the final precision measurement for the MSA Model G sampling units are shown in Table IV-4. These measurements were made with all the improvements in the test method discussed above. A gross CV estimate can be calculated from this data by pooling the CV for the individual runs. The resulting CV is 2.27 percent. This value contains position effects, and therefore is an upper bound on the true CV_t.

Table IV-4. Precision Test Data for MSA Model G. CMDPSU experimental design was run at a target concentration of 1 mg/cu.m.

Experiment	Run in experiment	Order of run	Concentration(mg/cu.m.) Pos. 1	2	3	4	Sampling Time(min)	Run CV
1	1	I	1.265	1.269	1.261	1.330	437.30	.0254
	2	III	0.725	0.737	0.724	0.741	477.38	.0116
2	1	II	0.754	0.804	0.794	0.794	476.65	.0286
	2	VI	1.201	1.297	1.258	1.280	480.00	.0334
3	1	IV	1.229	1.196	1.237	1.212	474.13	.0150
	2	V	1.272	1.307	1.274	1.284	482.02	.0125
							gross CV	.0227

A more sophisticated precision estimate may be obtained by an analysis of variance similar to those developed in Section II.C. The ANOVA model for this experimental design is given in Table IV-5. The terms are defined in Section II.C. The ANOVA results for the data in Table IV-4 are displayed in Table IV-6.

To calculate CV_t, we take a linear combination of the mean squares containing only the standard deviations for the inter-sampler variability σ_S and the intra-sampler variability σ_ε. By examination of Table IV-5, the precision estimate is given by:

$$(\hat{CV}_t)^2 \cong [MS(EXP*POS) + MS(Error)] / 2 \qquad (IV-2)$$

The mean squares are calculated by dividing the sums of squares from Table IV-6 by the corresponding d.o.f. The resulting estimate for CV_t is 0.0212. According to Satterthwaite's approximation (Eq. II-23), the degrees of freedom in this estimate are given by:

$$f* = \frac{[MS(EXP*POS) + MS(Error)]^2}{MS(EXP*POS)^2 / 6 + MS(Error)^2 / 9} \qquad (IV-3)$$

Thus, $\hat{CV}_t$ is estimated with approximate degrees of freedom $f* = 9.17$, which is truncated to 9 for the signifigance test. Using Eq. II-31, the confidence limits on the true CV_t are

$$0.0147 \leq CV_t \leq 0.039 \qquad (IV-4)$$

The inter-sampler variance may also be estimated from the ANOVA model in Table IV-4. The difference between estimated mean squares gives:

$$(\sigma_S)^2 \cong [MS(EXP*POS) - MS(Error)] / 2 \qquad (IV-5)$$

So, the coefficient of variation due to inter-sampler variability is:

$$CV_{inter} \cong \sigma_S \cong 0.0162 \qquad (IV-6)$$

Thus, the inter-sampler error for the MSA units is very small, especially compared to the precision found in the field (Bowman, Bartley, Breuer, and Shulman, 1983).

Further examination of the ANOVA results in Table IV-4 demostrate:

1) Significant position effects. The F-test for the POS term in the ANOVA gives $PR>F = 0.0353$, a small error for assuming the existence of position effects (confounded with other terms). The Duncan's Multiple Range Test by position (Table IV-7) reinforces this conclusion, and shows that position 1 has the significantly lower mean dust concentrations.

Table IV-5. Analysis of Variance Formalism for the Experimental Design in Table IV-1. The response variable is LNC = ln [C(mg/m^3)].

SOURCE: Busch (1981)

Source of Variation	Degrees of Freedom	Expected Mean Squares
EXP = experiments (confounded with S = samplers)	2	$8(\sigma_E)^2 + 4(\sigma_R)^2 + 2(\sigma_S)^2 + (\sigma_\epsilon)^2$
POS = positions (confounded with S = samplers)	3	$6\ \theta(POS) + 2(\sigma_S)^2 + (\sigma_\epsilon)^2$
EXP*POS interaction	6	$2(\sigma_S)^2 + (\sigma_\epsilon)^2$
RUN(EXP) = Runs in experiments	3	$4(\sigma_R)^2 + (\sigma_\epsilon)^2$
Residual error, RUN*POS(EXP) confounded with RUN*S(EXP)	9	$(\sigma_\epsilon)^2$
Total	23	

Table IV-4. Analysis of variance for the final precision run, showing 1) the sum of squares for the residual ERROR term, 2) the sum of squares for the EXP*POS term, and 3) the F-test demonstrating significant inter-sampler variation.

GENERAL LINEAR MODELS PROCEDURE
CLASS LEVEL INFORMATION

CLASS	LEVELS	VALUES
EXP	3	1 2 3
POS	4	1 2 3 4
RUN	2	1 2

NUMBER OF OBSERVATIONS IN DATA SET = 24

GENERAL LINEAR MODELS PROCEDURE

DEPENDENT VARIABLE: LOGC

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE
MODEL	14	1.40001367	0.10000098
ERROR	9	0.00166922[1]	0.00018547
CORRECTED TOTAL	23	1.40168289	

MODEL F =	539.18	PR > F = 0.0001

R-SQUARE	C.V.	STD DEV	LOGC MEAN
0.998809	21.9356	0.01361870	0.06208503

SOURCE	DF	TYPE I SS	F VALUE	PR > F
EXP	2	0.31724206	855.24	0.0001
POS	3	0.00333498	5.99	0.0158
EXP*POS	6	0.00426338[2]	3.83	0.0353[3]
EXP(RUN)	3	1.07517324	1932.35	0.0001

Table IV-7. Position effects in the final precision test. Position 1 has mean concentrations significantly lower than positions 2 and 4.

GENERAL LINEAR MODELS PROCEDURE

DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE LOGC

MEANS WITH THE SAME LETTER ARE NOT SIGNIFICANTLY DIFFERENT.

ALPHA LEVEL=.05 DF=9 MS=1.9E-04

GROUPING	MEAN	N	POS
A	0.073977	6	4
A			
A	0.070279	6	2
A			
B A	0.060571	6	3
B			
B	0.043512	6	1

2) The residuals lack homogeneity. The plot of the residuals versus LNC (Figure IV-8) shows that concentrations were bunched into two groups, 0.7-0.8 mg/m^3 in runs II and III and 1.2-1.3 mg/m^3 in the other runs. Unfortunately, the residuals at the large concentrations show greater variability than those at the lower concentrations. This may indicate inhomogeneity in the variances, which would violate one of the ANOVA's postulates. This possibility is discussed more below.

DISCUSSION

First, an analysis of errors is done for the precision measurement to determine the factors which contribute to the 2.12 percent $\hat{CV}_t$. The weighing errors (CV_A) in the precision measurement can be estimated from Eq. II-51:

$$CV_A = s_M / M_S \qquad \text{(IV-7)}$$

$$= .0129 / .776 = 0.0173$$

where s_M is taken from Eq. IV-1 and M_S is calculated from the average of the 24 dust samples in the latest precision test. Combining this weighing error and the inter-sampler variation CV_{inter} estimated in Eq. IV-6 gives:

$$\sqrt{(CV_{inter})^2 + (CV_A)^2} = 0.0237 \qquad \text{(IV-8)}$$

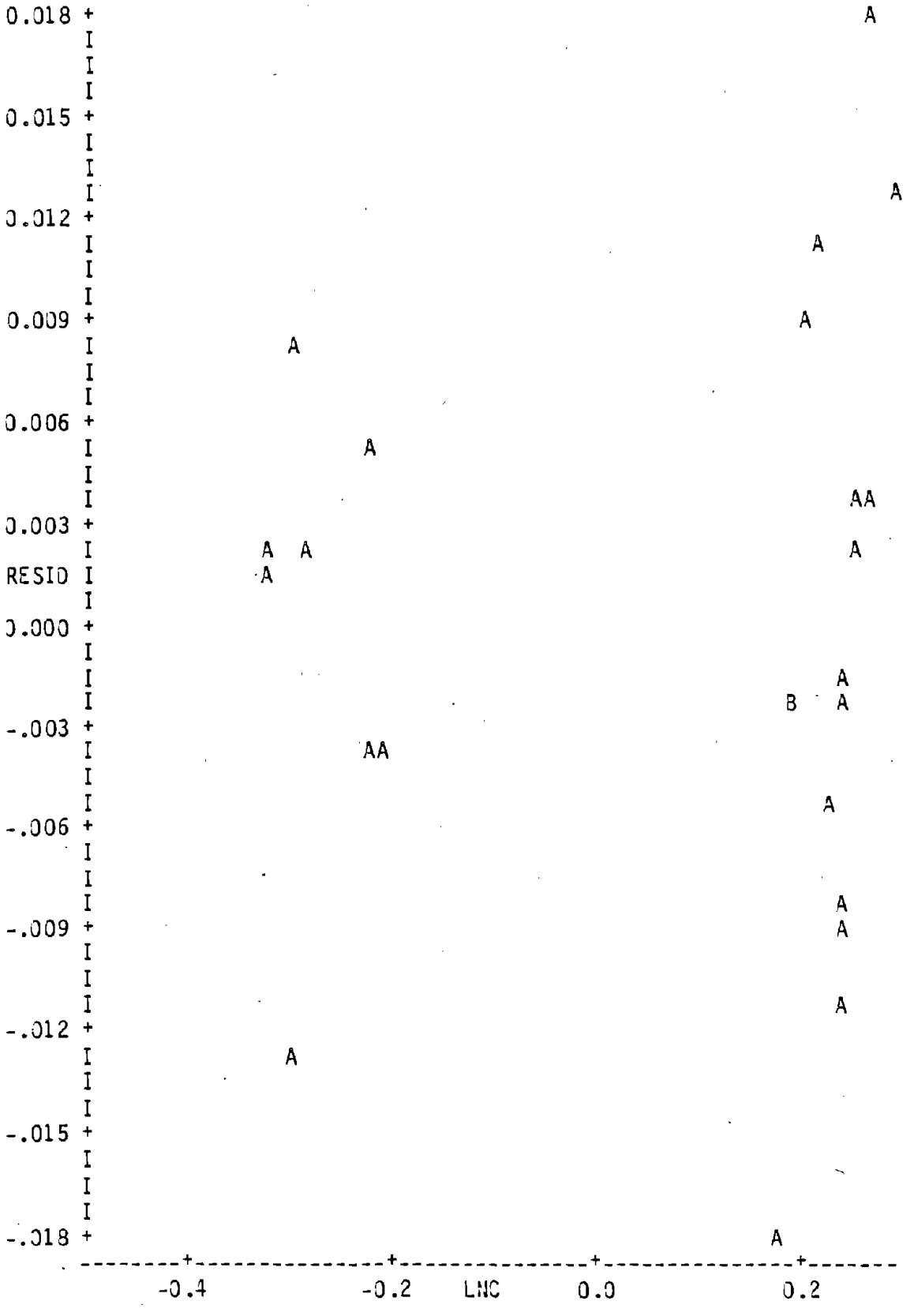
which completely accounts for the $\hat{CV}_t = 0.0212$.

The final potential component in $\hat{CV}_t$ is errors in the analysis of variance. The variance inhomogeneity discovered in Figure IV-7 would indicate some error in the ANOVA. However, $\hat{CV}_t$ cannot be smaller than the independently determined analytical error, CV_A = 1.7 percent, which allows only 0.5 percent error on the down-side. An upper bound on $\hat{CV}_t$ is set by the gross CV derived by pooling the run CVs (Table IV-5). The difference between the gross CV and the estimated $\hat{CV}_t$ allows only a 0.15 percent error in the ANOVA on the up-side. Therefore, any errors in the ANOVA could not make the precision estimate much different.

The analysis of errors therefore supports the $\hat{CV}_t$ estimate of 2.12 percent with an upper confidence limit of 3.9 percent. Compared to the other precision measurements in the literature (Table II-6), this $\hat{CV}_t$ value is by far the lowest value reported for 10 mm cyclone samplers.

In comparison to other forms of industrial hygiene sampling (Gunderson and Anderson, 1980), such precision is excellent. With strict quality controls, the random errors with the MSA sampling units in laboratory tests can be as small as the weighing errors allow.

Figure IV-8. ANOVA residuals (RESID) vs. the log of the concentrations.



We attribute this improvement in the cyclone precision estimate to our efforts in controlling the chamber effects and reducing the weighing errors. In comparison to the NIOSH's present accuracy test for CMDPSUs (McCawley and Roder, 1975), the precision test developed here has several advantages. First, the experimental design allows the measurement of CMDPSU precision free from run effects and position effects. The analysis of variance estimates CV_t with well-established confidence limits, and includes regular checks on the statistical assumptions. The establishment of quality control measures assures the weighing accuracy. Finally, the fluidized bed aerosol generator is a more reliable replacement for the Wright Dust Feeder currently used.

On the other hand, several gaps still exist in the experimental verification of the precision test. A satisfactory precision measurement on the Bendix CMDPSU has not been completed. The most recent attempt at a precision measurement with Bendix CMDPSUs gave CV_t = 17.3 percent at 4.0 mg/m^3. As discussed above, some of the Bendix Micronair pumps failed at times during this test. Also, we had difficulty in sealing the filter capsule to the cassette. To avoid this problem, the filters for the certification test should be pre-weighed to 0.01 mg and sealed into the cassettes by the manufacturer. Finally, the statistical analysis showed that run-position interactions existed during the measurement with the Bendix CMDPSUs, due to a negative net air flow in the chamber. Therefore, the precision of the Bendix sampler was not determined independent of the run-position interaction. To truely assess the Bendix CMDPSUs, another measurement should be done on new samplers, using the improved procedures recommended in Section III.A.

With the MSA sampling units, the precision has only been measured at the target concentration of 1 mg/m^3. Of course, the analytical precision (Eq. IV-7) would be smaller at the 4 mg/m^3 target used in the full experimental designs (Tables II-7 and II-8). Although the smaller analytical precision at 4 mg/m^3 would suggest that CV_t would be smaller as well, this must still be proven experimentally.

In addition, the new experimental designs in Tables II-7 and II-8 have not been tried out. In particular, we recommend that the experimental designs for chambers without position effects be validated promptly. Our experience with the statistical corrections for position effects shows that run-position interactions can easily creep into the results, leading to an inflated estimate of CV_t. Now that a chamber design without significant position differences is availible (Rubow and Marple, 1983), the designs in Table II-7 seem the most promising for the precision measurement.

B. COLLECTION EFFICIENCY

Three MSA coal mine dust personal sampling units (CMDPSU) were tested by the technique given in Section III-B. The sampling units under test consisted of (1) a 10 mm nylon cyclone to separate respirable dust from total dust, (2) a filter cassette to collect the respirable dust, (3) a metal bracket to clamp together the cyclone and the filter cassette and to attach the sampling head assembly (cyclone, cassette, and bracket) in the breathing zone of the worker, (4) a 36" flexible plastic 1/4" ID tube with clips to connect the sampling head assembly to the pump, and (5) a personal sampling pump, MSA Model G, to pull air through the sampling head assembly and tubing at the required rate (currently 2.0 liters per minute for coal dust sampling). The samplers were labled A, B, and C. All the pumps were previously calibrated to 2.0 L/min ±5 percent. The data obtained are given in Table IV-8. Also shown are values for the calibrated collection efficiency, using k=1.38 as presently recommended by MSHA. The data are plotted in Figure IV-9. The 50 percent penetration point is seen to be about 3.2 micrometers aerodynamic diameter.

Table IV-8. CMDPSU Penetration Measurements

Run No.	Aerodynamic Diameter	Cyclone	Penetration	Calibrated Penetration (k=1.38)
9	1.88	A	0.950	1.31
		B	0.952	1.31
		C	0.952	1.31
3	2.46	A	0.930	1.28
		B	0.919	1.27
2	3.23	A	0.520	0.718
		B	0.496	0.689
7	3.49	A	0.507	0.700
		B	0.409	0.564
		C	0.456	0.629
1	3.95	A	0.218	0.301
		B	0.217	0.299
6	4.85	A	0.055	0.0759
		B	0.048	0.0662
		C	0.041	0.0566
4	5.48	A	0.037	0.0511
		B	0.033	0.0455
		C	0.042	0.0580
5	5.92	A	0.037	0.0511
		B	0.035	0.0483
		C	0.036	0.0497
8	9.01	A	0.0071	0.0098
		B	0.0056	0.0077
		C	0.0059	0.0081

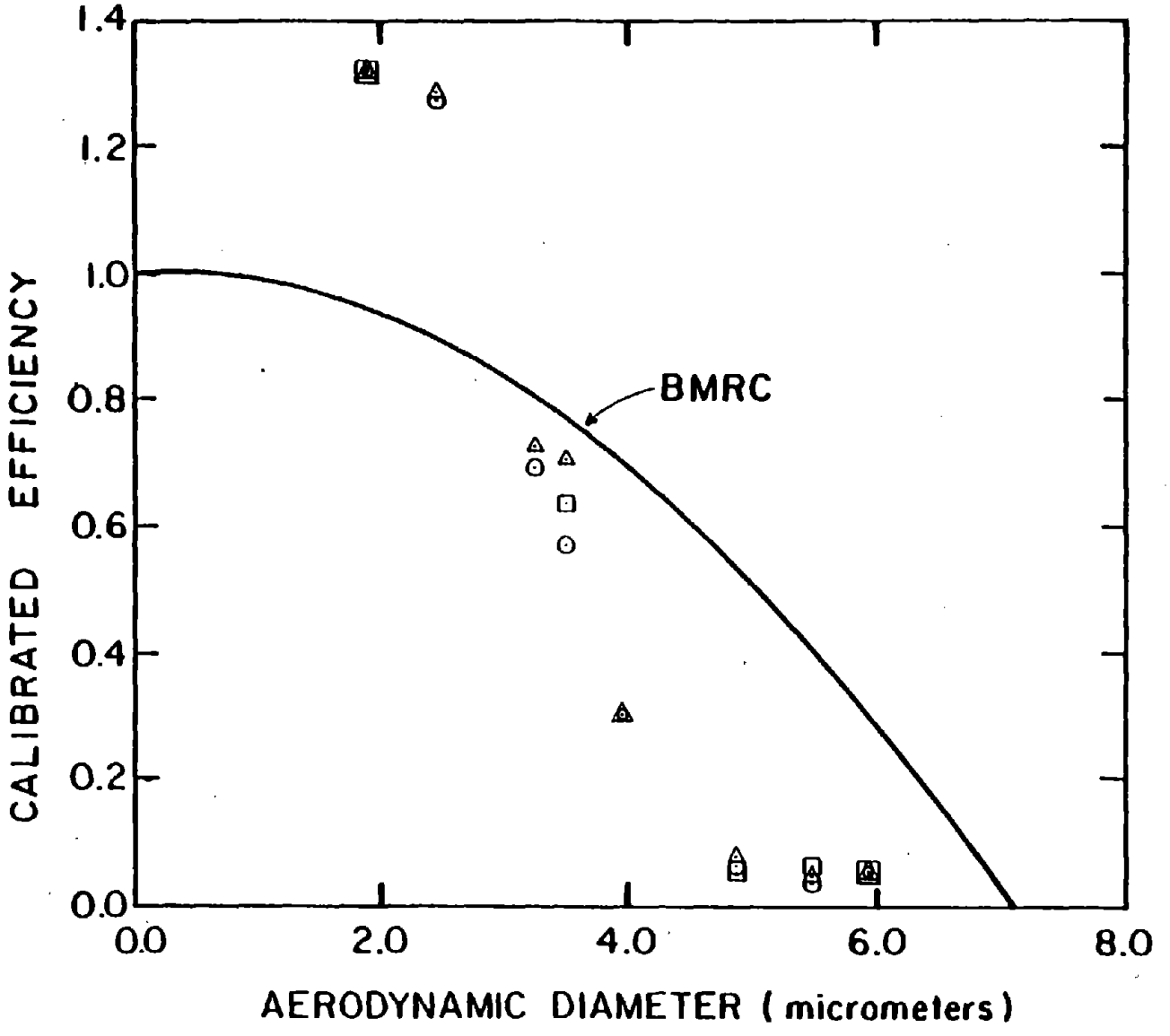


Figure IV-9: Calibrated transmission efficiency data from bias test of cyclone operated at 2.0 L/min using calibration coefficient k=1.38. For comparison the BMRC respirable dust definition is also plotted.

C. ACCURACY COMPUTATION

In this section, use of the TSO (interactive) computer programs for the calculation of accuracy and the certification decision are demonstrated. To this aim, the experimental data obtained during the sample runs described in Sections IV. A. and B. are employed. As shown below these data are stored in a data set, which is called here "Q20.DATA," and which must be allocated, as indicated, to FT04F001. As a record of the calculation results is probably desired, another data set, designated here as "OUT.DATA," is created and similarly allocated to FT07F001. Otherwise, the output is routed directly to the terminal. For the sake of clarity in the sample computer session presented, quantities which must be typed by the computer operator are underscored so as to be distinguished from statements printed by the computer. After logon, the computer prints:

```
READY
?
?ALLOCATE F(FT04F001) DA(Q20.DATA) NEW SPACE(10) TRACKS

READY
?
?CALL CYCLONE


THIS PROGRAMS COMPUTES CYCLONE PARAMETERS GIVEN
N MONODISPERSE DATA PAIRS:
(DIAMETER (MICROMETERS), (FRACTIONAL) EFFICIENCY)

N =
?
?8

D =  SAMPLING EFFICIENCY =
?
?1.88 0.95

D =  SAMPLING EFFICIENCY =
?
?2.46 0.93
D =  SAMPLING EFFICIENCY =
?
?3.23 0.52
D =  SAMPLING EFFICIENCY =
?
?3.49 0.507
```

```
D =  SAMPLING EFFICIENCY =
?
?3.95 0.218
D =  SAMPLING EFFICIENCY =
?
?4.85 0.055
D =  SAMPLING EFFICIENCY =
?
?5.48 0.037
D =  SAMPLING EFFICIENCY =
?
?5.92 0.037

MONODISPERSE CYCLONE DATA

N =     8

DIAMETER =    0.188000E+01 PENETRATION =    0.950000E+00
DIAMETER =    0.246000E+01 PENETRATION =    0.930000E+00
DIAMETER =    0.323000E+01 PENETRATION =    0.520000E+00
DIAMETER =    0.349000E+01 PENETRATION =    0.507000E+00
DIAMETER =    0.395000E+01 PENETRATION =    0.218000E+00
DIAMETER =    0.485000E+01 PENETRATION =    0.550000E-01
DIAMETER =    0.548000E+01 PENETRATION =    0.370000E-01
DIAMETER =    0.592000E+01 PENETRATION =    0.370000E-01

CYCLONE PARAMETER ESTIMATES

DCUT =    0.331281E+01 SIGMA (CYCLONE) =    0.290939E+00
95 PERCENT CONFIDENCE INTERVAL FOR DCUT IS +/-  0.248547E+00
VARIANCE (DCUT) =    0.103169E-01

IF CALIBRATED CYCLONE DATA AND RESULTS ARE TO
BE STORED IN A DATA SET (ALLOCATED TO F(FT04F001))
ENTER 1; OTHERWISE 0
?
?1

CALIBRATION CONSTANT =
?
?1.38
CV FROM PRECISION EXPERIMENT =
?
?0.0212
NUMBER OF DEGREES OF FREEDOM =
?
?9.17
```

```
READY

?LIST Q20.DATA

DSNAME='$MLW.Q20.DATA'
        0.103169E-01
        0.212000E-01
        0.917000E+01
         8
        0.188000E+01  0.131100E+01
        0.246000E+01  0.128340E+01
        0.323000E+01  0.717600E+00
        0.349000E+01  0.699660E+00
        0.395000E+01  0.300840E+00
        0.485000E+01  0.759000E-01
        0.548000E+01  0.510600E-01
        0.592000E+01  0.510600E-01
END OF DATA

READY

?ALLOCATE F(FT07F001) DA(OUT.DATA) NEW SPACE(10) TRACKS

READY

?CALL COMPLY

OUTPUT DEVICE = (6=TERMINAL AND 7=DATA SET)
?
?7

FOR CALIBRATION BALANCING, ENTER 1;

PROCEDURE PERMITS NO PRIOR NEGATIVE CRITICAL CV;

OTHERWISE, ENTER 0:

?

?0


READY


?
```

?LIST OUT.DATA

DSNAME='$MLW.OUT.DATA'

THIS PROGRAM DETERMINES SAMPLER COMPLIANCE AT A
VARIETY OF TEST DUST DISTRIBUTIONS GIVEN ESTIMATED
VALUES FOR VARIANCE (DCUT), RELATIVE STANDARD
DEVIATION (CV) AND MONODISPERSE (CALIBRATED)
COLLECTION EFFICIENCY DATA, WHICH MUST BE IN A DATA
SET ALLOCATED TO F(FT04F001)

ESTIMATED VARIANCE (DCUT) = 0.103169E-01

ESTIMATED RELATIVE STANDARD DEVIATION = 0.212000E-01

NUMBER OF DEGREES OF FREEDOM (PRECISION) = 0.917000E+01

MONODISPERSE DATA:

DIAMETER = 0.188000E+01 SAMPLING EFFICIENCY = 0.131100E+01

DIAMETER = 0.246000E+01 SAMPLING EFFICIENCY = 0.128340E+01

DIAMETER = 0.323000E+01 SAMPLING EFFICIENCY = 0.717600E+00

DIAMETER = 0.349000E+01 SAMPLING EFFICIENCY = 0.699660E+00

DIAMETER = 0.395000E+01 SAMPLING EFFICIENCY = 0.300840E+00

DIAMETER = 0.485000E+01 SAMPLING EFFICIENCY = 0.759000E-01

DIAMETER = 0.548000E+01 SAMPLING EFFICIENCY = 0.510600E-01

DIAMETER = 0.592000E+01 SAMPLING EFFICIENCY = 0.510600E-01

COMPLIANCE COMPUTATION RESULTS:

AT MMD = 0.300000E+01 AND GSD = 0.190000E+01

BIAS = 0.885491E-01 BIAS UNCERTAINTY = 0.635728E-01

CRITICAL BIAS EDGE = 0.152122E+00

CRITICAL CV = 0.373153E-01

AT MMD = 0.290000E+01 AND GSD = 0.220000E+01

BIAS = 0.121012E+00 BIAS UNCERTAINTY = 0.536509E-01

CRITICAL BIAS EDGE = 0.174663E+00

CRITICAL CV = 0.281705E-01

AT MMD = 0.310000E+01 AND GSD = 0.280000E+01

BIAS = 0.136792E+00 BIAS UNCERTAINTY = 0.460635E-01

CRITICAL BIAS EDGE = 0.182856E+00

CRITICAL CV = 0.249331E-01

AT MMD = 0.101000E+02 AND GSD = 0.290000E+01

BIAS = -0.877596E-01 BIAS UNCERTAINTY = -0.701015E-01

CRITICAL BIAS EDGE = -0.157861E+00

CRITICAL CV = 0.480572E-01

AT MMD = 0.770000E+01 AND GSD = 0.300000E+01

BIAS = -0.191251E-01 BIAS UNCERTAINTY = -0.639135E-01

CRITICAL BIAS EDGE = -0.830386E-01

CRITICAL CV = 0.797410E-01

AT MMD = 0.172000E+02 AND GSD = 0.280000E+01

BIAS = -0.214224E+00 BIAS UNCERTAINTY = -0.776879E-01

CRITICAL BIAS EDGE = -0.291912E+00

CRITICAL CV = -0.722481E+00

SAMPLER OUT OF COMPLIANCE AT THIS DUST DISTRIBUTION

AT MMD = 0.186000E+02 AND GSD = 0.230000E+01

BIAS = -0.383896E+00 BIAS UNCERTAINTY = -0.819190E-01

CRITICAL BIAS EDGE = -0.465815E+00

CRITICAL CV = -0.722481E+00

SAMPLER OUT OF COMPLIANCE AT THIS DUST DISTRIBUTION

AT MMD = 0.800000E+01 AND GSD = 0.215000E+01

BIAS = -0.222541E+00 BIAS UNCERTAINTY = -0.820400E-01

CRITICAL BIAS EDGE = -0.304581E+00

CRITICAL CV = -0.722481E+00

SAMPLER OUT OF COMPLIANCE AT THIS DUST DISTRIBUTION

THE FRACTION OF TEST DUST DISTRIBUTIONS

AT WHICH SAMPLER IS OUT OF COMPLIANCE = 0.375000E+00

EXTREME 95% CONFIDENCE BIAS EDGES ARE AS FOLLOWS:

AT MMD = 0.186000E+02 AND GSD = 0.230000E+01

BIAS EDGE IS AT -0.465815E+00

AT MMD = 0.310000E+01 AND GSD = 0.280000E+01

BIAS EDGE IS AT 0.182856E+00

D. ACCURACY RESULTS AND DISCUSSION

The preceding section contains results of an example calculation of sampler accuracy between the experimental results with the 10 mm cyclone as presently used and the BMRC definition of respirable dust. Table IV-9 shows the extreme range of expected bias taken from the computer output. Listed are the extreme limits on the biases calculated over all the size distributions considered in this study. The limits are at the 95 percent confidence level accounting for experimental error in the penetration and precision measurements.

For the cyclone with Q=2.0 L/min and k=1.38, the systematic error relative to the BMRC definition ranges between -49 and 19 percent over the full range of coal mine dust distributions considered. The systematic errors alone do not meet the 25 percent accuracy criterion (95 percent confidence level), even if random sampling errors were zero.

Also shown in Table IV-9 are the results of an attempted improvement in the calibration constant as described above in Section II-H. The calibration constant is shifted from k = 1.38 to k = 1.70. Although the bias becomes somewhat more evenly balanced between positive and negative values, improvement is minimal. This is partly due to increased uncertainty in the bias.

Table IV-9 also shows the accuracy estimated for the 10mm cyclone operated at Q = 1.2 L/min and k = 0.91 in order to optimize the fit of the penetration curve with the BMRC definition (Bartley and Breuer, 1982). In this case, the extreme bias limits range between -7.3 and 8.1 percent. If random sampling errors (as yet unmeasured) are comparable to the values reported in Section IVA for cyclones operated at Q = 2.0 L/min, then the accuracy calculation indicates that the cyclone operated at the lower flow rate would meet the NIOSH accuracy criteria.

Table IV-9.
Extreme 95 Percent Confidence Limits on Cyclone Bias
(relative to the BMRC definition)

Cyclone Parameters	Extreme Positive Bias			Extreme Negative Bias		
	Bias (percent)	MMD (μm)	GSD	Bias (percent)	MMD (μm)	GSD
Q = 2.0 L/min k = 1.38	+19	3.1	2.8	-47	18.6	2.3
Q = 2.0 L/min k = 1.70	+47	3.1	2.8	-37	18.6	2.3
Q = 1.2 L/min k = 0.91	+8	18.6	2.3	-7	3.1	2.8

V. CONCLUSIONS AND RECOMMENDATIONS

Tests for the precision and collection efficiency of gravimetric Coal Mine Dust Personal Sampling Units have been developed. From these test results, limits to CMDPSU accuracy can be estimated for single samples taken in a coal mine. The accuracy criterion suggested here for CMDPSU certification is identical to that currently used by NIOSH for the validation of industrial hygiene sampling and analytical methods--±25 percent at the 95 percent confidence level.

The precision test proposed here consists of replicate CMDPSU samples taken in a coal dust chamber. New experimental design and statistical analysis have been developed in order to estimate the coefficient of variation due to the sampling unit, while minimizing the contribution from random errors caused by the dust chamber and the other experimental procedures. Quality control measures have also been developed for the filter weighing. The precision test has been run successfully with MSA's Model G CMDPSU. However, experience with the Bendix CMDPSU suggests that a more realistic precision test would be done with filter cassettes pre-weighed, assembled, and sealed by the manufacturers.

One difficulty with this alternative is that the manufacturers currently report the filter preweight to only 0.1 mg. At the respirable dust concentrations around 1 mg/m^3 in the precision test, the random error in this preweight can alone be larger than the NIOSH accuracy criteria, disregarding the other sources of error. In order to solve this problem, filters used in the precision test could be preweighed to 0.01 mg by the CMDPSU manufacturers.

The bias estimates are made by combining measurements of dust size distributions conducted in various coal mines with certification laboratory determinations of the CMDPSU's collection efficiency. For an example, the MSA sampler units as presently operated at flow rate Q = 2.0 L/min and calibration coefficient k = 1.38 is compared to the BMRC respirable dust definition. From the analysis of the collection efficiency measurements, the 10 mm cyclone sampling units have a mean bias as great as -47 percent (in the worst-case situations over the test size distributions).

Although the cyclone's bias at the 2.0 L/min flow rate alone exceeds the ±25 percent accuracy criterion, Bartley and Breuer (1982) have shown that the 10mm cyclone can meet the BMRC or ACGIH definitions within the NIOSH accuracy limits by using a reduced flow rate and an optimized calibration factor. To match the BMRC definition, the optimum cyclone flow rate is estimated to equal 1.2 L/min, and the calibration factor is 0.91.

Bartley and Breuer (1982) have performed the bias test with the optimum BMRC operating parameters. Using these results, the bias magnitude in sampling coal mine dust with respect to BMRC respirable dust can be held to within approximately 8 percent at the 95 percent confidence level. The precision testing for the optimized cyclone sampling method is now in progress, and will be published shortly. Thus, the optimum operating parameters is one means for

the 10mm cyclone to match the BMRC or ACGIH definitions within the NIOSH accuracy limits. (Under a certification program based on performance criteria, the responsibility for determining the best flow rate and calibration factor would ultimately belong to the CMDPSU manufacturer.)

Another purpose for revising CMDPSU certification is to "develop a system for the performance certification of new types of mine dust sampling equipment," as MSHA has stated (DOL, 1980). Of particular interest has been the certification of a new generation of self-regulating, quieter pumps such as the Du Pont P-2500 or P-4000. Under the present regulations, these pumps cannot be approved for use in CMDPSU's because i) they have design features such as an interior switch and the absence of a rotameter which violate the regulations and ii) their manufacturers do not make the other components of the CMDPSU, and therefore cannot offer the entire unit for certification, as required by the regulations.

The question of a certification procedure for the pump module itself, independent of the sampling head module, was the subject of research described by Bartley, Breuer, Baron and Bowman (1984). The original intent of that project was to:

(i) determine how to revise the fluctuation-related parts of the present certification program,

(ii) consider optional modular sampling unit certification, and

(iii) advance knowledge of cyclone operating characteristics for use in sampler design.

On the basis of this investigation, it is proposed simply that most of the design and performance requirements for the pump (30 CFR 74.3(a)) be eliminated. This suggestion is direct, guaranteed to cut DSR's operating costs and does not require collaborative testing between DPSE and DSR for its implementation.

The present pump pulsation regulations control 14 separate categories of pump design and performance. Some requirements such as the switch location (74.3(a)(5)) and the flow rate indicator (74.3(a)(11)) definitely eliminate existing pumps such as the P-2500 from consideration for CMDPSUs. Other design requirements such as the dimensions (74.3(a)(1)), weight (74.3(a)(2)), exhaust location (74.3(a)(4)), belt clips (74.3(a)(9)), and flow rate range (74.3(a)(12)) overconstrain the pump design, and their purposes (although usually desirable from NIOSH's point of view) can also be performed by a more open market for samplers. Even an essential performance criterion such as duration of operation (74.3(a)(14)) could be acheived (although with some trial and error) by the experience of CMDPSU users constrained to take valid (i.e., 8 hour) samples under Federal regulations.

It is recommended that the only properties of CMDPSU pumps to be regulated must clearly pertain to health and safety. For example, the pumps must be permissible in an explosive environment. Pump permissibility is now certified by MSHA, and this must continue.

Problemmatic are the pump pulsation criteria, which read as follows:

> "(i) The irregularity in flow rate due to pulsation shall have a fundamental frequency of not less than 20 Hz.
>
> (ii) On and after July 1, 1974 the quantity of respirable dust collected with a sampler unit shall be within 5 percent of that collected with a sampling head assembly operated with nonpulsating flow."
>
> 30 CFR Part 74.3(a)(8)

These regulations originated in part from research reported by Caplan, Doemeny and Sorenson (1973) and by Lamonica and Treaftis (1972). Both groups of researchers describe a significant shift in cyclone penetration characteristics due to pump fluctuations. As a result, pulsation dampeners have become standard equipment on present sampling units.

The above test procedures are unnecessary on several counts. There is no known justification for constraining the fundamental pulsation frequency to exceed 20 Hz. In fact, Bartley, Breuer, Baron and Bowman (1984) give evidence for a significant increase in cyclone penetration shift with pulsation frequency increase beyond 20 Hz. Furthermore, a broad resonance near 80 Hz is described which is due to the excitability of the air column in the Tygon tubing between pump and sampling head. The effect of this resonance is to increase the pulsation amplitude at the cyclone as the frequency increases through 20 Hz. There thus seems to be little reason for imposing a minimum pump pulsation frequency. More generally, since it is a combination of pulsation frequencies, amplitudes, mean flow and overall calibration constant which determines accuracy of the dust concentration estimates, no limitation on pulsation frequency alone seems justified.

The intent behind the 5 percent test in 30 CFR Part 74.3(a)(8)--namely, to control the bias in dust concentration estimates--is essential to a dust control program with rational basis. Adequate, simpler, and by far more general control of both bias and random sampling errors due to pulsation is afforded by the revised certification procedure detailed in the present document. Therefore, it is recommended that the pump pulsation criteria in 30 CFR Part 74.3(a)(8) be eliminated in favor of the unified bias test described in this report.

These recommendations would not permit modular certification. Pumps and sampling heads would still have to be certified as a unit. The testing of specific cyclone sampler pumps and heads separately goes against the grain of generalizing sampler certification to permit sampler heads beyond the 10 mm nylon cyclone. No present knowledge is available for specifically controlling the fluctuation-induced bias of, for example, the Cassella cyclone or personal cascade impactor. More specific to the 10 mm cyclone, it is very likely that a pump with large, though constant (i.e., reproducible) pulsation could, by proper selection of the mean flow through a specific sampling head, provide adequate sampling according to whatever definition is desired.

Furthermore, present knowledge of pulsation effects is limited to the MSA unit. As shown in Bartley, Breuer, Baron and Bowman (1984), the effect of pump pulsation on collection characteristics involves a complex interaction between pump, air line and the particular pre-selector, filter and filter holder of use. Therefore, no empirical basis exists for generalizing pulsation testing beyond the MSA unit.

Moreover, pump manufacturers such as Du Pont could certainly arrange for their pumps to be part of one or more units through contractual arrangements with the manufacturers of sampling heads. Although a modular sampling process could conceivably be developed, we feel that its primary benefit--interchange of sampling heads and pumps--does not offset its disadvantages, both philosophical and practical.

Finally, it is noted that a program of optional certification of pumps for use with specific cyclone sampling heads (at present, the MSA unit head) under conditions of low fluctuation could feasibly be designed. However, the complexity and expense of such a program hardly seem justifiable. Moreover, such a certification option would overly focus respirable dust sampling techniques into cyclone-based systems. Modular certification need be considered only as a decision to restrict sampling instrumentation to specific type.

For the above reasons, governmental regulation of sampling pump pulsation appears unnecessary, and the elimination of the present pulsation criteria (74.3(a)(8)) at the implementation of the revised certification criteria is strongly suggested. Establishment of optional modular certification is, likewise, not to be recommended. Proposed is the certification testing of only complete sampling units.

Still to be investigated is the certification of light-scattering dust monitors. These newly developed instruments have many desirable properties for monitoring hazardous aerosols, but their accuracy raise several difficult questions. The bias test developed for gravimetric samplers would need major modification for light-scattering instruments since their bias depends on the aerosol's density and optical properties, as well as the size distribution. However, the precision test recommended here and the statistical criteria for

making the accuracy determination could still be applied to the new dust monitors. The accuracy of light scattering devices is now being studied by NIOSH, MSHA and the Bureau of Mines, and the results of this research should provide a basis for comprehensive certification criteria.

The present state of the art is adequate for revising the certification regulations for gravimetric dust samplers (currently Volume 30, Code of Federal Regulations, Part 74). We do recommend additional validation for these accuracy test procedures in other laboratories before these tests are incorporated into certification regulations.

As has been widely recognized (e.g. Youden and Steiner, 1975), inter-laboratory variations in test results can be considerable. This source of variation should be allowed for in the accuracy criteria, especially if the new CMDPSU certification regulations will give the manufacturer a role in the performance testing. As recognized by EPA (Hauser and Shearer, 1975) and by the NIOSH consultants (Brief et al., 1980), such deregulation of instrument certification can be a more effective system of quality assurance. Since the test results from the government and industry laboratories will certainly disagree by some amount, this inter-laboratory variability should be incorporated into the uncertainty of the accuracy determination used for CMDPSU certification.

The last step in reforming the CMDPSU certification program will be drafting and adopting new regulations. In the certification regulations, the test methods should preferrable not be firmly specified. Since new aerosol techniques are constantly being developed, the test methods should be described in a government report which can be updated as the technology changes. The certification regulations would incorporate the test procedures by reference to the report.

Performance criteria regulations for CMDPSU certification could increase the effectiveness of coal mine dust sampling both scientifically and administratively. New procedures recommended here could increase the sampler's accuracy, and the use of performance criteria should encourage the development of improved equipment. This combination should make the coal dust sampling program easier to run, while increasing confidence in the results.

APPENDIX A

Computer program CYCLONE for calculation of cyclone parameters (including the translational uncertainty in the collection efficiency) from experimental data. The program also creates a data set for use by the program COMPLY (Appendix C).

```
000010          DOUBLE PRECISION BD,YD,XD,XBARD,YBARD,S1,S2,S3,MUC
000020          DIMENSION D(15),Y(15),PEN(15),XD(15),YD(15),T(15)
000030          T(1)=12.706
000040          T(2)=4.303
000050          T(3)=3.182
000060          T(4)=2.776
000070          T(5)=2.571
000080          T(6)=2.447
000090          T(7)=2.365
000100          T(8)=2.306
000110          T(9)=2.262
000120          WRITE (6,500)
000130          WRITE (6,20)
000140    20    FORMAT (' THIS PROGRAMS COMPUTES CYCLONE PARAMETERS GIVEN')
000150          WRITE (6,30)
000160    30    FORMAT (' N MONODISPERSE DATA PAIRS:')
000170          WRITE (6,50)
000180    50    FORMAT (' (DIAMETER (MICROMETERS), (FRACTIONAL) EFFICIENCY)')
000190          WRITE (6,500)
000200          WRITE (6,40)
000210    40    FORMAT (' N = ')
000220          READ (5,*) N
000230          XBARD=0.0D0
000240          YBARD=0.0D0
000250          WRITE (6,500)
000260          DO 100 I=1,N
000270          WRITE (6,10)
000280    10    FORMAT (' D =  SAMPLING EFFICIENCY = ')
000290          READ (5,*) D(I),PEN(I)
000300 C        WRITE (6,500)
000310          YD(I)=ERFI(1.-2.*PEN(I))
000320          XD(I)=ALOG(D(I))
000330          XBARD=XBARD+XD(I)
000340          YBARD=YBARD+YD(I)
000350    100   CONTINUE
000360          WRITE (6,500)
000370    500   FORMAT (' ')
000380          WRITE (6,700)
000390    700   FORMAT (' MONODISPERSE CYCLONE DATA')
000400          WRITE (6,500)
000410          WRITE (6,510) N
000420    510   FORMAT (' N = ',I4)
000430          WRITE (6,500)
```

```
000440          DO 800 I=1,N
000450          WRITE (6,900) D(I),PEN(I)
000460    900   FORMAT (' DIAMETER = ',E14.6,' PENETRATION = ',E14.6)
000470    800   CONTINUE
000480          WRITE (6,500)
000490          XBARD=XBARD/(N*1.)
000500          YBARD=YBARD/(N*1.)
000510          SD1=0.0D0
000520          SD2=0.0D0
000530          DO 300 I=1,N
000540          SD1=SD1+(XD(I)-XBARD)*(YD(I)-YBARD)
000550          SD2=SD2+(XD(I)-XBARD)**2
000560    300   CONTINUE
000570          BD=SD1/SD2
000580          SD3=0.0D0
000590          DO 400 I=1,N
000600          SD3=SD3+(YBARD+BD*(XD(I)-XBARD)-YD(I))**2
000610    400   CONTINUE
000620          VARY=SD3/((N-2)*1.)
000630          MUC=XBARD-YBARD/BD
000640          SMUC=MUC*1.E0
000650          DCUT=EXP(SMUC)
000660          SIGC=1./(BD*SQRT(2.))
000670          VARB=VARY/SD2
000680          VARYB=VARY/(N*1.)
000690          VARDC=2.*DCUT**2*SIGC**2*((XBARD-MUC)**2*VARB+VARYB)
000700          CONF=T(N-2)*SQRT(VARDC)
000710          WRITE (6,410)
000720    410   FORMAT (' CYCLONE PARAMETER ESTIMATES')
000730          WRITE (6,500)
000740          WRITE (6,420) DCUT,SIGC
000750    420   FORMAT (' DCUT = ',E14.6,' SIGMA (CYCLONE) = ',E14.6)
000760          WRITE (6,440) CONF
000770    440   FORMAT (' 95 PERCENT CONFIDENCE INTERVAL FOR DCUT IS +/-',E14.6)
000780          WRITE (6,430) VARDC
000790    430   FORMAT (' VARIANCE (DCUT) = ',E14.6)
000800          WRITE (6,500)
000810          WRITE (6,439)
000820    439   FORMAT (' IF CALIBRATED CYCLONE DATA AND RESULTS ARE TO')
000830          WRITE (6,441)
000840    441   FORMAT (' BE STORED IN A DATA SET (ALLOCATED TO F(FT04F001))')
000850          WRITE (6,442)
000860    442   FORMAT (' ENTER 1; OTHERWISE 0')
000870          READ (5,*) JANS
000880          IF (JANS) 443,443,444
000890    444   CONTINUE
000900          WRITE (6,500)
000910          WRITE (6,445)
000920    445   FORMAT (' CALIBRATION CONSTANT = ')
```

```
000930         READ (5,*) CAL
000940         WRITE (4,446) VARDC
000950    446  FORMAT ('       ',E14.6)
000960         WRITE (6,554)
000970    554  FORMAT (' CV FROM PRECISION EXPERIMENT = ')
000980         READ (5,*) CV
000990         WRITE (4,447) CV
001000    447  FORMAT ('       ',E14.6)
001010         WRITE (6,555)
001020    555  FORMAT (' NUMBER OF DEGREES OF FREEDOM = ')
001030         READ (5,*) DF
001040         WRITE (4,448) DF
001050    448  FORMAT ('       ',E14.6)
001060         WRITE (4,551) N
001070    551  FORMAT ('       ',I4)
001080         DO 552 JJ=1,N
001090         CPEN=CAL*PEN(JJ)
001100         WRITE (4,553) D(JJ),CPEN
001110    553  FORMAT ('       ',2E14.6)
001120    552  CONTINUE
001130    443  CONTINUE
001140         STOP
001150         END
001160         FUNCTION ERFI(X)
001170         N=10000
001180         PI=3.14159265
001190         Y0=X
001200         DO 200 I=1,N
001210         Y0=Y0+2./SQRT(PI)*(X-ERF(Y0))*EXP(-1.*Y0**2)
001220    200  CONTINUE
001230         ERFI=Y0
001240         RETURN
001250         END
```

APPENDIX B

COMPUTER PROGRAMS FOR ANALYZING PRECISION TEST

These programs are structured to be run as a batch job on the Parklawn Computer Center system (IBM 370). The actual computer commands are in capital letters. Variable quantities to be filled in by the individual user are in lower case letters.

1. Program for analyzing the results of the experimental design without position effects (Table II-7).

```
000010 //run id  JOB (agency,project,),identfier,CLASS=priority
000020 /*PASS   password
000030 //  EXEC SAS,OPTIONS='LS=72',REGION=300K
000040 //FT11F001 DD SYSOUT=T,HOLD=YES
000050 //FT12F001 DD SYSOUT=T,HOLD=YES
000060 //COAL DD DSN=data set name.DATA,DISP=SHR
000070 DATA;
000080   INFILE COAL;
000090   INPUT T RUN S $ CONC;
000100   LNC=LOG(CONC);
000110 PROC PRINT;
000120 PROC GLM;
000130   CLASSES T S RUN;
000140   MODEL LNC=T RUN(T) S(T);
000160   OUTPUT OUT=NEW RESIDUAL=RESID;
000170 PROC PLOT;
000180   PLOT RESID*LNC;
000190 /*
000200 //
```

2. Program for analyzing the results of the experimental design with position effects (Table II-8).

```
00010 //run id JOB (agency,project,),identifier,CLASS=priority
00020 /*PASS   password
00030 //  EXEC SAS,OPTIONS='LS=72',REGION=300K
00040 //FT11F001 DD SYSOUT=T,HOLD=YES
00050 //FT12F001 DD SYSOUT=T,HOLD=YES
00060 //COAL DD DSN=data set name.DATA,DISP=SHR
00070 DATA;
00080   INFILE COAL;
00090   INPUT T EXP RUN POS CONC;
00100   LNC=LOG(CONC);
00110 PROC PRINT;
00120 PROC GLM;
00130   CLASSES T EXP POS RUN;
00140   MODEL LNC=T EXP(T) RUN(EXP T) POS POS*T POS*EXP(T);
00150   MEANS POS/DUNCAN;
00152   TEST H=POS E=POS*EXP(T);
00154   TEST H=POS*T E=POS*EXP(T);
00160   OUTPUT OUT=NEW RESIDUAL=RESID;
00170 PROC PLOT;
00180   PLOT RESID*LNC;
00190   PLOT RESID*POS=T;
00200 PROC PLOT;
00210   BY T;
00220   PLOT RESID*POS=EXP;
00230 /*
00240 //
```

APPENDIX C

Computer program COMPLY giving acceptance/non-acceptance decisions from measured CV, measured calibrated collection efficiency, the translational uncertainty in the collection efficiency and test coal dust distributions. Input must be in a data set as created by CYCLONE. Program also searches for improved calibration constant.

```
000010          REAL MMD
000020          DIMENSION DI(30),FI(30),T(30),RMMD(50),RGSD(50),DS(15),FS(15)
000030          WRITE (6,614)
000040    614   FORMAT(' OUTPUT DEVICE = (6=TERMINAL AND 7=DATA SET)')
000050          READ (5,*) JOUT
000060          WRITE (JOUT,500)
000070          NDUST=8
000080          WRITE (JOUT,1)
000090    1     FORMAT (' THIS PROGRAM DETERMINES SAMPLER COMPLIANCE AT A')
000100          WRITE (JOUT,2)
000110    2     FORMAT (' VARIETY OF TEST DUST DISTRIBUTIONS GIVEN ESTIMATED')
000120          WRITE (JOUT,3)
000130    3     FORMAT (' VALUES FOR VARIANCE (DCUT), RELATIVE STANDARD')
000140          WRITE (JOUT,4)
000150    4     FORMAT (' DEVIATION (CV) AND MONODISPERSE (CALIBRATED)')
000160          WRITE (JOUT,5)
000170    5     FORMAT (' COLLECTION EFFICIENCY DATA, WHICH MUST BE IN A DATA')
000180          WRITE (JOUT,6)
000190    6     FORMAT (' SET ALLOCATED TO F(FT04F001)')
000200          WRITE (JOUT,500)
000210          NS=9
000220          DS(2)=1.5
000230          FS(2)=1.00
000240          DS(3)=2.25
000250          FS(3)=.87
000260          DS(4)=3.5
000270          FS(4)=.69
000280          DS(5)=4.2
000290          FS(5)=.62
000300          DS(6)=5.6
000310          FS(6)=.41
000320          DS(7)=5.7
000330          FS(7)=.33
000340          DS(8)=6.4
000350          FS(8)=.19
000360          DS(9)=7.8
000370          FS(9)=.03
000380          DS(10)=9.0
000390          FS(10)=.04
000400          DS(1)=DS(2)-.01
000410          FS(1)=FS(2)
000420          DS(11)=10.0
000430          FS(11)=0.0
```

```
000440         DS(12)=10.01
000450         FS(12)=0.0
000460         T(1)=6.314
000470         T(2)=2.920
000480         T(3)=2.353
000490         T(4)=2.132
000500         T(5)=2.015
000510         T(6)=1.943
000520         T(7)=1.895
000530         T(8)=1.860
000540         T(9)=1.833
000550         T(10)=1.812
000560         T(11)=1.796
000570         RMMD(1)=3.0
000580         RGSD(1)=1.9
000590         RMMD(2)=2.9
000600         RGSD(2)=2.2
000610         RMMD(3)=3.1
000620         RGSD(3)=2.8
000630         RMMD(4)=10.1
000640         RGSD(4)=2.9
000650         RMMD(5)=7.7
000660         RGSD(5)=3.0
000670         RMMD(6)=17.2
000680         RGSD(6)=2.8
000690         RMMD(7)=18.6
000700         RGSD(7)=2.3
000710         RMMD(8)=8.0
000720         RGSD(8)=2.15
000730         WRITE (JOUT,500)
000740 C       WRITE (JOUT,10)
000750   10    FORMAT (' VARIANCE (DCUT) = ')
000760         READ (4,*) VARDC
000770         WRITE (JOUT,500)
000780         WRITE (JOUT,500)
000790 C       WRITE (JOUT,40)
000800   40    FORMAT (' ESTIMATED (FRACTIONAL) CV = ')
000810         READ (4,*) CV
000820         WRITE (JOUT,500)
000830 C       WRITE (JOUT,15)
000840   15    FORMAT (' NUMBER OF DEGREES OF FREEDOM (PRECISION) = ')
000850         READ (4,*) DF
000860         WRITE (JOUT,500)
000870         READ (4,*) N
000880         DO 100 I=1,N
000890         READ (4,*) DI(I+1),FI(I+1)
000900   100   CONTINUE
000910   101   CONTINUE
000920         CVL=-10.
000930         IOUT=0
```

```
000940         ZMAX=-100.
000950         ZMIN=+100.
000960         WRITE (JOUT,500)
000970         WRITE (JOUT,41) VARDC
000980    41   FORMAT (' ESTIMATED VARIANCE (DCUT) = ',E14.6)
000990         WRITE (JOUT,500)
001000         WRITE (JOUT,42) CV
001010    42   FORMAT (' ESTIMATED RELATIVE STANDARD DEVIATION = ',E14.6)
001020         WRITE (JOUT,500)
001030         WRITE (JOUT,25) DF
001040    25   FORMAT (' NUMBER OF DEGREES OF FREEDOM (PRECISION) = ',E14.6)
001050         WRITE (JOUT,500)
001060         WRITE (JOUT,43)
001070    43   FORMAT (' MONODISPERSE DATA:')
001080         WRITE (JOUT,500)
001090         DO 44 J=1,N
001100         WRITE (JOUT,45) DI(J+1),FI(J+1)
001110    45   FORMAT (' DIAMETER = ',E14.6,' SAMPLING EFFICIENCY = ',E14.6)
001120    44   CONTINUE
001130         WRITE (JOUT,500)
001140         WRITE (JOUT,46)
001150    46   FORMAT (' COMPLIANCE COMPUTATION RESULTS:')
001160         WRITE (JOUT,500)
001170         DI(1)=DI(2)-.01
001180         FI(1)=FI(2)
001190         DI(N+2)=10.0
001200         FI(N+2)=0.0
001210         DI(N+3)=10.01
001220         FI(N+3)=0.0
001230         DO 200 I=1,NDUST
001240         MMD=RMMD(I)
001250         GSD=RGSD(I)
001260         W=BIAS(MMD,GSD,DI,FI,N,0.0,DS,FS,NS)
001270         DELD=0.1E0
001280         DW=BIAS(MMD,GSD,DI,FI,N,0.0,DS,FS,NS)
001290         DW=DW-BIAS(MMD,GSD,DI,FI,N,DELD,DS,FS,NS)
001300         VARB=(DW/DELD)**2*VARDC
001310    500  FORMAT (' ')
001320         DBIAS=T(N-2)*SQRT(VARB)
001330         CVC=CVCRIT(W+DBIAS)/(1.+1.645/SQRT(2.*DF))
001340         CVCM=CVCRIT(W-DBIAS)/(1.+1.645/SQRT(2.*DF))
001350         IF (CVCM-CVC) 50,55,60
001360    50   DBIAS=-DBIAS
001370         CVC=CVCM
001380         GOTO 60
001390    55   DBIAS=ABS(DBIAS)*W/ABS(W)
001400    60   CONTINUE
001410         Z=W+DBIAS
001420         IF (Z-ZMAX) 320,320,310
001430    310  WA=W
001440         DBA=DBIAS
```

```
001450          ZMAX=Z
001460          ZMMDA=MMD
001470          ZGSDA=GSD
001480    320   IF (Z-ZMIN) 330,340,340
001490    330   WI=W
001500          DBI=DBIAS
001510          ZMIN=Z
001520          ZMMDI=MMD
001530          ZGSDI=GSD
001540    340   CONTINUE
001550          WRITE (JOUT,500)
001560          WRITE (JOUT,70) RMMD(I),RGSD(I)
001570    70    FORMAT (' AT MMD = ',E14.6,' AND GSD = ',E14.6)
001580          WRITE (JOUT,80) W,DBIAS
001590    80    FORMAT (' BIAS = ',E14.6,' BIAS UNCERTAINTY = ',E14.6)
001600          WRITE (JOUT,85) Z
001610    85    FORMAT (' CRITICAL BIAS EDGE = ',E14.6)
001620          WRITE (JOUT,90) CVC
001630    90    FORMAT (' CRITICAL CV = ',E14.6)
001640          IF (CVC-CV) 110,200,200
001650    110   WRITE (JOUT,120)
001660          IOUT=IOUT+1
001670    120   FORMAT (' SAMPLER OUT OF COMPLIANCE AT THIS DUST DISTRIBUTION')
001680          WRITE (JOUT,500)
001690    200   CONTINUE
001700          FRACT=(1.0*IOUT)/(1.0*NDUST)
001710          WRITE (JOUT,500)
001720          WRITE (JOUT,500)
001730          WRITE (JOUT,201)
001740    201   FORMAT (' THE FRACTION OF TEST DUST DISTRIBUTIONS')
001750          WRITE (JOUT,202) FRACT
001760    202   FORMAT (' AT WHICH SAMPLER IS OUT OF COMPLIANCE = ',E14.6)
001770          WRITE (JOUT,500)
001780          WRITE (JOUT,204)
001790    204   FORMAT (' EXTREME 95% CONFIDENCE BIAS EDGES ARE AS FOLLOWS:')
001800          WRITE (JOUT,205) ZMMDI,ZGSDI
001810    205   FORMAT (' AT MMD = ',E14.6,' AND GSD = ',E14.6)
001820          ZI=WI-ABS(DBI)
001830          WRITE (JOUT,206) ZI
001840    206   FORMAT (' BIAS EDGE IS AT ',E14.6)
001850          WRITE (JOUT,205) ZMMDA,ZGSDA
001860          ZA=WA+ABS(DBA)
001870          WRITE (JOUT,206) ZA
001880          WRITE (6,500)
001890          WRITE (6,410)
001900    410   FORMAT (' FOR CALIBRATION BALANCING, ENTER 1;')
001910          WRITE (6,411)
001920    411   FORMAT(' PROCEDURE PERMITS NO PRIOR NEGATIVE CRITICAL CV;')
001930          WRITE (6,415)
001940    415   FORMAT (' OTHERWISE, ENTER 0:')
001950          READ (5,*) NANS
```

```
001960          IF (NANS) 600,600,420
001970    420   CONTINUE
001980          DO 425 JJ=1,1001
001990          CAL=(JJ-501)*.001E0
002000          DELBA=CAL*(1.+WA)
002010          DELBI=CAL*(1.+WI)
002020          CVA=CVCRIT(WA+DELBA+DBA)
002030          CVAM=CVCRIT(WA+DELBA-DBA)
002040          CVA=AMIN1(CVA,CVAM)
002050          CVI=CVCRIT(WI+DELBI+DBI)
002060          CVIM=CVCRIT(WI+DELBI-DBI)
002070          CVI=AMIN1(CVI,CVIM)
002080          CVMIN=AMIN1(CVA,CVI)
002090          IF (CVMIN-CVL) 425,425,530
002100    530   CVL=CVMIN
002110          CALMAX=CAL
002120    425   CONTINUE
002130          CALADJ=1.+CALMAX
002140          WRITE (JOUT,430)
002150    430   FORMAT (' BALANCED CALIBRATION IS OBTAINED THROUGH')
002160          WRITE (JOUT,435) CALADJ
002170    435   FORMAT (' RECALIBRATION BY A FACTOR OF ',E14.6)
002180          WRITE (JOUT,500)
002190          WRITE (JOUT,440)
002200    440   FORMAT (' THE FOLLOWING PERTAINS TO ')
002210          WRITE (JOUT,445)
002220    445   FORMAT (' RECALIBRATED MONODISPERSE DATA:')
002230          WRITE (JOUT,500)
002240          NP=N+3
002250          DO 450 JJ=1,NP
002260          FI(JJ)=CALADJ*FI(JJ)
002270    450   CONTINUE
002280          GO TO 101
002290    600   CONTINUE
002300          STOP
002310          END
002320          FUNCTION ACGIH(DA)
002330          IF (DA-2.0E0) 50,50,10
002340     10   Z=175.1068-53.4905*DA+6.0339*DA**2-.2924*DA**3+.004884*DA**4
002350          ACGIH=Z*.01E0
002360          IF (DA-10.E0) 60,60,40
002370     40   ACGIH=0.0E0
002380          GO TO 60
002390     50   ACGIH=.9
002400     60   CONTINUE
002410          RETURN
002420          END
002430          FUNCTION BIAS(MMD,GSD,DI,FI,N,TRANS,DS,FS,NS)
002440          DIMENSION DI(15),FI(15),DS(15),FS(15)
```

```
002450       REAL MU,MP,MMD
002460       PI=3.14159
002470       MU=ALOG(MMD)
002480       SIG=ALOG(GSD)
002490       TOT=0.E0
002500       TOT1=0.0E0
002510       DD=.01
002520       DO 100 I=1,1000
002530       D=DD*I
002540       MP=EXP(-.5*(ALOG(D)-MU)**2/SIG**2)/D/SIG/(2*PI)**.5
002550 C     FOLLOWING IS 'BMRC(D)', 'ACGIH(D)', OR 'SPLINE(D,DS,FS,NS)'
002560       Z=BMRC(D)
002570       DP=D-TRANS
002580       Z1=SPLINE(DP,DI,FI,N)
002590       TOT=TOT+Z*MP
002600       TOT1=TOT1+Z1*MP
002610   100 CONTINUE
002620       TOT=TOT*DD
002630       TOT1=TOT1*DD
002640       BIAS=(TOT1-TOT)/TOT
002650       RETURN
002660       END
002670       FUNCTION BMRC(DA)
002680       BMRC=1-(DA/7.1)**2
002690       IF (DA-7.1E0) 60,60,40
002700   40  BMRC=0.0E0
002710       GO TO 60
002720   60  CONTINUE
002730       RETURN
002740       END
002750       FUNCTION SPLINE(DD,DI,FI,N)
002760       DIMENSION DI(15),FI(15),D(4),F(4)
002770       IF (DD-DI(2)) 401,401,402
002780   401 SPLINE=FI(2)
002790       GO TO 500
002800   402 CONTINUE
002810       NN=N+2
002820       DO 405 J=3,NN
002830       IF (DD-DI(J)) 408,408,405
002840   408 IF (DD-DI(J-1)) 405,404,404
002850   404 DO 403 JJ=1,4
002860       D(JJ)=DI(JJ+J-3)
002870       F(JJ)=FI(JJ+J-3)
002880   403 CONTINUE
002890   405 CONTINUE
002900       SPLINE=0.00
002910   122 IF (DD-DI(N+2)) 123,300,300
002920   123 CONTINUE
002930       DO 300 I=1,4
```

```
002940          G=F(I)
002950          DO 200 J=1,4
002960          IF(J-I) 150,200,150
002970    150   CONTINUE
002980          G=G*(DD-D(J))
002990          G=G/(D(I)-D(J))
003000    200   CONTINUE
003010    299   SPLINE=SPLINE+G
003020    300   CONTINUE
003030    500   CONTINUE
003040          RETURN
003050          END
003060          FUNCTION CVCRIT(BIAS)
003070          ACC=0.25
003080          IF (ABS(BIAS).LT.ACC) GOTO 5
003090          CVCRIT=-1.00
003100          RETURN
003110    5     CONTINUE
003120          X=0.0
003130          DO 100 J=1,20
003140          ARG1=X*(ACC-BIAS)
003150          IF (ARG1**2.GT.50.) E1=0.0
003160          IF (ARG1**2.LE.50.) E1=EXP(-ARG1**2)
003170          ARG2=X*(ACC+BIAS)
003180          IF (ARG2**2.GT.50.) E2=0.0
003190          IF (ARG2**2.LE.50.) E2=EXP(-ARG2**2)
003200          F=-.95+.5*( ERF(ARG1) + ERF(ARG2) )
003210          FP=(ACC-BIAS)*E1 + (ACC+BIAS)*E2
003220          X=X-F/FP
003230    100   CONTINUE
003240          CVCRIT=1./(X*SQRT(2.)*(1.+BIAS))
003250          RETURN
003260          END
```

APPENDIX D

DATA SHEETS FOR PRECISION EXPERIMENT

CMDPSU Precision Test - Pump Flow Rates

CMDPSU: Operator:

Target Con: Target Flow Rate :

Run No.: Target Run Time:

Date: Chamber Humidity:

* = time at which rotameter is adjusted back to target

Chamber Position	1	2	3	4
Pump ID				
Rotameter Target Reading				
Reading Times: Initial* 1/2 hr* 1 hr				
Mean flow rate (L/min) Deviation from target(%) Std.Dev. (1/min) C.V. (%)				

CMDPSU Precision Test - Final Data

CMDPSU: Date:

Target Concentration: Analysts:

Target, EXP, RUN	Sampler	Initial Weight (mg)	Final Weight (mg)	Dust Wt (mg)	Time (min)	Conc. (mg/cu.m.)

CMDPSU Precision Test - Weighing Data

CMDPSU: Date:

Target Conc: Weighing Done By:

Run Number: QC Done By:

Filter ID	Chamber Position	Sampler ID	Mfg. Wt. (mg)	Initial Weight (mg)	Final Weight (mg)
	200 mg standard weight				
	Blank filter				
	1				
	2				
	3				
	4				
	5				
	Quality Control				
	Standard weight				
	Blank filter				
		Humidity			
		Temperature			
		Atm. pressure			

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