

**MEASUREMENT PRECISION  
WITH THE COAL MINE DUST PERSONAL SAMPLER**

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## **Abstract**

The purpose of this study was to quantify the precision of coal mine dust concentration measurements made with state-of-the-art gravimetric techniques and samplers incorporating flow control technology. Using a specially designed, portable dust chamber, twenty-two tests were conducted in an underground coal mine. Each test consisted of collecting 16 simultaneous dust samples, using 16 coal mine dust sampler units, symmetrically mounted in a container with a central inlet. Dust filter capsules were weighed in the same laboratory before and after exposure, with pre- and post-exposure weights recorded to the nearest  $\mu\text{g}$  (0.001 mg). The average weight gain observed within tests ranged from 62  $\mu\text{g}$ , collected over a 325-minute sampling period, to 6750  $\mu\text{g}$ , collected over 320 minutes. Based on a weighted least squares, repeated measures regression analysis, a point estimate for the standard deviation of error in recorded weight gain is 9.1  $\mu\text{g}$ , with an upper one-tailed 95%-confidence limit (UCL) of 10.3  $\mu\text{g}$ . The corresponding estimate of measurement imprecision deriving from other sources (including inter-sampler variability) is 4.3 percent (UCL = 6.8%). For dust samples collected over a 480-minute period, this results in an estimate of overall measurement imprecision ( $CV_{\text{total}}$ ) decreasing asymptotically from 7.8 percent (UCL = 8.9%) at dust concentrations of 0.2  $\text{mg}/\text{m}^3$  to 4.3 percent (UCL = 6.4%) at concentrations greater than 2.0  $\text{mg}/\text{m}^3$ . To confirm the regression estimate of imprecision due to sources other than weighing error, an analysis of variance was performed on 12 tests (186 measurements) for which weighing error was expected to contribute only a small fraction of  $CV_{\text{total}}$ . Based on this second analysis, variability attributable to physical differences between sampler units is estimated at 2.3 percent (UCL = 3.1%) and measurement imprecision attributable to the combined effects of variability in air flow and flow rate adjustment is estimated at 4.0 percent (UCL = 4.4%). These combine to form an estimate of 4.6 percent (UCL = 5.1%) for average  $CV_{\text{total}}$  at weight gains greater than 500  $\mu\text{g}$ .

Key words: respirable dust, gravimetrics, cyclone, weighing error, sampling variability, sampling and analytical error.

## **Introduction**

Since 1970, a number of attempts<sup>(1),(2),(3)</sup> have been made to quantify the accuracy and precision of respirable dust measurements obtained using the sampling equipment approved for use in U.S. coal mines. These studies have generally involved equipment which, though possibly still in service, is far from state-of-the-art. Furthermore, many of the attempts to quantify precision have relied on data originally collected for a different purpose. One example was data obtained from testing to establish an equivalency factor between the coal mine dust sampler and the MRE sampler<sup>(4)</sup>. These and other data were used by the National Institute for Occupational Safety and Health (NIOSH) in its 1982 attempt to estimate the precision of respirable coal mine dust measurements.<sup>(5)</sup>

The past reliance on data collected for other purposes has resulted in estimates of measurement uncertainty confounded by uncontrolled factors. For example, one study relied on samples collected up to 14 inches apart and, therefore, included spatial variability in dust concentration as part of the estimated measurement imprecision.<sup>(2)</sup> Actual differences in the aerosol concentration at the different locations sampled may have contributed to a relatively high estimate of measurement uncertainty.

Three independent factors have been identified as contributing to the variability, or imprecision, of aerosol dust concentration measurements collected in identical mine atmospheres with coal mine dust personal sampler units. These include (1) weighing errors; (2) variability attributable to the pump, including both variability in the initial adjustment of air flow rate and random fluctuations in air flow during the sampling period; and (3) physical differences between individual sampler units.<sup>(6)</sup> Imprecision due to these factors are quantified, respectively, as  $CV_{\text{weight}}$ ,  $CV_{\text{pump}}$  (comprising the combined effects of variability in air flow and flow rate adjustment), and  $CV_{\text{sampler}}$ . Overall measurement imprecision ( $CV_{\text{total}}$ ) can be obtained by combining the independent components of the measurement system using the following equation:

$$CV_{\text{total}}^2 = CV_{\text{weight}}^2 + CV_{\text{sampler}}^2 + CV_{\text{pump}}^2 \quad (1)$$

Since past studies frequently relied on combining estimates of these components obtained from different bodies of data, some of them have suffered from methodological problems related to combining individual sources of uncertainty. In 1984, for example, NIOSH identified several conceptual errors in earlier studies that had led to double- or even triple-counting of some variability components.<sup>(6)</sup>

A different approach to estimating measurement imprecision is to conduct a study that derives  $CV_{\text{total}}$  directly from a sufficiently large number of simultaneous measurements of the same dust aerosol. Using this approach, the Dust Division of MSHA Technical Support recently completed a study of the precision of measurements made with the coal mine dust personal sampler unit. The purpose of this study was to quantify the total imprecision of measurements made using the most recently approved coal mine dust sampling equipment and state-of-the-art analytical techniques. Therefore, the study was conducted with sampling pumps incorporating flow control technology and a robotic weighing system capable of weighing the sample collection filters to 1 microgram. The results of this study can be used to determine the precision attainable if: (1) samples are collected with pumps utilizing flow control technology, (2) both pre- and post-exposure weights are measured to the nearest microgram on a balance calibrated within MSHA's laboratory, and (3) truncation of weights is discontinued.

## **Experimental Procedures**

An enclosed, slightly tapered cylindrical container with an inlet located in the center of the top was constructed. The purpose of the container was to minimize differences in aerosol concentration which might be seen by different samplers due to spatial heterogeneity in the test environment. Figure 1 shows a schematic of the container. Within the container, a ring is suspended from which 16 coal mine dust sampler sampling heads are hung. Distance between sampler inlets is approximately 5 cm for side-by-side samplers and 20 cm for oppositely facing samplers. Tygon tubing passes from the sampling heads through the walls of the container to personal sampling pumps hung on rings around the outside of the container. Eight MSA Flow-Lite ET pumps and eight MSA Escort ELF pumps were used. A total of 32 new 10-mm nylon cyclones were used for the tests.

MSA filter capsules were weighed to 0.001 mg at MSHA's Respirable Dust Sample Processing Laboratory, using a Mettler MT5 balance. The capsules were then sealed in cassettes and placed into sampling heads mounted in the container. The container was taken into an underground coal mine and placed into either the immediate return of a continuous miner section, a belt entry or dump point, or the track area. All pumps were started as close to the same time as possible, and the device was left in place. The pumps were checked and the container was rotated 90 or 180 degrees approximately every hour during a test. No adjustments in flow rate were made beyond what would routinely be done by an MSHA inspector. After each test, the filter capsules were reweighed at the same facility that performed the initial weighing. Twenty-two tests were conducted, with sixteen simultaneous dust samples collected in each test. Test duration was between 260 and 360 minutes.

## **Data Analysis**

Nine of the 352 samples collected in the 22 multi-port tests were voided due to pump or hose malfunction. Table 1 summarizes weight gains and associated statistics for the remaining 343 valid dust samples. The MRE-equivalent dust concentration corresponding to the weight gain observed with a 10-mm nylon cyclone at a flow rate set at 2 lpm is calculated as

$$X = (1.38 \cdot G)/(2t) \quad (2)$$

where: X = dust concentration (mg/m<sup>3</sup>)  
 G = observed weight gain (µg)  
 t = sampling time (min).

The last column of Table 1 refers to dust concentrations corresponding to the observed weight gains spread over a hypothetical 480-minute sampling period. No statistically significant difference was observed between pump models.

As indicated by Table 1, the 22 tests were conducted over a broad range of dust concentrations. Imprecision of a dust concentration measurement, however, refers only to variability of measurements as they deviate from the true time-weighted average dust concentration *within* tests. Since the estimate of  $CV_{total}$  presented in Table 1 for each test is based on at most 16 samples, it is not a reliable estimate of the true  $CV_{total}$  to be expected, even for dust concentrations and sampling times identical to those of the test. In general, far more observations are required to reliably estimate the standard deviation or coefficient of variation than to achieve a comparably reliable estimate of the mean. The fact, however, that dust concentrations varied widely *between* tests makes it possible to efficiently estimate  $CV_{total}$  as a function of weight gain, using information from all 343 of the available observations.

Let  $\mu_i$  denote the true time-weighted average dust concentration sampled in the  $i^{th}$  test, let  $G_{ij}$  denote the  $j^{th}$  weight gain observed in the  $i^{th}$  test, and let  $X_{ij}$  denote the MRE-equivalent dust concentration corresponding to  $G_{ij}$ .  $X_{ij}$  differs from  $\mu_i$  by a measurement error, which consists of weighing, pump, and sampler components. The average of  $n_i$  weight gain measurements observed in the  $i^{th}$  test is denoted by  $G_i$ .

The average dust concentration within the  $i^{th}$  test,  $\bar{X}_i$ , provides a relatively precise estimate of  $\mu_i$ . The relative standard error of this estimate is obtained from Table 1 by dividing the corresponding coefficient of variation (CV) by  $\sqrt{n_i}$ , where  $n_i$  is the number of valid samples in the  $i^{th}$  test. For example, the average dust concentration observed in Test 22 was  $X_i = 1.18 \text{ mg/m}^3$ , with  $CV = 3.38\%$ . Therefore, the standard error of  $1.18 \text{ mg/m}^3$  as an estimate of  $\mu_{22}$  is  $3.38/\sqrt{16} = 0.845$  percent of 1.18, or  $0.010 \text{ mg/m}^3$ . Similarly,  $G_i$  is a good estimate of  $\Gamma_i$ , the weight gain expected in the  $i^{th}$  test, given a concentration equal to  $\mu_i$  sampled over a time period of length  $t_i$ .

Measurement imprecision is quantified by  $CV_{total}$ , which is the coefficient of variation of  $X_{ij}$  relative to  $\mu_i$ . Using  $E\{\}$  to denote the expected value and  $\text{Var}\{\}$  to denote the variance of any random variable, note that  $E\{X_{ij}\} = E\{X_i\} = \mu_i$  and  $\text{Var}\{X_{ij}\} = E\{(X_{ij} - \mu_i)^2\}$ . The underlying, or true value of  $CV_{total}^2$ , as distinguished from an estimate based on a finite number of samples, is expressed by:

$$\begin{aligned} CV_{total}^2 &= \text{Var}\{X_{ij}\} \div E^2\{X_{ij}\} \\ &= E\{(X_{ij} - \mu_i)^2\} \div \mu_i^2 \\ &= [n_i/(n_i - 1)] \cdot E\{(X_{ij} - X_i)^2\} \div \mu_i^2 \end{aligned} \quad (3)$$

The factor of  $n_i/(n_i - 1)$  corrects for the bias introduced by substitution of  $\bar{X}_i$  for  $\mu_i$ .

Since  $\mu_i$  is unknown, it will simplify the analysis to remove it from the formula for  $CV_{\text{total}}$  by applying a logarithmic transformation to each  $X_{ij}$ , yielding

$$CV_{\text{total}}^2 \approx [n_i/(n_i - 1)] \cdot E\{(Y_{ij} - \bar{Y}_i)^2\} \quad (4)$$

where  $Y_{ij}$  is the natural logarithm of  $X_{ij}$ . Equation 4 follows from Equation 3 because  $\text{Var}\{Y_{ij}\} \approx \text{Var}\{X_{ij}\} \div \mu_i^2$ .<sup>(7),(8)</sup>

The components of measurement variance due to differences in air flow rate among pumps, random fluctuations in air flow during the sampling period, and differences in the physical characteristics of individual sampler units all increase as more dust is accumulated.<sup>6</sup> Since the quantity of dust accumulated is proportional to dust concentration, this increase is reflected by constant values for  $CV_{\text{sampler}}$  and  $CV_{\text{pump}}$ , which express variability relative to dust concentration. In contrast, the weight of accumulated dust is calculated by subtracting pre- from post-exposure weighings of the entire filter capsule, and this difference typically amounts to less than one percent of the total weight being measured. Since the weight gain is a small fraction of the total mass being weighed, weighing errors can be assumed to be independent of sampling time and the quantity of dust accumulated on the filter. It follows that  $CV_{\text{weight}}$  (the ratio of a constant weighing error effect to a variable dust concentration) is inversely proportional to dust concentration. Also, Equation 2 implies that  $X_i = 1.38 \cdot G_i/(2t_i)$  and  $\text{Var}\{X_{ij}\} = [1.38/(2t_i)]^2 \text{Var}\{G_i\}$ . Consequently,

$$\begin{aligned} CV_{\text{total}}^2 &= CV_{\text{weight}}^2 + CV_{\text{sampler}}^2 + CV_{\text{pump}}^2 \\ &= [(1.38 \cdot \sigma_G / 2t_i) \div \mu_i]^2 + CV_{\eta}^2 \\ &\approx [(1.38 \cdot \sigma_G / 2t_i) \div \bar{X}_i]^2 + CV_{\eta}^2 \\ &= (\sigma_G^2) \cdot (1/\bar{G}_i)^2 + CV_{\eta}^2. \end{aligned} \quad (5)$$

where:  $t_i$  is the average sampling time associated with the  $i^{\text{th}}$  test;

$\sigma_G$  is the unknown standard deviation, in micrograms, of error in the weight gain measurement;

and  $CV_{\eta}^2 = CV_{\text{sampler}}^2 + CV_{\text{pump}}^2$  is an unknown constant.

Equation 5 shows that  $CV_{\text{total}}^2$ , expressed as a function of  $(1/\bar{G}_i)^2$ , has the form of a straight line, with slope equal to  $\sigma_G^2$  and intercept equal to  $CV_{\eta}^2$ . It follows that  $\sigma_G^2$  and  $CV_{\eta}^2$  can be estimated by linear regression, using  $(1/\bar{G}_i)^2$  and an estimate of  $CV_{\text{total}}^2$  for each test as the independent and dependent variable, respectively. In practice, more stable estimates of  $\sigma_G$  and  $CV_{\eta}$  can be achieved by estimating them directly, using a conceptually similar nonlinear regression model:

$$CV_i = (\sigma_G^2 / \bar{G}_i^2 + CV_{\eta}^2)^{1/2} + \epsilon_i \quad (6)$$

In this model,  $CV_i$  is the sample coefficient of variation observed in the  $i^{\text{th}}$  test, as shown in Table 1; and  $\epsilon_i$  is the residual regression error -- i.e., the difference between estimated and true values of  $CV_{\text{total}}$  for the  $i^{\text{th}}$  test.

Since they are based on only 22 aggregated data points, the regression analyses corresponding to Equations 5 and 6 do not fully utilize all the available information. Significant information may be lost by using  $CV_i$  to summarize the results of each test. Therefore, the principal regression approach to be pursued here uses all 343 observations directly. For comparison, however, Appendix A contains nonlinear regression estimates based on applying Equation 6 to summary data for the 22 tests.

By combining Equations 4 and 5, an expression is obtained that enables estimation of  $\sigma_G$  and  $CV_{\eta}$  by regression on all 343 individual observations:

$$[n_i/(n_i - 1)] \cdot E\{(Y_{ij} - \bar{Y}_i)^2\} = \sigma_G^2 / \bar{G}_i^2 + CV_{\eta}^2 \quad (7)$$

Equation 7 is equivalent to the regression model,

$$W_{ij} = \sigma_G^2 / \bar{G}_i^2 + CV_{\eta}^2 + \epsilon_{ij} \quad (8)$$

where  $W_{ij} = [n_i/(n_i - 1)] \cdot (Y_{ij} - \bar{Y}_i)^2$ , and the residual  $\epsilon_{ij}$  represents the deviation of  $W_{ij}$  from its expected value,  $CV_{\text{total}}^2$ , at a particular dust concentration. The standard deviation of  $\epsilon_{ij}$  is denoted  $\sigma_{\epsilon}$ .

Since the same observed value of  $\bar{Y}_i$  appears in  $W_{ij}$  for each replication (j) within a given test (i), the  $W_{ij}$ 's are correlated within tests. Essentially, the correlation arises because  $\epsilon_{ij}$  consists of two random components: (1) a component (representing estimation error in  $Y_i$ ) that is constant for measurements repeated within each test and (2) a component (representing pure measurement error) that is independently and identically distributed for all 343 measurements. A *repeated measures* model was used to explicitly separate these two components, thereby accounting for the correlation of  $W_{ij}$  within tests.

The repeated measures model represents  $\epsilon_{ij}$  as  $\tau_i + \zeta_{ij}$ , where  $\tau_i$  is a random effect of the  $i^{\text{th}}$  test and  $\zeta_{ij}$  is a random residual effect independent of  $\tau_i$ . Therefore  $E\{\tau_i\zeta_{ij}\} = 0$  and  $\sigma_\epsilon^2 = \text{Var}\{\tau_i\} + \text{Var}\{\zeta_{ij}\}$ . Because the  $\zeta_{ij}$ 's are also independent of one another, it follows that  $E\{\zeta_{ij}\zeta_{ik}\} = 0$ , so that the correlation between measurements repeated within tests is given by:

$$\begin{aligned} \rho &= E\{(\epsilon_{ij})(\epsilon_{ik})\} \div \sigma_\epsilon^2 \\ &= E\{(\tau_i + \zeta_{ij})(\tau_i + \zeta_{ik})\} \div \sigma_\epsilon^2 \\ &= E\{(\tau_i^2 + \tau_i\zeta_{ij} + \tau_i\zeta_{ik} + \zeta_{ij}\zeta_{ik})\} \div \sigma_\epsilon^2 \\ &= \text{Var}\{\tau_i\} \div [\text{Var}\{\tau_i\} + \text{Var}\{\zeta_{ij}\}]. \end{aligned} \quad (9)$$

$\text{Var}\{\tau_i\}$  represents uncertainty in the regression analysis due to estimating the true dust concentration within each test by the average of  $n_i$  measurements. Since  $\text{Var}\{\tau_i\}$  is inversely proportional to  $n_i$ , this uncertainty, along with the correlation of measurements within tests, decreases with increasing  $n_i$ . In the repeated measures model employed in the present analysis,  $\text{Var}\{\tau_i\}$  is estimated from the correlation observed within tests and added into the estimate for  $\sigma_\epsilon^2$  used to construct confidence limits.

To produce a regression curve for  $\text{CV}_{\text{total}}$  as a function of dust concentration sampled for 480-minutes, Equation 10 is obtained from the last step of Equation 5 by multiplying  $(1/G_i)^2$  by unity, expressed as  $[(1.38/960) \div (1.38/960)]^2$ , and then identifying  $X_i = 1.38G_i/960$  with  $\mu$ . For any dust concentration  $\mu$ , the regression estimate of  $\text{CV}_{\text{total}}$  (expressed as a percentage) is:

$$100 \cdot \text{CV}_{\text{total}} = 100[[(1.38 \cdot \sigma_G / 960) \div \mu]^2 + \text{CV}_\eta^2]^{1/2} \quad (10)$$

where  $\text{CV}_{\text{total}}$  is now interpreted as an estimate obtained by substituting the corresponding least-squares estimators for  $\sigma_G$  and  $\text{CV}_\eta^2$  into Equation 10.

The upper 95-percent confidence curve for this regression estimate of  $\text{CV}_{\text{total}}$  was estimated by application of the standard method to the linear model defined by Equation 8.<sup>(9)</sup> It should be noted that the relevant confidence limit pertains to the location of the regression line itself -- not to the scatter of individual observations around the line. Error in locating the regression line, at a particular dust concentration, is asymptotically Normal, even if the residuals themselves are not Normally distributed. Therefore, for each value of  $\mu$  sampled for 480 minutes, the 95-percent 1-tailed upper confidence limit (UCL) for  $\text{CV}_{\text{total}}$  obtained from the 343-point regression is:



$$\text{UCL} = [\text{CV}_{\text{total}}^2 + 1.645 \cdot \hat{\sigma}_\epsilon [1/343 + (z - \bar{z})^2 / \sum n_i (z_i - \bar{z})^2]^{1/2}]^{1/2} \quad (11)$$

where  $\hat{\sigma}_\epsilon$  is the estimated standard deviation of  $\epsilon_{ij}$

$$z = (1.38/960\mu)^2 = 1/\Gamma^2 \text{ for the expected weight gain } \Gamma = 960\mu/1.38$$

$$z_i = 1/\bar{G}_i^2 \text{ and } \bar{z} = \sum n_i z_i / 343$$

1.645 is the 95% 1-tailed confidence coefficient for Normally distributed random errors.

Note that because dust concentration is the variable of interest, the abscissa plotted using Equation 11 is actually  $\mu = 1.38\Gamma/960 = 1.38/(960\sqrt{z})$  instead of  $z$ .

For weight gains greater than 500  $\mu\text{g}$ , the estimated value of  $\sigma_G$  declines from 1.8% of a 500  $\mu\text{g}$  weight gain to 0.5% of a 2000  $\mu\text{g}$  weight gain (see results below). Therefore, weighing imprecision contributes little to  $\text{CV}_{\text{total}}$  for those 12 tests showing average weight gain greater than 500  $\mu\text{g}$ , and its effect can be assumed to be a negligible constant. Doing so makes it possible to estimate  $\text{CV}_{\text{sampler}}$  and  $\text{CV}_{\text{pump}}$  from the 186 valid samples collected in these 12 tests.

Maximum likelihood estimates of  $\text{CV}_{\text{sampler}}$  and  $\text{CV}_{\text{pump}}$  were obtained by analysis of variance (ANOVA), based on the following variance components model:

$$Y_{ij} = \phi_i + \zeta_j + \pi_{ij} \quad (12)$$

where  $i$  now ranges from 1 to 12 and indexes those tests in which  $\bar{G}_i > 500 \mu\text{g}$ ;

$j$  now ranges from 1 to 32 and indexes a particular dust sampler unit;

$Y_{ij}$  is the natural logarithm of  $X_{ij}$ , the dust concentration measurement observed using the  $j^{\text{th}}$  sampler in the  $i^{\text{th}}$  test;

$\phi_i$  is a fixed effect of the  $i^{\text{th}}$  test, representing (on a logarithmic scale) the true dust concentration for that test;

$\zeta_j \approx \mathbb{N}(0, \sigma_\zeta^2)$  is a Normally distributed random effect of the  $j^{\text{th}}$  dust sampler unit, with  $\text{Var}\{\zeta_j\}$  denoted by  $\sigma_\zeta^2$  equal to  $\text{CV}_{\text{sampler}}^2$ ;

$\pi_{ij} \approx \mathbb{N}(0, \sigma_\pi^2)$  is a Normally distributed random residual effect, identified with that portion of  $\text{Var}\{Y_{ij}\}$  not attributed to variability in  $\phi_i$  and  $\zeta_j$ . For  $Y_{ij}$  based on relatively large weight gains,  $\pi_{ij}$  is assumed to be dominated by initial adjustment of the pump and subsequent variability in air flow.  $\text{Var}\{\pi_{ij}\}$  is denoted by  $\sigma_\pi^2$  and is identified with  $\text{CV}_{\text{pump}}^2$  for the 12 tests examined.

As indicated in connection with Equation 4,  $\text{Var}\{Y_{ij}\}$  closely approximates  $\text{CV}_{\text{total}}^2$ . Therefore, ignoring  $\text{CV}_{\text{weight}}$  for tests with  $G > 500 \mu\text{g}$ ,

$$\begin{aligned}
 \text{Var}\{Y_{ij}\} &\approx \text{CV}_{\text{total}}^2 \\
 &\approx \text{CV}_{\text{sampler}}^2 + \text{CV}_{\text{pump}}^2 \\
 &= \text{CV}_\eta^2 \\
 &\approx \sigma_\zeta^2 + \sigma_\pi^2
 \end{aligned} \tag{13}$$

Using only those tests for which  $\bar{G} > 500 \mu\text{g}$ , separate estimates of  $\text{CV}_{\text{sampler}}$  and  $\text{CV}_{\text{pump}}$  were obtained by taking the square root of the corresponding ANOVA estimates of  $\sigma_\zeta^2$  and  $\sigma_\pi^2$ . Because it contains a small component of weighing error, the square root of the estimated  $\sigma_\zeta^2 + \sigma_\pi^2$  should slightly exceed the estimate of  $\text{CV}_\eta$  obtained from the regression analysis.

The 343-point regression and ANOVA analyses described above were both carried out using BMDP module 3V.<sup>(10)</sup> Each observation was weighted by its associated sampling time, relative to the mean sampling time across all observations used in the analysis. These weights, however, had little effect on the results.

## **Results**

Table II contains estimates of  $\sigma_G$  and  $\text{CV}_\eta$ , as defined in Equation 5, along with related results obtained from the 343-point repeated measures regression analysis based on Equation 8. The estimate shown for  $\sigma_\epsilon$  takes into account the correlation of residuals within tests. Corresponding results from the aggregated regression defined by Equation 6 are presented in Appendix A.

The values shown in Table II were substituted into Equations 10 and 11 to generate the estimates of  $\text{CV}_{\text{total}}$  and its UCL plotted, as a function of dust concentration ( $\mu$ ), in Figure 2. The true dust concentration being measured is specified on the horizontal axis. Measurement imprecision is represented on the vertical axis by the estimated coefficient of variation in MRE-equivalent 480-minute dust concentration measurements

( $CV_{\text{total}}$ ). The slope of the regression line illustrates the changing contribution of  $CV_{\text{weight}}$  to  $CV_{\text{total}}$ . As dust concentration increases,  $CV_{\text{weight}}$  approaches zero, and the regression line asymptotically approaches the combined effect of  $CV_{\text{sampler}}$  and  $CV_{\text{pump}}$ . At an average dust concentration of  $0.1 \text{ mg/m}^3$  sampled over a 480-minute period, the regression estimate for  $CV_{\text{total}}$  is 13.8 percent (UCL = 14.9%). From there, the regression estimate for  $CV_{\text{total}}$  drops to 7.8 percent (UCL = 8.9%) at dust concentrations of  $0.2 \text{ mg/m}^3$  and declines asymptotically to 4.3 percent (UCL = 6.4%) at concentrations greater than  $2.0 \text{ mg/m}^3$ .

By Equation 11, the distance between the regression estimate of  $CV_{\text{total}}$  and its UCL increases with increasing distance between a specified value of  $z$  and  $\bar{z}$ , the mean value of the independent variable used in the analysis. That is to say, uncertainty in the regression estimate of  $CV_{\text{total}}$  increases the further  $z$  departs from the mean value of  $z_i = 1/G_i^2$  observed in the experiment. This relation, however, is not apparent in Figure 2, since the abscissa plotted there is  $\mu = 1.38/(960\sqrt{z})$  instead of  $z$ . Therefore, to better illustrate uncertainty in the regression analysis, equivalent results for  $CV_{\text{total}}^2$  and its UCL are plotted in Figure 3 as a function of  $z = 1/\Gamma^2$ , where  $\Gamma$  is expected weight gain. Values of  $z$  are plotted along the horizontal axis, and  $\bar{z}$  is identified by the point labeled "mean."

To check the sensitivity of the regression results to outlying values of the dependent variable, robust (Huber) versions of the analysis were performed with progressively less importance attached to unusually small or large values of  $W_{ij}$ . The effect was to substantially decrease the estimate for  $\sigma_G$  and slightly increase the estimate for  $CV_{\eta}$ . Using a Huber constant of 23, the estimate for  $\sigma_G$  is reduced from  $9.1 \mu\text{g}$  to  $7.8 \mu\text{g}$ , while the estimate for  $CV_{\eta}$  is increased from 4.3% to 4.4%. Huber constants are defined in the documentation for BMDP Module 3R.<sup>10</sup>

Because of moderate colinearity in the regression estimates of  $\sigma_G$  and  $CV_{\eta}$ , the sensitivity of the  $\sigma_G$ -estimate to potential underestimation of  $CV_{\eta}$  was also examined. This was done by computing least-squares estimates of  $\sigma_G$ , subject to the constraint that  $CV_{\eta}$  assume specified values greater than the unconstrained estimate of  $CV_{\eta}$ . The effect was to substantially decrease the estimate of  $\sigma_G$ . Details of the constrained analyses are presented in Appendix B.

Table III contains estimates of  $CV_{\text{sampler}}$  and  $\sigma_{\pi}$ , obtained from the ANOVA analysis based on Equation 12 for dust concentrations calculated from weight gains greater than  $500 \mu\text{g}$ . Since the contribution of weighing error to  $CV_{\text{total}}$  appears to be negligible, on average, for the 12 tests in this range, the RML estimate for  $\sigma_{\pi}$  provides a reasonable estimate of  $CV_{\text{pump}}$ . However, this estimate of  $CV_{\text{pump}}$  is inflated slightly by the average contribution of  $CV_{\text{weight}}$  to  $\sigma_{\pi}$  in the 12 tests.

## **Discussion**

Based on the regression analysis summarized in Table III, the standard deviation of errors in recorded weight gain ( $\sigma_G$ ) is 9.1  $\mu\text{g}$  (UCL = 10.3  $\mu\text{g}$ ). The corresponding estimate of imprecision deriving from other sources ( $CV_\eta$ ) is 4.3 percent (UCL = 6.8%). These estimates exceed those obtained by the aggregated regression described in Appendix A. Furthermore, as demonstrated in Appendix B, if  $CV_\eta$  were actually greater than 4.3 percent, then this would force a decrease in the regression estimate of  $\sigma_G$ . Therefore, the 9.1  $\mu\text{g}$  estimate for  $\sigma_G$  may be regarded as conservative if  $CV_\eta$  is being underestimated. The regression estimate for  $\sigma_G$  derived in the present study is consistent with previously reported results for pre- and post-weighing to a microgram within the same laboratory.<sup>(6),(11)</sup>

For dust samples collected over a 480-minute period, Table II implies that  $CV_{\text{total}}$  and its UCL are estimated by the graph shown in Figure 2. Weight gains greater than 500  $\mu\text{g}$  correspond to dust concentrations greater than 0.72  $\text{mg}/\text{m}^3$ , based on 480-minute samples. For such concentrations, the ANOVA estimate of  $CV_{\text{total}} \approx CV_\eta = 4.6\%$ , shown in Table III, falls between the regression estimate given in Figure 2 and its UCL. Therefore, estimates obtained from the ANOVA are consistent with those obtained from the regression analysis. The 5.1% ANOVA UCL for  $CV_\eta$ , representing an upper bound on imprecision not attributable to weighing error, falls well below the regression UCL of 6.8%.

The ANOVA estimate of  $CV_{\text{sampler}} = 2.3\%$  (UCL = 3.1%) derived here falls below an estimate of  $CV_{\text{sampler}} = 5\%$  previously published by Dr. David Bartley et al.<sup>(12)</sup> Bartley's estimate, however, was based on a test of only eight cyclones (compared to 32 in the present study) and was, therefore, subject to considerable statistical uncertainty. Still, cyclones used in the present study were all new and might, for that reason, have exhibited less variability than the older cyclones used in the study on which Bartley's estimate was based. On the other hand, Bartley's 5% estimate was presented as being "...conservative in view of a value, 1.6%, reported by Bowman et al...".<sup>(6)</sup>

## **Conclusions**

As measured by  $CV_{\text{total}}$ , overall measurement imprecision associated with a single respirable coal dust sample collected over a 480-minute period was found, with 95-percent confidence, to be less than 9 percent for dust concentrations at or above 0.2  $\text{mg}/\text{m}^3$  and less than 7 percent for dust concentrations at or above 0.5  $\text{mg}/\text{m}^3$ . The corresponding maximum likelihood estimates for  $CV_{\text{total}}$  are 7.8 percent and 5.0 percent, respectively. At dust concentrations greater than 2.0  $\text{mg}/\text{m}^3$ , the maximum likelihood estimate for  $CV_{\text{total}}$  is

4.3 percent (UCL = 6.4%) based on the regression analysis or 4.6 percent (UCL = 5.1%) based on the analysis of variance (ANOVA).

Separate estimates were also obtained for the components of  $CV_{total}$ . Based on the ANOVA,  $CV_{sampler}$  and  $CV_{pump}$  were determined to be 2.3 percent (UCL = 3.1%) and 4.0 percent (UCL = 4.4%), respectively. These combine to form an estimate of 4.6 percent (UCL = 5.1%) for  $CV_{\eta}$ , which is statistically consistent with that derived from the regression analysis. (The estimates for  $CV_{pump}$  and  $CV_{\eta}$  are inflated slightly by  $CV_{weight}$ , averaged over those 12 tests in which the average observed weight gain exceeded 500  $\mu\text{g}$ .) The maximum likelihood estimate for the standard deviation of errors in recorded weight gain ( $\sigma_G$ ) was determined to be 9.1  $\mu\text{g}$ , with a UCL of 10.3  $\mu\text{g}$ . Since the ANOVA estimate for  $CV_{\eta}$  slightly exceeds the corresponding regression estimate and the regression estimates for  $CV_{\eta}$  and  $\sigma_G$  are inversely correlated, the estimate presented here for imprecision attributable to weighing error is considered conservative.

### Appendix A -- Regression of $CV_{total}$ Aggregated by Test

The aggregated, 22-point nonlinear regression analysis described by Equation 6 was carried out using BMDP Module 3R<sup>(10)</sup> on the 22 tests as summarized in Table I. Table IV contains the resulting estimates of  $\sigma_G$  and  $CV_{\eta}$ , as defined in Equation 5, along with the standard error of the regression estimate,  $\sigma_{\epsilon}$ . The estimated value of  $CV_{total}$  for each test was weighted by a factor proportional to the number of observations and average sampling time for that test. The upper 95-percent, one-tailed Cook-Weisberg confidence limits (UCL) presented for  $\sigma_G$  and  $CV_{\eta}$  were calculated by the BMDP statistical software. Cook-Weisberg limits more accurately represent nonlinear regression parameters than the more commonly used symmetric Wald approximations.<sup>(13)</sup>

From Table IV, the distance between the 22-point nonlinear regression estimate for  $\sigma_G$  and its UCL is  $10.20 - 8.88 = 1.32 \mu\text{g}$ . The corresponding value for the 343-point linear regression, obtained from Table II, is  $10.34 - 9.12 = 1.22 \mu\text{g}$ . As shown by the distance between the regression estimate and its UCL, the 22-point nonlinear regression model provides a slightly broader confidence interval for  $\sigma_G$  than the 343-point linear regression. The confidence interval for  $CV_{\eta}$ , on the other hand, is significantly more focussed. In particular, the UCL for  $CV_{\eta}$  calculated from the 22-point model falls well below the corresponding value in Table II and is closer to the UCL for  $CV_{\eta}$  estimated from the ANOVA model, as shown in Table III.

In Figure 4, the values of  $CV_{total}$  recorded for each test are plotted along with the regression line obtained from Equation 6. The regression line itself represents the expected or "true" value of  $CV_{total}$  predicted by the model for a given accumulation of dust mass (i.e., weight gain). Residuals are defined by the vertical distance of points from the

regression line and represent the effect of estimating  $CV_{\text{total}}$  within each of the 22 tests by a limited number of samples (13 to 16). Presumably, the average size of these residuals would decrease as the square root of the number of samples within each test increased. Note that residuals in the aggregated regression model of Equation 6 have different units than the residuals defined by Equation 8. Therefore, the  $\sigma_{\epsilon}$  of Table IV is not directly comparable to that of Table II.

### Appendix B -- Constrained Estimation of Weighing Imprecision

Table V contains regression results for the weighing component of measurement imprecision ( $\sigma_G$ ), when the non-weighing component ( $CV_{\eta}$ ) is constrained to be greater than the least-squares regression estimate. These results were obtained using the 22-point aggregated nonlinear regression model defined by Equation 6.

The Values assumed for  $CV_{\eta}$  correspond to the following assumptions for  $CV_{\text{pump}}$  and  $CV_{\text{sampler}}$ :

$$CV_{\eta} = 5.19\% \quad CV_{\text{pump}} = 4.24\% \quad \text{and} \quad CV_{\text{sampler}} = 3\%$$

$$CV_{\eta} = 6.56\% \quad CV_{\text{pump}} = 4.24\% \quad \text{and} \quad CV_{\text{sampler}} = 5\%$$

$$CV_{\eta} = 9.95\% \quad CV_{\text{pump}} = 4.24\% \quad \text{and} \quad CV_{\text{sampler}} = 9\%$$

Table V shows that the effect of assuming greater values of  $CV_{\eta}$  is to force a reduction in the regression estimate of  $\sigma_G$  and its UCL. (For comparison, the corresponding, unconstrained estimates are, from Table IV, 3.73% for  $CV_{\eta}$  and 8.88  $\mu\text{g}$  with UCL = 10.20  $\mu\text{g}$  for  $\sigma_G$ .) Therefore, the unconstrained regression estimate of  $\sigma_G$  is conservative with respect to possible underestimation of  $CV_{\eta}$ ; i.e., if the unconstrained estimate of  $CV_{\eta}$  is too low, then the unconstrained estimate of  $\sigma_G$  is probably too high.

## **References**

1. National Bureau of Standards; *An Evaluation of the Accuracy of the Coal Mine Dust Sampling Program Administered by the Department of the Interior*. Final Report to the Senate Committee on Labor and Public Welfare (1975).
2. Hall, T.A., Corn, M., Zeger, S., and Law, C.C.; "Respirable Dust and Free Silica Variation in Mine Environments," *Proceedings of the VIIth International Pneumoconiosis Conference*. DHHS (NIOSH) Publication No. 90-108, pp.1152-1159 (1990).
3. Breslin, J.A., Page, S.J., and Jankowski, R.A.; *Precision of Personal Sampling of Respirable Dust in Coal Mines*. USBM RI 8740 (1983).
4. Tomb, T.F., Treaftis, H.N., Mundell, R.L., and Parobeck, P.S.; *Comparison of Respirable Dust Concentrations Measured with MRE and Modified Personal Gravimetric Sampling Equipment*. USBM RI 7772 (1973).
5. Bowman, J.D., Bartley, D.L., Breuer, G.M., and Shulman, S.A.; *The Accuracy of Sampling Coal Mine Dust*. Draft NIOSH Report (1982).
6. Bowman, J., et al., *The Precision of Coal Mine Dust Sampling*, NIOSH, NTIS Publication No. PB-85-220721 (1984).
7. Snedecor, G.W. and Cochran, W.G.; "Logarithmic Transformation," Ch. 15.13 of *Statistical Methods, 7th Edition*. Iowa State University Press (1980).
8. Ku, H.H.; Notes on the Use of Propagation of Error Formulas, *Precision Measurement and Calibration, Ch. 5.3*. National Bureau of Standards (now National Institute of Standards and Technology), Special Pub. 300 (1969).
9. Draper, N.R. and Smith, H.; *Applied Regression Analysis, 2nd Edition*, pp 210-211. Wiley and Sons (1981).
10. "3R: Nonlinear Regression" and "3V: General Mixed Model Analysis of Variance"; BMDP Statistical Software, Inc., 12121 Wilshire Blvd., #300, Los Angeles CA 90025.
11. Vaughan, N.P et al; "Filter Weighing Reproducibility and the Gravimetric Detection Limit," *Annals of Occupational Hygiene* 33(3):331-337 (1989).
12. Bartley, D.L. et al; Respirable Aerosol Sampler Performance Testing. *Am Ind Hyg Assoc J.* 55(11):1036-1046 (1994).
13. Cook, R.D. and Weisberg, S. "Confidence Curves in Nonlinear Regression"; *Journal of the American Statistical Association* 85:544-551 (1990).

**Table 1.** Summary of multi-port sample data. Top entry within each cell is arithmetic mean. Bottom entry is sample Coefficient of Variation ( $CV_{total}$ ), in percent.

TEST	Number of Valid Samples	Sampling Time (Minutes)	Weight Gain (Micrograms)	Dust Concentration ( $mg/m^3$ )	480-Minute Equivalent ( $mg/m^3$ )
1	16	305.0 0.00	4166.0 5.62	9.42 5.62	5.99 5.62
2	16	311.9 0.18	5465.5 1.09	12.09 1.15	7.86 1.09
3	16	304.8 0.19	2370.7 3.13	5.37 3.24	3.41 3.13
4	16	283.6 0.18	4021.2 2.80	9.78 2.79	5.78 2.80
5	16	324.0 0.00	2304.1 3.81	4.91 3.81	3.31 3.81
6	16	320.5 0.16	6749.9 2.75	14.53 2.76	9.70 2.75
7	16	301.8 0.13	155.0 3.96	0.354 3.99	0.223 3.96
8	14	343.1 0.19	89.1 8.55	0.179 8.59	0.128 8.55
9	16	320.5 0.20	67.9 13.41	0.146 13.46	0.098 13.41
10	16	259.6 0.24	93.5 10.73	0.249 10.74	0.134 10.73
11	15	270.0 0.00	2756.2 2.96	7.04 2.96	3.96 2.96
12	16	330.0 0.00	931.7 2.87	1.95 2.87	1.34 2.87
13	15	360.0 0.00	2029.6 4.65	3.89 4.65	2.92 4.65
14	13	300.0 0.00	1003.0 11.19	2.31 11.19	1.44 11.19
15	16	330.0 0.00	64.5 14.37	0.135 14.37	0.092 14.37
16	16	330.0 0.00	80.2 6.20	0.168 6.20	0.115 6.20
17	16	330.0 0.00	68.1 11.51	0.142 11.51	0.098 11.51
18	15	300.0 0.00	84.5 18.62	0.194 18.62	0.121 18.62
19	16	325.0 0.00	61.5 17.46	0.131 17.46	0.088 17.46
20	16	270.0 0.00	102.1 10.98	0.261 10.98	0.146 10.98
21	15	330.0 0.00	953.9 3.60	1.99 3.60	1.37 3.60
22	16	305.0 0.00	519.5 3.38	1.18 3.38	0.747 3.38

NOTE:  $CV_{total}$  may differ for weight gain and dust concentration if sampling times vary within test.



**Table 2.** Results of 343-point regression analysis.

PARAMETER	RMLE <sup>A</sup>	Standard Error of RMLE	95-percent 1-tailed UCL
$\sigma_G^2$	83.15	14.46	106.94
$\sigma_G$ ( $\mu\text{g}$ ) <sup>B</sup>	9.12	N/A	10.34
$CV_\eta^2$	18.34	17.27	46.75
$CV_\eta$ (percent) <sup>B</sup>	4.28	N/A	6.84
$\sigma_\epsilon^2$	3.973	N/A	N/A
$\sigma_\epsilon$ (percent <sup>2</sup> ) <sup>B</sup>	1.99	N/A	N/A

<sup>A</sup>Restricted Maximum Likelihood Estimate. Restriction is to the class of unbiased estimates.

<sup>B</sup>Obtained by taking square root of estimate above.

**Table 3.** Results of 186-point random effects analysis of variance.

PARAMETER	RMLE <sup>A</sup>	Standard Error of RMLE	95-percent 1-tailed UCL
$\sigma_{\zeta}^2$	5.33	2.50	9.45
CV <sub>sampler</sub> (percent) <sup>B</sup>	2.3	N/A	3.07
$\sigma_{\pi}^2$	16.21	2.02	19.54
CV <sub>pump</sub> (percent) <sup>B</sup>	4.0	N/A	4.42
$\sigma_{\zeta}^2 + \sigma_{\pi}^2$	21.54	2.81	26.16
CV <sub>n</sub> (percent) <sup>B</sup>	4.6	N/A	5.11

<sup>A</sup>Restricted Maximum Likelihood Estimate. Restriction is to the class of unbiased estimates.

<sup>B</sup>Obtained by taking square root of estimate above.

**Table 4.** Results of 22-point aggregated regression analysis.

PARAMETER	Least Squares Estimate	Asymptotic Standard Error	95-percent 1-tailed UCL <sup>A</sup>
$\sigma_G$ ( $\mu\text{g}$ )	8.88	0.784	10.20
$\text{CV}_\eta$ (percent)	3.73	0.849	5.17
$\sigma_\epsilon$ (percent)	2.91	N/A	N/A

<sup>A</sup>Cook-Weisberg Upper Confidence Limit.

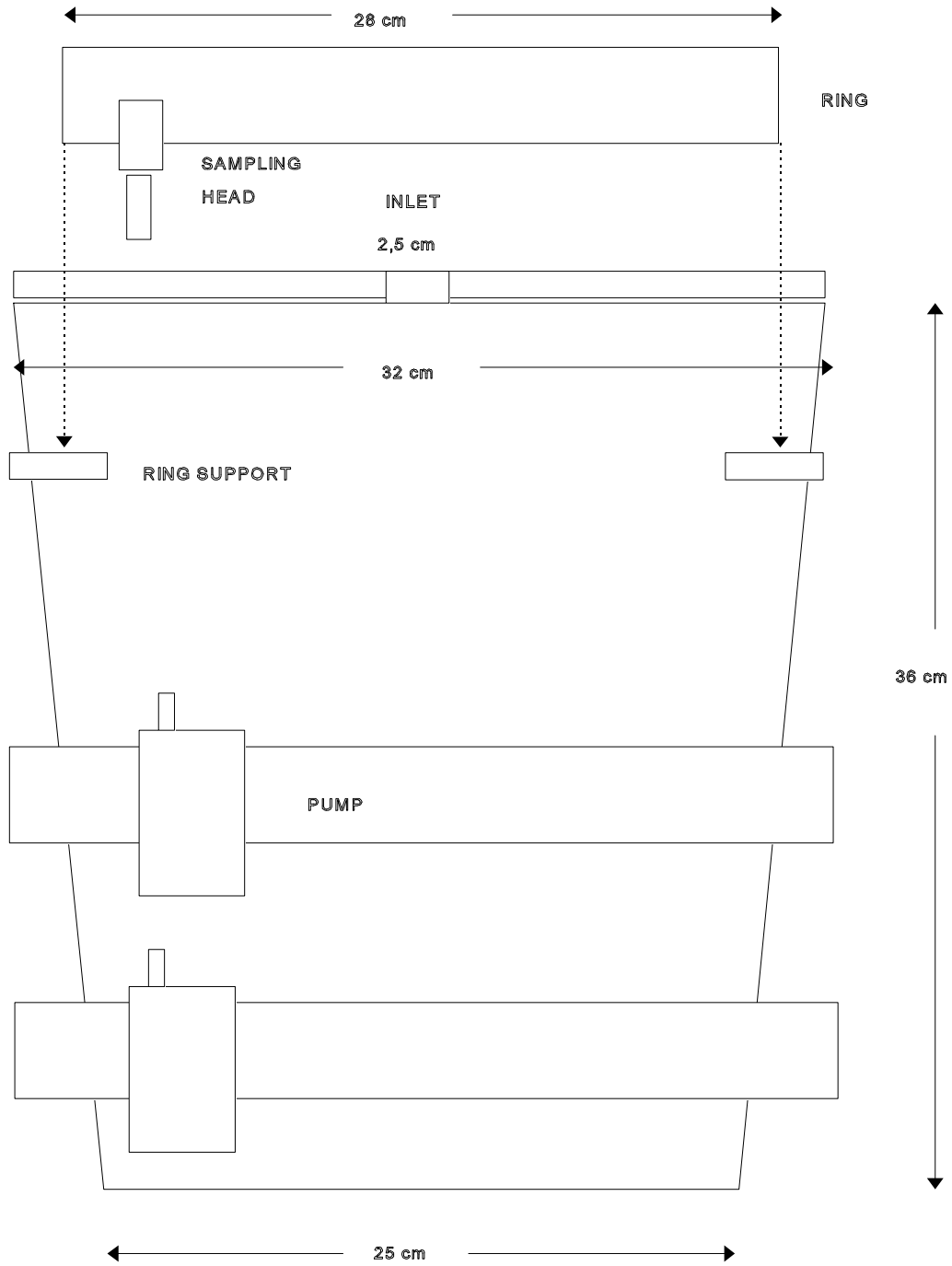
**Table 5.** Estimates of weighing imprecision, assuming specified values of imprecision from other sources.

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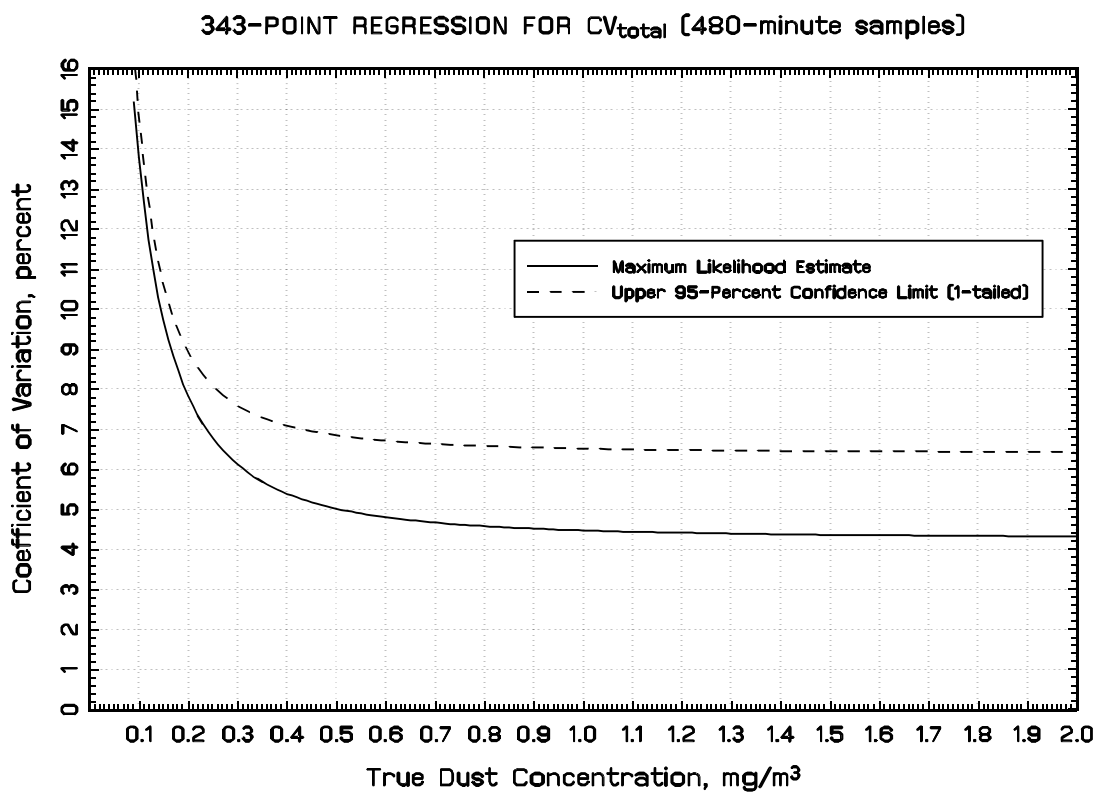
ASSUMED VALUE OF $CV_{\eta}$ (percent)	$\sigma_G$ ( $\mu\text{g}$ )		
	Least Squares Estimate	Asymptotic Standard Error	95-percent 1-tailed UCL <sup>A</sup>
5.19	8.43	0.830	9.84
6.56	7.88	1.037	9.61
9.95	5.67	2.243	9.00

<sup>A</sup>Cook-Weisberg Upper Confidence Limit.

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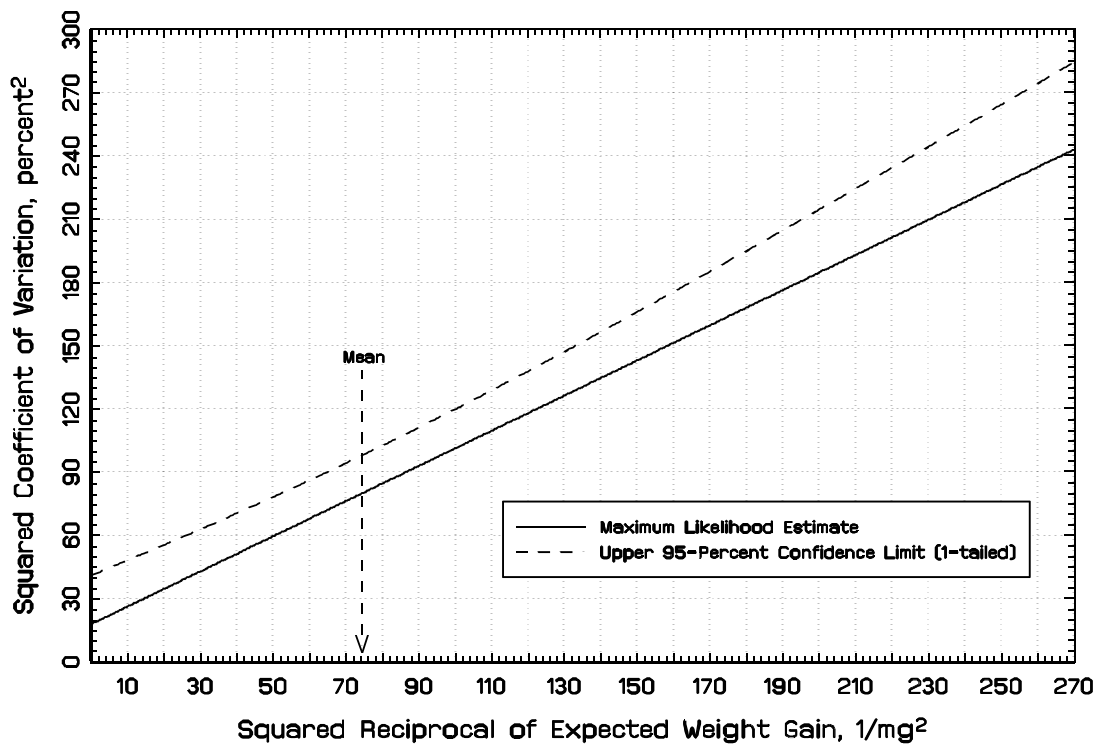


**Figure 1.** Schematic of sampler container.

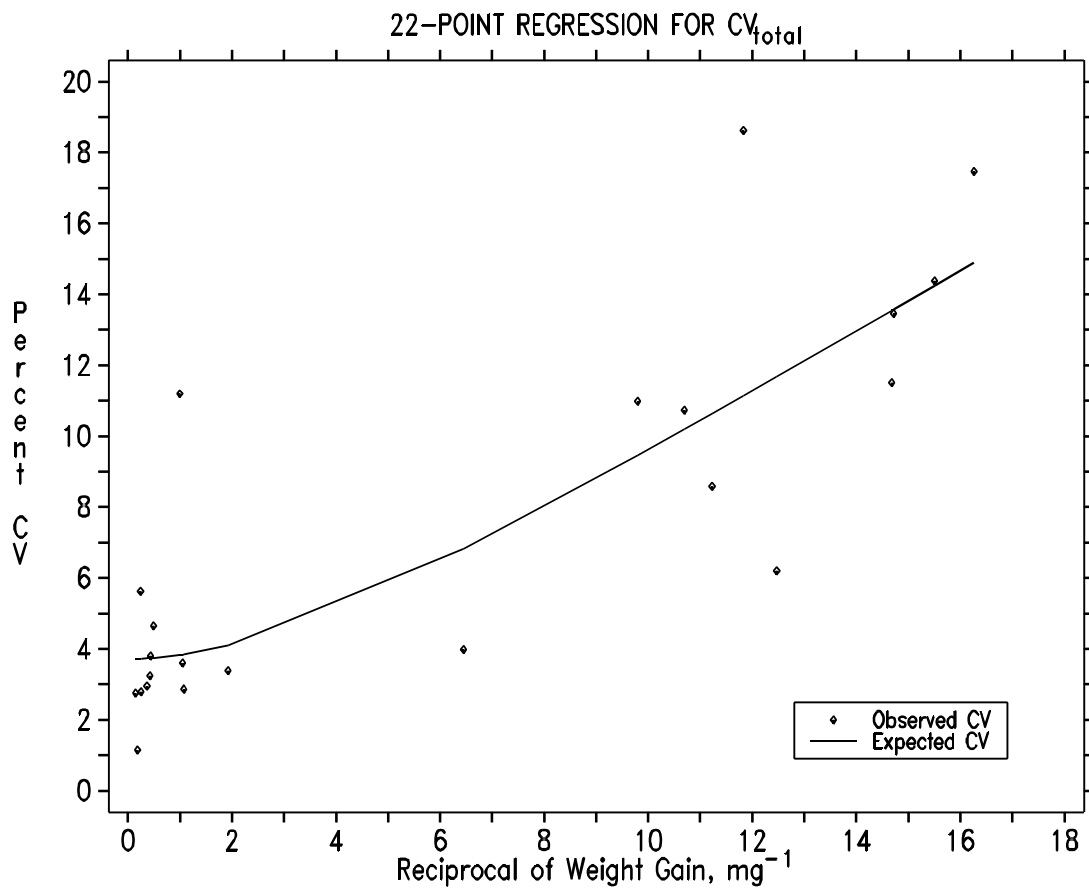


**Figure 2.** Expected measurement imprecision as a function of dust concentration sampled for 480 minutes.

### 343-POINT REGRESSION FOR $CV_{total}$



**Figure 3.** Square of expected measurement imprecision as a function of the squared reciprocal of expected weight gain.



**Figure 4.** Measurement imprecision as a function of the reciprocal of weight gain.