MEASUREMENT PRECISION WITH THE COAL MINE DUST PERSONAL SAMPLER

Jon Kogut,[A] Thomas F. Tomb,[B]
Paul S. Parobeck,[B] Andrew J. Gero,[B] Karen L. Suppers[B]

[A]Mine Safety and Health Administration, Denver Safety and Health Technology Center
[B]Mine Safety and Health Administration, Pittsburgh Safety and Health Technology Center

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 µg (0.001 mg). The average weight gain observed within tests ranged from 62 µg, collected over a 325-minute sampling period, to 6750 µg, 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 µg, with an upper one-tailed 95%-confidence limit (UCL) of 10.3 µg. 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_{total}) decreasing asymptotically from 7.8 percent (UCL = 8.9%) at dust concentrations of 0.2 mg/m^3 to 4.3 percent (UCL = 6.4%) at concentrations greater than 2.0 mg/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_{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_{total} at weight gains greater than 500 µg.

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

Introduction

Since 1970, a number of attempts[(1),(2),(3)] 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[(4)]. 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.[(5)]

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.[(2)] 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.[(6)] Imprecision due to these factors are quantified, respectively, as CV_{weight}, CV_{pump} (comprising the combined effects of variability in air flow and flow rate adjustment), and $CV_{sampler}$. Overall measurement imprecision (CV_{total}) can be obtained by combining the independent components of the measurement system using the following equation:

$$CV^2_{total} = CV^2_{weight} + CV^2_{sampler} + CV^2_{pump}. \qquad (1)$$

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.[(6)]

A different approach to estimating measurement imprecision is to conduct a study that derives CV_{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 re-weighed 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

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

where: X = dust concentration (mg/m^3)
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 μ_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 μ_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 $\bar{G}_i$.

The average dust concentration within the i^{th} test, $\bar{X}_i$, provides a relatively precise estimate of μ_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 $\bar{X}_i$ = 1.18 mg/m^3, with CV = 3.38%. Therefore, the standard error of 1.18 mg/m^3 as an estimate of μ_{22} is $3.38/\sqrt{16} = 0.845$ percent of 1.18, or 0.010 mg/m^3. Similarly, $\bar{G}_i$ is a good estimate of Γ_i, the weight gain expected in the i^{th} test, given a concentration equal to μ_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 μ_i. Using E{} to denote the expected value and Var{} to denote the variance of any random variable, note that $E\{X_{ij}\} = E\{\bar{X}_i\} = \mu_i$ and $Var\{X_{ij}\} = E\{(X_{ij} - \mu_i)^2\}$. The underlying, or true value of CV^2_{total}, as distinguished from an estimate based on a finite number of samples, is expressed by:

$$\begin{aligned} CV^2_{total} &= Var\{X_{ij}\} \div E^2\{X_{ij}\} \\ &= E\{(X_{ij} - \mu_i)^2\} \div \mu_i^2 \\ &= [n_i/(n_i - 1)]\cdot E\{(X_{ij} - \bar{X}_i)^2\} \div \mu_i^2 \end{aligned} \tag{3}$$

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

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

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

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

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.[6] Since the quantity of dust accumulated is proportional to dust concentration, this increase is reflected by constant values for $CV_{sampler}$ and CV_{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_{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 $Var\{X_{ij}\} = [1.38/(2t_i)]^2 Var\{G_i\}$. Consequently,

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

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

σ_G is the unknown standard deviation, in micrograms, of error in the weight gain measurement;

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

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

$$CV_i = (\sigma^2_G / \overline{G}^2_i + CV^2_\eta)^{1/2} + \epsilon_i \tag{6}$$

In this model, CV_i is the sample coefficient of variation observed in the i^{th} test, as shown in Table 1; and ϵ_i is the residual regression error -- i.e., the difference between estimated and true values of CV_{total} for the i^{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 σ_G and CV_η by regression on all 343 individual observations:

$$[n_i/(n_i - 1)]\cdot E\{(Y_{ij} - \overline{Y}_i)^2\} = \sigma^2_G / \overline{G}^2_i + CV^2_\eta \tag{7}$$

Equation 7 is equivalent to the regression model,

$$W_{ij} = \sigma^2_G / \overline{G}^2_i + CV^2_\eta + \epsilon_{ij} \tag{8}$$

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

Since the same observed value of $\overline{Y}_i$ 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 ϵ_{ij} consists of two random components: (1) a component (representing estimation error in $\overline{Y}_i$) 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 ϵ_{ij} as $\tau_i + \zeta_{ij}$, where τ_i is a random effect of the i^{th} test and ζ_{ij} is a random residual effect independent of τ_i. Therefore $E\{\tau_i\zeta_{ij}\} = 0$ and $\sigma_\epsilon^2 = Var\{\tau_i\} + Var\{\zeta_{ij}\}$. Because the ζ_{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:

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

$Var\{\tau_i\}$ represents uncertainty in the regression analysis due to estimating the true dust concentration within each test by the average of n_i measurements. Since $Var\{\tau_i\}$ 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, $Var\{\tau_i\}$ is estimated from the correlation observed within tests and added into the estimate for σ_ϵ^2 used to construct confidence limits.

To produce a regression curve for CV_{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 μ. For any dust concentration μ, the regression estimate of CV_{total} (expressed as a percentage) is:

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

where CV_{total} is now interpreted as an estimate obtained by substituting the corresponding least-squares estimators for σ_G and CV_η^2 into Equation 10.

The upper 95-percent confidence curve for this regression estimate of CV_{total} was estimated by application of the standard method to the linear model defined by Equation 8.[9] 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 μ sampled for 480 minutes, the 95-percent 1-tailed upper confidence limit (UCL) for CV_{total} obtained from the 343-point regression is:

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

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

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

$z_i = 1/\bar{G}_i^2$ 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 µg, the estimated value of σ_G declines from 1.8% of a 500 µg weight gain to 0.5% of a 2000 µg weight gain (see results below). Therefore, weighing imprecision contributes little to CV_{total} for those 12 tests showing average weight gain greater than 500 µg, and its effect can be assumed to be a negligible constant. Doing so makes it possible to estimate $CV_{sampler}$ and CV_{pump} from the 186 valid samples collected in these 12 tests.

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

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

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

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^{th} sampler in the i^{th} test;

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

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

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

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

$$\begin{aligned} \mathrm{Var}\{Y_{ij}\} &\approx CV_{total}^2 \\ &\approx CV_{sampler}^2 + CV_{pump}^2 \\ &= CV_\eta^2 \\ &\approx \sigma_\varsigma^2 + \sigma_\pi^2 \end{aligned} \tag{13}$$

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

The 343-point regression and ANOVA analyses described above were both carried out using BMDP module 3V.[10] 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 σ_G and CV_η, 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 σ_ϵ 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 CV_{total} and its UCL plotted, as a function of dust concentration (µ), 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_{total}). The slope of the regression line illustrates the changing contribution of CV_{weight} to CV_{total}. As dust concentration increases, CV_{weight} approaches zero, and the regression line asymptotically approaches the combined effect of $CV_{sampler}$ and CV_{pump}. At an average dust concentration of 0.1 mg/m^3 sampled over a 480-minute period, the regression estimate for CV_{total} is 13.8 percent (UCL = 14.9%). From there, the regression estimate for CV_{total} drops to 7.8 percent (UCL = 8.9%) at dust concentrations of 0.2 mg/m^3 and declines asymptotically to 4.3 percent (UCL = 6.4%) at concentrations greater than 2.0 mg/m^3.

By Equation 11, the distance between the regression estimate of CV_{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_{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_{total}^2 and its UCL are plotted in Figure 3 as a function of $z = 1/\Gamma^2$, where Γ 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 σ_G and slightly increase the estimate for CV_η. Using a Huber constant of 23, the estimate for σ_G is reduced from 9.1 µg to 7.8 µg, while the estimate for CV_η is increased from 4.3% to 4.4%. Huber constants are defined in the documentation for BMDP Module 3R.[10]

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

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

Discussion

Based on the regression analysis summarized in Table III, the standard deviation of errors in recorded weight gain (σ_G) is 9.1 µg (UCL = 10.3 µg). The corresponding estimate of imprecision deriving from other sources (CV_η) 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_η were actually greater than 4.3 percent, then this would force a decrease in the regression estimate of σ_G. Therefore, the 9.1 µg estimate for σ_G may be regarded as conservative if CV_η is being underestimated. The regression estimate for σ_G derived in the present study is consistent with previously reported results for pre- and post-weighing to a microgram within the same laboratory.[(6),(11)]

For dust samples collected over a 480-minute period, Table II implies that CV_{total} and its UCL are estimated by the graph shown in Figure 2. Weight gains greater than 500 µg correspond to dust concentrations greater than 0.72 mg/m^3, based on 480-minute samples. For such concentrations, the ANOVA estimate of $CV_{total} \approx CV_\eta = 4.6\%$, 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_η, representing an upper bound on imprecision not attributable to weighing error, falls well below the regression UCL of 6.8%.

The ANOVA estimate of $CV_{sampler} = 2.3\%$ (UCL = 3.1%) derived here falls below an estimate of $CV_{sampler} = 5\%$ previously published by Dr. David Bartley et al.[(12)] 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...". [(6)]

Conclusions

As measured by CV_{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 mg/m^3 and less than 7 percent for dust concentrations at or above 0.5 mg/m^3. The corresponding maximum likelihood estimates for CV_{total} are 7.8 percent and 5.0 percent, respectively. At dust concentrations greater than 2.0 mg/m^3, the maximum likelihood estimate for CV_{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_{η}, which is statistically consistent with that derived from the regression analysis. (The estimates for CV_{pump} and CV_{η} are inflated slightly by CV_{weight}, averaged over those 12 tests in which the average observed weight gain exceeded 500 µg.) The maximum likelihood estimate for the standard deviation of errors in recorded weight gain (σ_G) was determined to be 9.1 µg, with a UCL of 10.3 µg. Since the ANOVA estimate for CV_{η} slightly exceeds the corresponding regression estimate and the regression estimates for CV_{η} and σ_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(10) on the 22 tests as summarized in Table I. Table IV contains the resulting estimates of σ_G and CV_{η}, as defined in Equation 5, along with the standard error of the regression estimate, σ_{ϵ}. 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 σ_G and CV_{η} were calculated by the BMDP statistical software. Cook-Weisberg limits more accurately represent nonlinear regression parameters than the more commonly used symmetric Wald approximations.(13)

From Table IV, the distance between the 22-point nonlinear regression estimate for σ_G and its UCL is 10.20 - 8.88 = 1.32 µg. The corresponding value for the 343-point linear regression, obtained from Table II, is 10.34 - 9.12 = 1.22 µg. 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 σ_G than the 343-point linear regression. The confidence interval for CV_{η}, on the other hand, is significantly more focussed. In particular, the UCL for CV_{η} calculated from the 22-point model falls well below the corresponding value in Table II and is closer to the UCL for CV_{η} 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_{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 σ_ϵ 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 (σ_G), when the non-weighing component (CV_η) 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_η correspond to the following assumptions for CV_{pump} and $CV_{sampler}$:

CV_η = 5.19% CV_{pump} = 4.24% and $CV_{sampler}$ = 3%

CV_η = 6.56% CV_{pump} = 4.24% and $CV_{sampler}$ = 5%

CV_η = 9.95% CV_{pump} = 4.24% and $CV_{sampler}$ = 9%

Table V shows that the effect of assuming greater values of CV_η is to force a reduction in the regression estimate of σ_G and its UCL. (For comparison, the corresponding, unconstrained estimates are, from Table IV, 3.73% for CV_η and 8.88 µg with UCL = 10.20 µg for σ_G.) Therefore, the unconstrained regression estimate of σ_G is conservative with respect to possible underestimation of CV_η: i.e., if the unconstrained estimate of CV_η is too low, then the unconstrained estimate of σ_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 os 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[A]	Standard Error of RMLE	95-percent 1-tailed UCL
σ_G^2	83.15	14.46	106.94
σ_G (µg)[B]	9.12	N/A	10.34
CV_η^2	18.34	17.27	46.75
CV_η (percent)[B]	4.28	N/A	6.84
σ_ϵ^2	3.973	N/A	N/A
σ_ϵ (percent^2)[B]	1.99	N/A	N/A

[A]Restricted Maximum Likelihood Estimate. Restriction is to the class of unbiased estimates.
[B]Obtained by taking square root of estimate above.

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

PARAMETER	RMLE[A]	Standard Error of RMLE	95-percent 1-tailed UCL
σ^2_ς	5.33	2.50	9.45
$CV_{sampler}$ (percent)[B]	2.3	N/A	3.07
σ^2_π	16.21	2.02	19.54
CV_{pump} (percent)[B]	4.0	N/A	4.42
$\sigma^2_\varsigma + \sigma^2_\pi$	21.54	2.81	26.16
CV_η (percent)[B]	4.6	N/A	5.11

[A]Restricted Maximum Likelihood Estimate. Restriction is to the class of unbiased estimates.
[B]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[A]
σ_G (µg)	8.88	0.784	10.20
CV_η (percent)	3.73	0.849	5.17
σ_ϵ (percent)	2.91	N/A	N/A

[A]Cook-Weisberg Upper Confidence Limit.

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

ASSUMED VALUE OF CV_η (percent)	σ_G (µg)		
	Least Squares Estimate	Asymptotic Standard Error	95-percent 1-tailed UCL[A]
5.19	8.43	0.830	9.84
6.56	7.88	1.037	9.61
9.95	5.67	2.243	9.00

[A]Cook-Weisberg Upper Confidence Limit.

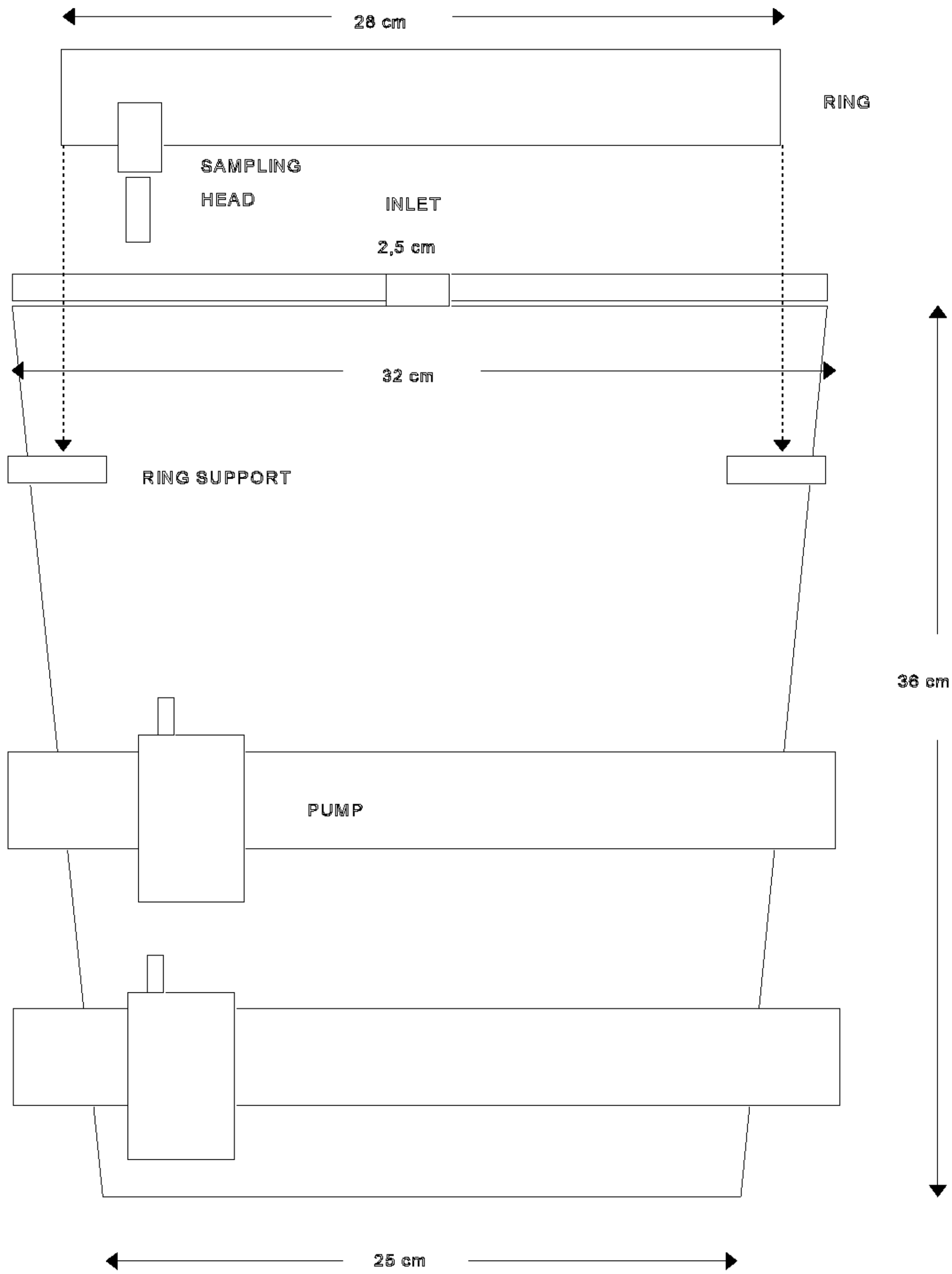

Figure 1. Schematic of sampler container.

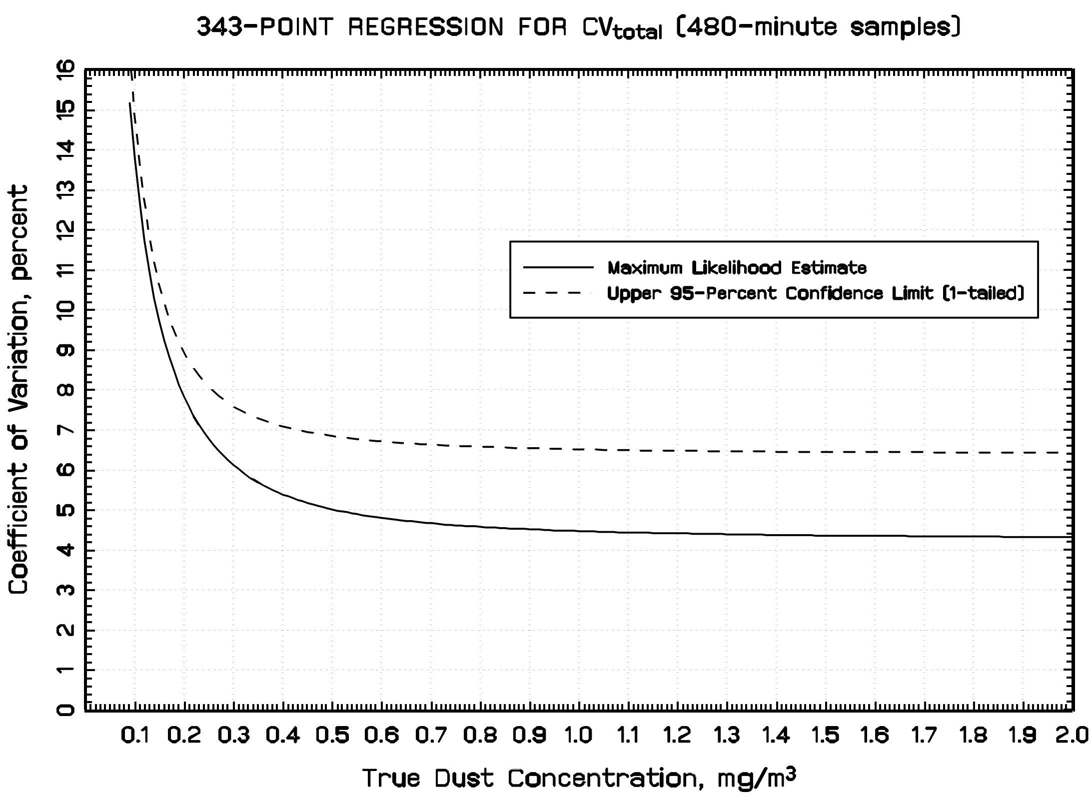

Figure 2. Expected measurement imprecision as a function of dust concventration sampled for 480 minutes.

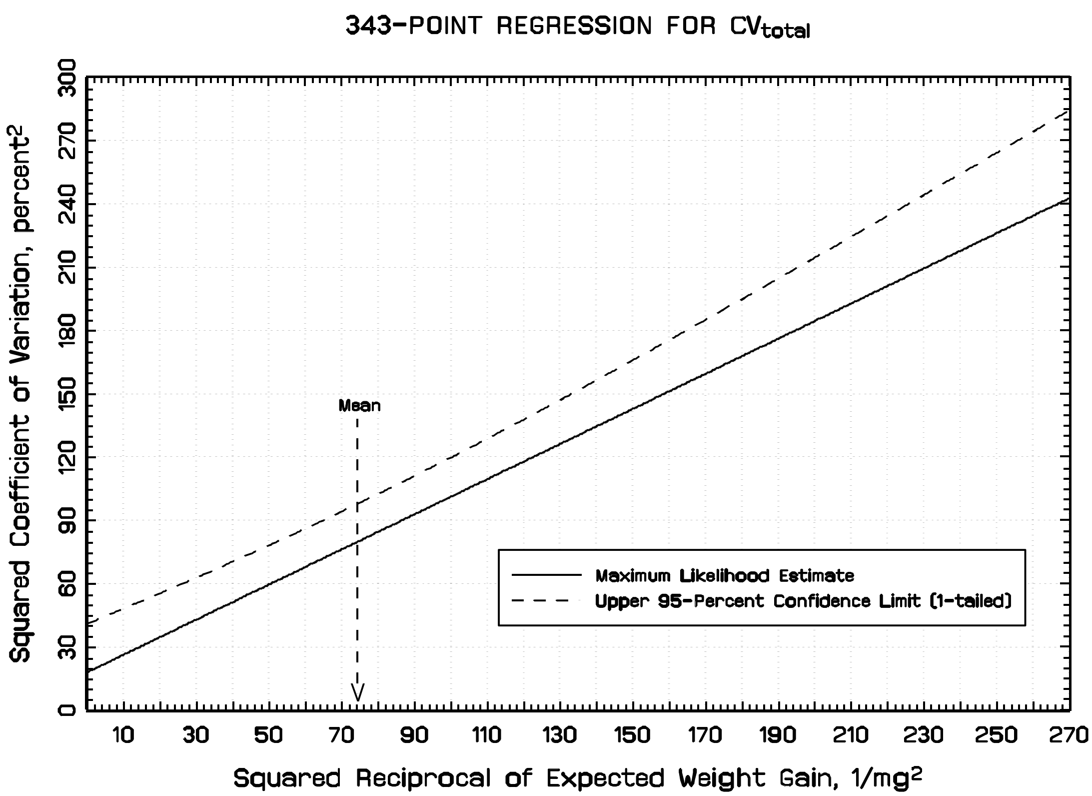

Figure 3. Square of expected measurement imprecision as a function of the squared reciprocal of expected weight gain.

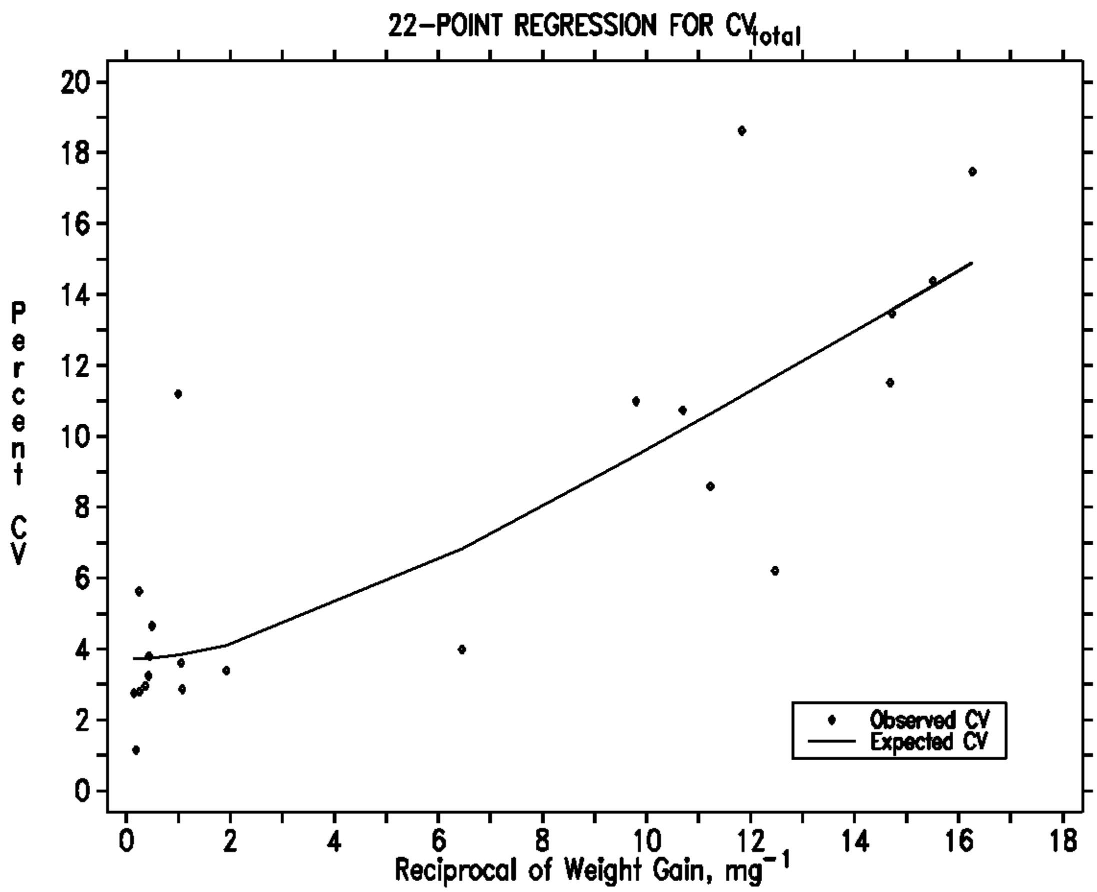

Figure 4. Measurement imprecision as a function of the reciprocal of weight gain.