AN EXAMPLE FROM PROFICIENCY TESTING OF THE APPLICATION OF PRINCIPAL COMPONENTS TO THE ESTIMATION OF VARIANCE COMPONENTS IN MIXED MODELS

Stanley A. Shulman, National Inst. for Occupational Safety and Health
NIOSH, 4676 Columbia Parkway, MS-R3, Cincinnati, OH 45226

Key Words: Variance Components; Interaction; Multivariate Methods

Abstract

A common problem in lab proficiency testing is the estimation of measurement error and lab-to-lab variability, when duplicate samples are not available. Since measurement errors are often assumed to constitute a statistically independent set, the variance-covariance matrix should be the sum of a diagonal matrix (corresponding to measurement error) and a matrix different from an identity matrix [1]. Multivariate statistical methods and mixed model methods provide tests that a variance-covariance matrix is of this form. In this paper, these methods are applied to data from an industrial hygiene laboratory proficiency testing program, the Proficiency Analytical Testing Program(PAT). Variances and covariances are assumed to be approximately proportional to an unknown power of the mean, and various possibilities for the form of the associated variance-covariance matrix are considered. The form depends on the scale on which the analyses are carried out, the possibility that the measurement error and non-measurement error have variances that are not proportional to the same power of the mean, and the dimensionality of the non-identity matrix. Examples are presented from PAT.

1)Introduction

Suppose that data are in a two-way table, where the column variables have fixed means and the row variables are randomly chosen from a population. An example is data from the Proficiency Analytical Testing Program(PAT). Quarterly, participating labs receive four asbestos samples at fiber loadings unknown to them. Each lab must report a fiber count for each of the samples. In this example, each column refers to a different sample set, and the entries in the columns are the sample values, as determined by each lab. The entries by row are all the determinations by the same lab, each of which is assumed to be randomly chosen from a larger population. Aims in the analysis of these kinds of data are the determination of covariance structure, the estimation of variance components, and the estimation of coefficient of variation (CV=standard deviation/mean).

A suggestion of Srivastava and Khatri [1] is to separate independently distributed measurement error from variances associated with measuring devices (here laboratories) by designating the following form for the covariance structure:

$$\boldsymbol{\Sigma} = \mathbf{K} + w\mathbf{I} = \boldsymbol{\Lambda}\boldsymbol{\Lambda}' + w\mathbf{I}, \quad (1)$$

where bold symbols are used for matrices or vectors. $\boldsymbol{\Sigma}$ is the variance-covariance matrix for the samples (in our example, a 4x4 matrix), $\mathbf{I}$ is the 4-dimensional identity matrix, and the rank of $\boldsymbol{\Lambda}$ depends on the particular problem. It may have as few as one column. In the work to be presented here, $w\mathbf{I}$ is replaced by a diagonal matrix $\mathbf{D}(\mathbf{w}_i)$, to allow different variances for different samples. Thus, (1) will be rewritten as:

$$\boldsymbol{\Sigma} = \mathbf{K} + w\mathbf{I} = \boldsymbol{\Lambda}\boldsymbol{\Lambda}' + \mathbf{D}(\mathbf{w}_i). \quad (2)$$

The structure of $\boldsymbol{\Lambda}$ is studied, and goodness of fit is assessed via maximum likelihood. A simple form for the variances and covariances is proposed that leads to a simple form for the CV. Related work has been done by Song[2] and Goodman and Haberman[3]. Multivariate methods shown here use suggestions of Srivastava and Khatri[1], based partly on Kshirsagar[4].

The results will be presented as follows. Example data are introduced in Section 2 as the basis of the models presented in Sections 3 through 6. The estimation method for the parameters is discussed in section 7. In Section 8, an example is presented which can be successfully modeled under several alternative models, though all these models use $\boldsymbol{\Lambda}$ with just one column. Simulation results are presented in Section 9, to indicate how precisely the exponent parameter in the model of interest can be estimated. Section 10 presents an example which can be fitted by only one of the models shown in Section 8. In Section 11 , a situation is described where the single column form of $\boldsymbol{\Lambda}$ is not adequate. Simulation results relevant to this model and example data are shown in sections 12 and 13. Results are summarized in section 14.

2)Example

Figure 1 shows asbestos data from PAT Round 60. The data have been transformed to the square root scale to achieve approximate normality. There were four sample sets. In the figure, the natural logs of the 4 variances and 6 pairwise covariances associated with these sample sets are plotted. Variances are plotted against twice the natural log of the associated sample mean.

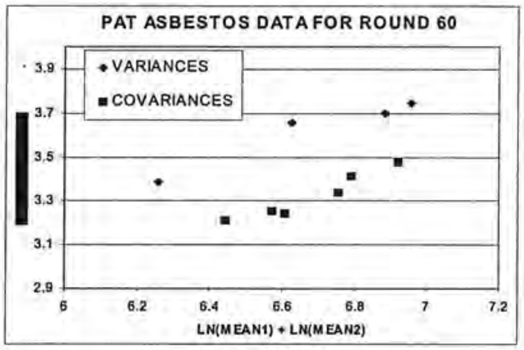
Covariances are plotted against the sum of the natural logs of the two sample means. For these data, the variances and covariances appear to fall on parallel straight lines.

covariance ~[(mean1)(mean2)]^a (3)

variance ~(mean1)^{2a}, for some power "a," where the "~" indicates proportionality.

In this example a was approximately 0.5.

Figure 1



3)Statistical Model

A model for measurements x_{tslk} at sampling time (or round) t is the mixed model:

$x_{tslk}= (\mu_{ts}) + (b_{tsl} + w_{tslk})$,
(4)

t=t,...,T, s=1,..,S, l=1,...,L, k=1. In (4) Greek letters indicate fixed effects, pertaining to overall mean or individual sample means, and Latin letters indicate random effects. In (4) s denotes the sample sets in round t; l denotes the labs participating in round t; b_{tsl} is the random term indicating between lab variability, or variability of lab determinations over samples. For lab l and round t, the S b_{tsl} terms are correlated, since they are random terms that apply to the same lab. Conversely, the within lab random effects w_{tslk} account for measurement error and are assumed to be statistically independent of the b_{tsl}s and to constitute a statistically independent set. Model (4) describes a mixed model [5]. With one lab determination (k=1) for each sample set, it is not apparent how to separate interaction between lab and sample set from the within error. (In the presentation to follow, we remove the subscript k, since it always equals 1.) When model (2) is applicable, d_{tsl} can be separated from w_{tsl}.

A class of models consistent with (2) is suggested by the example data. Assume that the vector $(x_{t1l}, x_{t2l},...,x_{tSl})' \overset{d}{=} N_S(\mathbf{M}, \Sigma)$, where $\mathbf{M}=(\mu_{t1}, \mu_{t2}, ..., \mu_{tS})'$, where "$\overset{d}{=}$" indicates "distributed as." The vector is an S-dimensional normal distribution with variance-covariance matrix, $\mathbf{\Sigma}$, estimated by:

$\boldsymbol{S}= 1/(L\text{-}1)\ \Sigma_l (x_l - \bar{x})(x_l - \bar{x})'$, (5)

where $\mathbf{x}_l=(x_{t1l}, x_{t2l},..., x_{tSl})'$, & $\bar{x}=(\bar{x}_{t1.}, \bar{x}_{t2.}, ..., \bar{x}_{tS.})$, for $\bar{x}_{ts..}$ average of x_{tsl}s over all l. The example suggests that the variance components of (4) may have following form:

Between Lab Var(b_{tsl}) ~ $L_t^2\,[\mu_{t,s}]^{2a(t)}$, (6)

& Within Lab Var(w_{tsl})

=Measurement error Var(w_{tsl}) ~$m_t^2[\mu_{t,s}]^{2a(t)}$,

for power a(t) varying by round. The proportionality factors are L_t^2 for variance between labs & m_t^2 for measurement error variance.

Under this power of mean model for variances and covariances:

Var(x_{tsl})=[$L_t^2 + m_t^2$] $[\mu_{t,s}]^{2a(t)}$ (7)

Cov(x_{tsl},x_{tvl})=$L_t^2\,[\mu_{ts}]^{a(t)}\,[\mu_{tv}]^{a(t)}$ for sample sets in the same round t. The model can be extended to correlations between sample sets in different rounds:

Cov$(x_{tsl},x_{t+1,vl})$=$(L_{t,t+1})$ $[\mu_{ts}]^{a(t)}$ $[\mu_{t+1,v}]^{a(t+1)}$, for determinations by the same lab in sample sets in consecutive rounds (t, t+1), where $L_{t,t+1}$ is a proportionality factor. Under assumptions (6), model (4) becomes:

$x_{tsl}= (\mu_{ts}) + (b_{tsl} + w_{tsl})$

$= \mu_{ts} + \{ [\mu_{ts}]^{2a(t)}\}^{0.5}\, [\, (L_t)\, b^*_{tl} + (m_t)\, w^*_{tsl}\,]$, (8)

where $\{b^*_{tl}\}$, $\{w^*_{tsl}\}$ IID $\overset{d}{=}$ N(0,1), & IID means "independent, identically distributed." The $\{w^*_{tsl},\ s=1,..,S\ ;\ l=1,2,...L;\ t=1,..T\}$ correspond to measurement error and are statistically independent of the $\{b^*_{tl},\ l=1,..,L \text{ and } t=1,..T\}$.

Note: In (8), the interaction term b_{tsl} is assumed to be of the form $\{ [\mu_{ts}]^{2a(t)}\}^{0.5}\,(L_t)\, b^*_{tl}$, which indicates that any differences of lab determinations for different samples is due entirely to the function of the mean $f(\mu_{ts})=\{ [\mu_{ts}]^{2a(t)}\}^{0.5}\, L_t$. If the function $f(\mu_{ts})$ does not account for all these differences, then $b_{tsl}= \{ [\mu_{ts}]^{2a(t)}\}^{0.5}$ $(L_t)\, b^*_{tl} + (bb)^*_{tsl}$, where $(bb)^*_{tsl} = b_{tsl} - \{ [\mu_{ts}]^{2a(t)}\}^{0.5}\,(L_t)\, b^*_{tl} =$ $b_{tsl} - f(\mu_{ts}) b^*_{tl}$, and the appropriate model is:

$x_{tsl}= (\mu_{ts}) + (b_{tsl} + w_{tsl})$ = (8a)

$\mu_{ts} + (\{ [\mu_{ts}]^{2a(t)}\}^{0.5}\, L_t\, b^*_{tl} + (bb)^*_{tsl}\,) + \{ [\mu_{ts}]^{2a(t)}\}^{0.5}\, m_t\, w^*_{tsl}$

,

where $\{b^*_{tl}\}$, $\{w^*_{tsl}\}$ IID $\overset{d}{=}$ N(0,1) and all $\{w^*_{tsl}\}$ are statistically independent of all $\{b^*_{tl},\ (bb)^*_{tsl},\ s=1,...,S\}$. A possible understanding of the $(bb)^*_{tsl}$ is that for each sample set, the relationships of eq (7) may only be approximate, in that the variances for the different samples may have slightly different powers "a", or, alternatively, there are peculiarities about the samples that affect all labs. In either case, correlation would be expected between sample sets in excess of that produced by the dependence on $f\ (\mu_{ts})$. Comparison of the model based on (8) with that based on (8a) is a measure of lack-of-fit.

4) When Data Have Constant CV

If a(t)=1 in model (8) and the data are normally distributed, the CV is constant and the natural log transformation yields approximately constant variance=CV^2[6]. Following the ln transformation, the approximate new model is:

$$\ln(x_{tsl}) \cong \mu^*_{ts} + [L_t b^{**}_{tsl} + m_t w^{**}_{tsl}],$$
(9)

where "$\cong$" means "approximately."

$\mathrm{Var}[\ln(x_{tsl})] \cong [L_t^2 + m_t^2] = CV^2$ on the original scale, independent of concentration.

The variance-covariance matrix is equivalent to (2) (setting $\Lambda = L_t \mathbf{1}_S$): $\Sigma_S = L_t^2 \mathbf{1}'_S \mathbf{1}'_S + m_t^2 I_S =$

$$\begin{vmatrix} L_t^2 & L_t^2 & \dots & L_t^2 \\ L_t^2 & L_t^2 & \dots & L_t^2 \\ L_t^2 & L_t^2 & \dots & L_t^2 \\ L_t^2 & L_t^2 & \dots & L_t^2 \end{vmatrix} + \begin{vmatrix} m_t^2 & 0 & \dots & 0 \\ 0 & m_t^2 & \dots & 0 \\ & & \dots & \\ 0 & 0 & \dots & m_t^2 \end{vmatrix}$$

In the above expression, Σ_S is the SxS variance matrix; $\mathbf{1}_S$ is an S-dimensional vector of 1s; $\mathbf{I}_S$ is the S-dimensional identity matrix. Statistical tests for the above form of Σ require normality. The correlation, ρ, is $L_t^2 / [L_t^2 + m_t^2]$, and the correlation matrix is

$$\rho \mathbf{1}\mathbf{1}' + (1-\rho)\mathbf{I} \qquad (10)$$

5) Alternatives to (8) When Constant CV Lacking, Or When a=1 & Lack Normality on Log Scale

A)From (2), (4), (6), and (8),

$$\Sigma = \{\sigma_{ij}\} = \Lambda\Lambda' + D(w_i) \qquad (11)$$

$= \mathbf{D}(\mu^a_{ts})[\ L_t^2\ \mathbf{1}\mathbf{1}']\mathbf{D}(\mu^a_{ts}) + m_t^2\mathbf{D}(\mu^a_{ts})\ \ \mathbf{D}(\mu^a_{ts})$, for

$$= D(\mu^a_{ts}) \begin{vmatrix} L_t^2 & L_t^2 & \dots & L_t^2 \\ L_t^2 & L_t^2 & \dots & L_t^2 \\ L_t^2 & L_t^2 & \dots & L_t^2 \\ L_t^2 & L_t^2 & \dots & L_t^2 \end{vmatrix} D(\mu^a_{ts}) + D(\mu^a_{ts}) \begin{vmatrix} m_t^2 & 0 & \dots & 0 \\ 0 & m_t^2 & \dots & 0 \\ & & \dots & \\ 0 & 0 & \dots & m_t^2 \end{vmatrix} D(\mu^a_{ts})$$

a=a(t). Since $\sigma_{sv} = L_t^2\ \mu^a_{ts}\ \mu^a_{tv}$ for $s \neq v$, and $\sigma_{ss} = [L_t^2 + m_t^2]\ \mu^{2a}_{ts}$, $\rho = \mathrm{corr} = L_t^2\ /\ [L_t^2 + m_t^2]$. Thus, the same correlation matrix results that was obtained for constant CV after the natural log transformation: $\rho\mathbf{1}\mathbf{1}' + (1-\rho)\ \mathbf{I}$. In terms of ρ, the variance-covariance matrix may be rewritten as follows:

$$\Sigma = [L_t^2 + m_t^2]\ \ \mathbf{D}(\mu^a_{ts})\ [\ \rho\mathbf{1}\mathbf{1}' + (1-\rho)\ \mathbf{I}\ ]\mathbf{D}(\mu^a_{ts}), \qquad (12)$$

and the total CV may be expressed in terms of model parameters as: $CV_{ts} = [L_t^2 + m_t^2\ ]^{0.5}\ [\mu_{t,s}]^{[a(t)-1]}$

(13)

For a(t)=1, samples have constant CV= $[L_t^2 + m_t^2\ ]^{0.5}$.

If model (8a) is the correct model, then model (11) is not correct, and the form of Λ must be changed, most easily by adding at least one more column. This will be discussed more during discussion of the examples.

B) The between and measurement error components have different dependency on mean $\mu_{t,s}$.

$$\Sigma = L_t^2\ [\mathbf{D}(\mu^a_{ts})\mathbf{1}]\ [\mathbf{D}(\mu^a_{ts})\mathbf{1}\ ]' + m_t^2 \mathbf{D}(\mu^{a'}_{ts})\ \mathbf{I}\ \mathbf{D}(\mu^{a'}_{ts}), \qquad (14)$$

$$CV_{ts} = \{\ L_t^2\ \ [\mu_{t,s}]^{[2a(t)-2]} \ \ + m_t^2\ \ [\mu_{t,s}]^{[2a'(t)-2]}\ \}^{0.5}$$

where $a \neq a'$. An aim here is to determine how precisely "a" can be estimated.

6) A More General Form for Eq (8)

(8) can be rewritten as :

$$x_{tsl} = (\mu_{ts}) + (b_{tsl} + w_{tsl}) = \mu_{ts} + [\ f(\mu_{ts})\ b^*_{tl} + g(\mu_{ts}) w^*_{tsl}\ ], \qquad (15)$$

where $f(\mu_{ts})$ and $g(\mu_{ts})$ are functions of the sample means μ_{ts}, and the terms b^*_{tl} and w^*_{tsl} are defined following equation (8). Eq. (15) is similar to that given by Song[2] If model (15) seems sensible, in that the variances of different sample sets in the same round differ only because the sample means differ, then comparison of the functions $f(\mu_{ts})$ and $g(\mu_{ts})$ is of interest.

Under model (15), $\mathrm{Var}(x_{tsl}) = f^2(\mu_{ts}) + g^2(\mu_{ts})$, and $\mathrm{Cov}(x_{tsl}, x_{tvl}) = f(\mu_{ts})\ f(\mu_{tv})$.

$\mathrm{Corr}(x_{tslk}, x_{tvlk}) =$

$f(\mu_{ts})\ f(\mu_{tv}) / \{[f^2(\mu_{ts}) + g^2(\mu_{ts})]^{0.5}\ [f^2(\mu_{tv}) + g^2(\mu_{tv})]^{0.5}\}$

$= 1/\{[\ 1 + g^2(\mu_{ts})/ f^2(\mu_{ts})]^{0.5}\ [1 + g^2(\mu_{tv})/ f^2(\mu_{tv})\ ]^{0.5}\ \}$.

Thus, an equicorrelation matrix will result if and only if $: g^2(\mu_{ts})/ f^2(\mu_{ts}) = g^2(\mu_{tv})/ f^2(\mu_{tv})$, for each of the sample sets s and v, or

$g^2(\mu_{ts}) = [g^2(\mu_{tv})/ f^2(\mu_{tv})]\ f^2(\mu_{ts}) = \upsilon\ f^2(\mu_{ts})$,

(16)

for each sample set.

Thus, under model (15) for the S sample sets in the round t, an equicorrelation matrix results if and only if $g(\mu_{ts})$ is a multiple of $f(\mu_{ts})$ for each of these S sample sets. For proficiency test data, where sample sets differ only in the mass of analyte, equicorrelation within round may seem reasonable. In this case, it may be sensible to test whether $g(\mu_{ts})$ is a constant multiple of $f(\mu_{ts})$.

7)Maximum Likelihood Estimation of "a" & Likelihood Ratio Criterion for Adequacy of Model

For multivariate normal data, the maximum likelihood estimates are those that maximize the multivariate normal likelihood, where $\mathbf{\Sigma}$ is one of the two equivalent forms (11) or (12). Suppose there are four sample sets in a round. The number of parameters is seven: $\mathbf{D}(\mu_{ts}^{a})$ has four diagonal elements; also, the values of $L_t^2 + m_t^2$, ρ, and a must be estimated. The question of identification of parameters [7] must be considered. For model parameters to be identified, they must be able to be expressed as functions of the elements of the variance -covariance matrix. With four sample sets, there are four equations for variances, and six equations for covariances:

$Var(x_{tslk}) = [L_t^2 + m_t^2][\mu_{ts}]^{2a(t)}$, for each sample set s.

$Cov(x_{tslk}, x_{tvlk}) = \rho[L_t^2 + m_t^2]\,[\mu_{ts}]^{a(t)}\,[\mu_{tv}]^{a(t)}$, for each pair of sample sets s and v.

After conversion to the natural log scale:

$\ln[Var(x_{tslk})] = \ln[L_t^2 + m_t^2] + 2a(t)\ln[\mu_{ts}]$

$\ln[Cov(x_{tslk}, x_{tvlk})] = \ln(\rho) + \ln[L_t^2 + m_t^2] + a(t)\{[\ln[\mu_{ts}] + \ln[\mu_{tv}]\}$

These equations lead to a solution for ρ. However, since $0.5\{\ln[Var(x_{tslk})] + \ln[Var(x_{tvlk})]\}$ $= \ln[Cov(x_{tslk}, x_{tvlk})] - \ln(\rho)$, there is no further information to be obtained from the covariances. Thus, there are really just four equations for the six remaining parameters, and there can be no unique solution, without additional constraints.

Since most of the sample sets for proficiency tests are large, the sample means $\bar{x}_{ts}$ will be close to the true values, μ_{ts}. (For the example data of Figure 1, sample means and sample size are given in Section 8.) In the models used here, we propose to substitute the sample means for the theoretical values, and then use the likelihood approach to estimate power "a." If two of the means are specified, then "a" and $[L_t^2 + m_t^2]$ can be expressed as functions of these variances, and are, therefore, identified. This same device enables the parameters of model (14) to be identified, since L_t^2 and "a" can be expressed as functions of the covariances of model (14).

Models are fitted via the maximum likelihood in Proc Calis in SAS [8]. The form of the factors in Proc Calis may be obtained by using the "Matrix" and "Cosan" statistics, under the assumption of multivariate normality. The statistic is given in reference [9], and is provided by Proc Calis, which also provides the p-value for the assymptotic chi-square statistic associated with the likelihood function.

8)Example 1:Possible Variance Form, (11) or (14)

For the PAT round 60 asbestos data shown in Figure 1, L=182 and the sample means (square root scale for normality) were: (32.38 , 22.84 , 31.24, 27.47). The estimated correlation matrix was:

$$\begin{vmatrix} 1 & 0.72 & 0.78 & 0.75 \\ 0.72 & 1 & 0.75 & 0.73 \\ 0.78 & 0.75 & 1 & 0.71 \\ 0.75 & 0.73 & 0.71 & 1 \end{vmatrix}$$

The following analyses were performed:

1.) The maximum likelihood estimate for a(t) under model (11) was 0.47, and the 95% confidence interval was (0.22,0.72). $CV=1.27\,[\mu_{ts}]^{-0.53}$. The p-value for model lack of fit was 0.32. In the range of means from 22 to 32, the predicted CVs varied from 0.25 to 0.20 (decreasing with increasing mean), compared to a decrease from 0.24 to 0.20 for the actuals. From the model, the ratio of the between lab CV (decreasing from 0.18 to 0.15) to the measurement error CV (decreasing from 0.065 to 0.05) was $\rho/(1-\rho)$, estimated to be about 3. There appeared to be large lab to lab CV compared to measurement error CV. (Since analyses were carried out on the square root scale, CV values should be multiplied by 2 to convert to original scale CVs.)

2.) The maximum likelihood estimate for a(t) under model (14) was 0.62, and the 95% confidence interval was (0.44,0.80). P-value for the chi square statistic was 0.34. Thus, both models were acceptable.

9)Simulation Study Results for Estimation of Power "a" in Eq (11) - 4 Sample Sets

It is useful to obtain an idea how precisely "a" can be determined by these procedures. For each test, 320 random samples were generated from a quadrinormal distribution with L=500 and S=4 under model (12) with $a=a_1$. For the size of test, the value of "a" tested in (12) was chosen equal to "a_1" in the generated variables. For the power, $a \neq a_1$. Estimation was done via maximum likelihood in Proc Calis, and results are shown in Table I:

Test at 5%	ρ=0.5 m=0.4	ρ=0.5 m=0.4	ρ=0.5 m=1	ρ=0.5 m=0.2	ρ=0.5 m=0.2 True means	ρ=0.2 m=0.4	ρ=0.8 m=0.4
	a_1=1	a_1=0.5	a_1=0.5	a_1=0.5	a_1=0.5	a_1=0.5	a_1=0.5
Size	0.066	0.034	0.066	0.041	0..056	0.044	0.063
	Power:						
$a=a_1$-0.25	0.94	0.94	0.97	0.95	0.97	0.88	1.0
$a=a_1$-0.125	0.40	0.32	0.38	0.38	0.37	0.23	0.70
$a=a_1$+0.25	0.39	0.33	0.42	0.37	0.37	0.28	0.64
$a=a_1$+0.25	0.93	0.95	0.97	0.97	0.95	0.87	1.0

The main conclusion was the dependence of the power on the value of ρ. For ρ equal to 0.8, the probability of a significant result is much higher than for ρ equal to 0.5. For ρ equal to 0.2, the probabilities were somewhat lower than for ρ equal to 0.5. Dependence on the value of a_1 seemed small. There was little difference in results between true means(column 6) or estimated means (column 5), as discussed in 7) above.

10) Example 2: Variance Structure(11) in Question

For PAT Round 83 asbestos data, analysis was done on the square root scale. L=442 and the mean vector was (15.29, 26.67, 29.88, 18.59). The estimated correlation matrix was:

$$\begin{vmatrix} 1 & 0.54 & 0.53 & 0.50 \\ 0.54 & 1 & 0.60 & 0.53 \\ 0.53 & 0.60 & 1 & 0.60 \\ 0.50 & 0.53 & 0.60 & 1 \end{vmatrix}$$

The correlation of sample set 3 with samples 2 and 4 was somewhat higher than the other correlations. Model (11) did not yield acceptable results (p value <0.0001). When the model was refitted with different exponents (model B, eq (14), of section 5) for measurement error and between lab variances, the results were acceptable. The 95% confidence interval for the power "a" for between lab component (estimated as 0.45) was (0.39, 0.51); the 95% confidence interval for the power "a′" for the measurement error component (estimated as 0.32) was (0.21,0.43). (Both confidence intervals were constructed by assuming normality of the estimators.) From (14) the estimated CV was: $[0.74\ \mu^{0.90-2} + 1.90\ \mu^{0.64-2}]^{0.5}$. For the four sample mean values, the actual and predicted CVs are shown in Table II:

Table II* : CV Estimates for Round 83

Sample Mean	Estimated CV	Predicted CV (14)	CV-Between Labs From (14)	CV, Measurement Error From (14)	Ratio: CV(Bet)/ CV(Meas)
15.29	0.25	0.29	0.19	0.22	0.86
18.6	0.23	0.26	0.17	0.19	0.89
26.7	0.21	0.20	0.14	0.15	0.93
29.9	0.20	0.19	0.13	0.14	0.93

2 CV values converts to original scale CVs.

The predicted CVs indicated somewhat bigger decrease in CV than appeared in the actuals, with increasing mean. The predicted CV between labs was slightly less than that for measurement error, but the ratio remained about 0.9.

(11)Alternatives to Model (11) - Continued

C.) More complicated correlation structure . Suppose:

$$\mathrm{Var}(x_{tslk})=[\,L_t^2 + m_t^2\,]\,[\mu_{t,s}]^{2a(t)} \quad (17)$$
$$\mathrm{Cov}(x_{tslk},x_{tvlk})=L_t^2\,[\mu_{ts}]^{a(t)}\,[\mu_{tv}]^{a(t)}$$
$$\mathrm{Cov}(x_{tslk},x_{t+1,vlk})=L_{t,t+1}\,[\mu_{ts}]^{a(t)}\,[\mu_{t+1,v}]^{a(t+1)}.$$

Assume 4 analyses per round, & assume $L_t=L_{t+1}$, $m_t=m_{t+1}$, & $a(t)=a(t+1)$. (These can be tested.)

The correlation matrix is: Matrix $\Lambda\Lambda'$ is multiple of:

$$\begin{vmatrix} A & B \\ B & A \end{vmatrix} \qquad \begin{vmatrix} AA & B \\ B & AA \end{vmatrix} \qquad (18)$$

Here A ,B, and AA are each 4x4 matrices; $A=\{a_{ij}\}$, $a_{ij}=1$, if $i=j$ & $a_{ij}=\rho_1=L_t^2\ /\ [L_t^2 + m_t^2]$ if $i\neq j$; $B=\{b_{ij}\}$, $b_{ij}=\rho_2=L_{t,t+1}\ /\ \{[L_t^2+m_t^2][L_{t+1}^2+m_{t+1}^2]\}^{0.5}$; $AA=\{\rho_1\}$ The covariance matrix corresponding to (17) and (18) requires a modification of equation (11). For $\rho_1\neq\rho_2$, the one-dimensional matrix $\mathbf{1}\mathbf{1}'$ of (11) must be replaced by a sum of two one-dimensional matrices, since the matrix $\Lambda\Lambda'$ is two-dimensional, and has characteristic vectors $\mathbf{1}_{2S}$ and $[\,\mathbf{1}_S'\ \ -\mathbf{1}_S'\,]'$. These vectors can be used in the Matrix statement of Proc Calis.

12)Simulation Study Estimation of "a" - 8 Sample Sets

As in the simpler model, it seems useful to determine how precisely "a" can be estimated.

a.)For this simulation a single value of "a" was used for all eight samples. This is the same correlation form as for the 4 sample case (section 9), but eight samples were generated. b.)Two different correlation structures were used, as in section 11. In the notation of section 11, the larger correlation (a) was in the A submatrix, and the smaller (a_1) in the B submatrix. Estimation was done via maximum likelihood in Proc Calis. For both cases, 320 samples of size 500 were generated.

The probability of statistical significance is given in Table III. Comparison to Table I results indicates that, for case a), the power was better than for the corresponding 4 sample results. For instance, at $a=a_1\pm 0.125$, for eight samples the probability was about 0.48, but for four samples the probability was about 0.32. For case b) in Table III, the probabilities decreased for $a=a_1\pm 0.125$. Estimating both exponents individually reduced the power somewhat.

Table III

Test at 5% Level	a.) Same Corr. Structure; $\rho=0.5$, $m=0.4$, $a_1=0.5$	b.) 2 Diff. Corr. Structures; $\rho=\rho_1=0.5$, $m=0.4$, $a_1=0.5$
Size:	0.05	0.0531
Power:: $a=a_1-0.25$	0.997	0.984
$a=a_1-0.125$	0.482	0.347
$a=a_1+0.125$	0.495	0.372
$a=a_1+0.25$	0.981	0.984

13)Example: More Complicated Correlation

Pat Rounds 59 & 60 asbestos data were transformed to the square root scale. The number of labs, L, present in both rounds was 170. The estimated means were: (29.83, 17.10, 30.84, 21.78, 32.54, 22.90, 31.36, 27.60). The estimated correlation matrix was:

$$\begin{vmatrix} 1 & 0.63 & 0.70 & 0.63 & 0.32 & 0.35 & 0.29 & 0.29 \\ 0.63 & 1 & 0.56 & 0.61 & 0.19 & 0.26 & 0.28 & 0.14 \\ 0.70 & 0.56 & 1 & 0.65 & 0.30 & 0.35 & 0.32 & 0.32 \\ 0.63 & 0.61 & 0.65 & 1 & 0.27 & 0.28 & 0.34 & 0.27 \\ 0.32 & 0.19 & 0.30 & 0.27 & 1 & 0.70 & 0.77 & 0.73 \\ 0.35 & 0.26 & 0.35 & 0.28 & 0.70 & 1 & 0.73 & 0.71 \\ 0.29 & 0.28 & 0.32 & 0.34 & 0.77 & 0.73 & 1 & 0.68 \\ 0.29 & 0.14 & 0.32 & 0.27 & 0.73 & 0.71 & 0.68 & 1 \end{vmatrix}$$

Correlations for the upper diagonal submatrix (round 59) were mostly lower than those for the lower diagonal submatrix (round 60). Model (11) did not yield an acceptable fit ($p<0.05$) when the same correlation structure was used within rounds 59 & 60, or even when different correlation structures were used. None of the models fitted, including models that allowed for different "a" values for each round or different correlation structure within round, yielded acceptable p-values. The discussion of model (8a) considered the possibility that the simple structure of model (8) might not fit the data well. For this example an acceptable p-value(0.34) was obtained when the rank of Λ (in eq. (2)) was increased to three, by retaining the structure of the first two columns of Λ given below (18), but with no restriction on the form of the components of the added dimension. Other forms for the functions $f(\mu_{ts})$ & $g(\mu_{ts})$ of eq (15) need to be tried, to obtain a simple form for a rank two matrix Λ with an acceptable p-value. Simple forms are useful, since they yield simple forms for the CV. Some alternate forms are in [2].

14) Conclusions

For proficiency test data where each lab measures just one sample at each concentration level, and where between lab and measurement error variances are proportional to a power of the mean, the procedures described provide estimates of these variances, and a simple form for the CV. This model was applied to several asbestos data sets from an industrial hygiene proficiency test program, PAT program. Thereby, the usefulness of the method in modeling the CV as a function of the mean has been indicated, although modifications of the simplest form of the model were required. The maximum likelihood procedure used here allows the user to determine adequacy of the fitted model. Failure to obtain acceptable fit of the model can indicate dependence of between lab and measurement error variance components on different powers of the mean, as well as lack of fit of the power of mean model. Some examples of alternative variance-covariance models were provided, although more work needs to be done.

15)Acknowledgments

The author appreciates the helpful comments of NIOSH reviewers Edward Krieg, Jr., Martin Petersen, Paul Schlecht, and Ruiguang Song.

16)References

1)Srivastava, M.S. and Khatri, C.G. **An Introduction to Multivariate Statistics.** North Holland, N.Y.1979, p.292.

2)Song, Ruiguang. "Mixed Model with Inhomogeneous Variance Components." Unpublished manuscript. 1996.

3)Goodman, L.A. and Haberman, S.J. "The Analysis of Nonadditivity in Two-Way Analysis of Variance." **JASA,** 1990, Vol. 85: pp. 139-145

4)Kshirsagar, A.M. "The Goodness-of-Fit of a Single (Non-Isotropic) Hypothetical Principal Component." **Biometrika**,1961, Vol. 48:pp 397-407.

5)Scheffe, H. **The Analysis of Variance.** Wiley, NY, 1959, pp.261-269.

6)Leidel, N.A., Busch, K.A., & Lynch, J.R. **Occupational Exposure Sampling Strategy Manual.** NIOSH. Pub. No. 77-173, 1977.

7)Bollen, K. **Structural Equations with Latent Variables.** Wiley, NY,1989, p88.

8)**SAS/STAT User's Guide,** Version 8. SAS Institute, Inc. Cary, NC. 1999, p.435.

9)Anderson, T. W. **An Introduction to Multivariate Statistical Analysis.** Wiley: N.Y. 1958, p. 265.