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EFFECTS OF MEASUREMENT ERRORS ON LINEAR CALIBRATION EQUATIONS

SYLVESTER C. SUDA AND DONALD F. SHEPARD

July 1976

TECHNICAL SUPPORT ORGANIZATION
DEPARTMENT OF APPLIED SCIENCE

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ABSTRACT

This report summarizes the results of the developmental work in the area of statistical treatment of calibration data performed in the preparation of American National Standard N15.19. The effects of measurement errors in both the dependent and independent variables in linear calibrations are studied by examining (1) a simulation model in which the lack of fit was zero with assigned known values of the measurement errors, (2) data from a laboratory calibration of a process vessel where the measurement errors are small compared with the lack of fit, (3) data from a process vessel calibration where the lack of fit can be removed through the use of a cubic equation, and (4) data from a process vessel calibration where the major error appears to be in the dependent variable. The effect of measurement errors on the estimation of the standard deviation of the slope is found to be highly dependent on the experimental design used and on the subsequent treatment of the data.

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Effects of Measurement Errors on Linear Calibration Equations

I. INTRODUCTION

The choice of an appropriate linear calibration model depends on a priori information regarding the nature of the measurement errors in the independent and dependent variables. However, the a priori information is not always available at the onset of the calibration experiment, or the experimenter may have difficulty interpreting the information in terms of valid statistical analysis of the data. For example, in the analysis of tank calibration data it is usually assumed that the independent variable involved in the measurements based on standards or provers has negligible error. This is often the case, but by the nature of the experiment the determinations based on the provers are subject to cumulative fluctuations, and the possibility that these fluctuations may overshadow the independent errors in the dependent variable must be kept in mind.

Let x_i represent measurements of liquid added to a tank and y_i measurements of the height of liquid level. The fundamental relationship between the measured increments and the measured level is assumed to be

$$y = a + bX \quad (1)$$

where

y = measured level,

a = y intercept,

b = slope, and

$X = \sum x_i$, the sum of the measured increments.

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In the calibration of a tank both x_i and y_i are subject to measurement error. The tank is calibrated by adding measured quantities of liquid to the tank and measuring the liquid level. The x variable may be considered to be controlled since the quantities of liquid are set at assigned values. For measurements of special nuclear materials the x_i determinations must be based on precision provers whose calibrations are traceable to the National Bureau of Standards, and the x_i measurements are therefore generally very accurate. However, the errors of the measured x_i values, are cumulative. Errors in y_i are a function of the sensing and response instruments. Typically, the larger error is in the y_i measurement.

Jaech¹ considers two cases of fitting straight lines to tank calibration data: the cumulative error model and the classical or independent error model. In the cumulative model the error of a given point, $X = \sum x_i$, includes the errors associated with all previous points and the dependent variable y is assumed to be measured without error. The classical model assumes that the independent variable x is measured without error. The appropriate model is the one whose underlying assumptions are more closely satisfied by what is known about the errors affecting the two variables.

The important difference between the two models is in the estimate of the standard error of the slope. Mandel^{2a} notes that the variance estimate of the slope given by using the independent method where the cumulative model is appropriate results in a serious underestimation of the variance of the slope. This is a case where the smaller slope variance is not necessarily the "better" estimate. Mandel^{2b} further states that the lack of randomness in the residuals indicates the presence of cumulative error in the data. On the basis of analy-

sis of simulated data with cumulative error, the reliability of this randomness test for diagnosing the presence of cumulative error is questioned in this report.

The choice of model for fitting a straight line to tank calibration data should be based on analysis of which source of error is dominant. If the error in x_i is dominant, the cumulative model is the appropriate one. If the x_i are precise results or if the errors in the determination of x_i are considerably smaller than in the determination of y_i , the classical model applies.

The equation expressing errors associated with the calibration is

$$y + e_y = a + b(x + e_x) + e_t \quad (2)$$

where a and b are the intercept and slope, respectively, and

x = "true" volume (mass),

y = "true" liquid level,

e_x = random error associated with the increment measurements,

e_y = random error associated with the liquid level measurements, and

e_t = fixed error due to tank imperfections or instrument

nonlinearity (exhibited as a lack of fit of the
calibration equation).

In this report, the effects of the errors on the variance of the slope are studied by using (1) simulated data and (2) experimental data from several sources.

The increment measurements can be obtained using either gravimetric or volumetric provers. In the examples that follow volumetric provers are assumed.

II. SIMULATION MODEL

The effects of the error terms denoted by e_x and e_y in Eq. (2) on the standard error of the slope were studied by setting $e_t = 0$ and using Monte Carlo techniques with the following simulation model:

$$y_i = Y_i + e_y = -1.28 + 0.16X_i + e_x \quad (3)^*$$

where

$x_i = 100$ liters,

$Y_i =$ calculated liquid level in centimeters,

$-1.28 =$ intercept,

$0.16 =$ slope,

$e_x = N(0,1)\sigma_x$, and

$e_y = N(0,1)\sigma_y$.

The $N(0,1)$ are random normal deviates with mean zero and variance 1. Observations of e_x and e_y were generated by multiplying values of σ_x and σ_y , respectively, by randomly drawn normal deviates $N(0,1)$.

Synthetic data for the measured volume, $x + e_x$, are calculated by adding values of e_x to each x increment of 100 liters. For each value $X_i = x_i + e_x$, the value $Y_i = -1.28 + 0.16X_i$ is calculated. Values for e_y are calculated by using σ_y and an observation from $N(1,0)$. Values for the measured level response are $y_i = Y_i + e_y$. The calibration experiment is simulated by carrying out the

*An example of a process tank for which the relationship between the volume and the liquid-level height has a slope of 0.16 and a y-intercept of -1.28 is a vertical right circular cylindrical tank with a radius of about 1.4 meters and an instrument heel of 8 liters.

increment addition n times. The resulting n pairs of X, y data points represent a calibration run.

Multiple realizations of the calibration experiment with a given set of σ_x and σ_y values are observed by repeating the process with a different set of $N(0,1)$ deviates.

By varying the values of σ_x and σ_y and using the same series of $N(0,1)$ deviates, changes independent of the random effects in the standard error of the slope are observed.

A. Example 1 - Simulated Data

By using a Wang 700/702 computer and random normal deviates taken from Natrella,³ Monte Carlo experiments were done to simulate 304 calibration runs. The 304 runs consist of 19 sets of 16 runs; each run consists of 16 incremental additions. The same selection of 512 $N(0,1)$ deviates was used for each of the 19 sets. The experimental design is shown in Figure 1, and the values of σ_x and σ_y are given in Table 1.

Table 1				
Scheme for Numbering the Sets				
σ_y , cm	σ_x , liters			
	0.16	0.08	0.04	0.0
0.00	1	2	3	-
0.02	4	6	9	19
0.04	5	8	12	18
0.08	7	11	14	17
0.16	10	13	15	16

Figure 1. Design of simulated calibration experiment.

Sixteen 100-liter increments were used to generate a calibration run. Each increment of 100 liters (observed x value) was adjusted to its simulated value by adding a correction of e_x obtained by multiplying a $N(0,1)$ deviate by the appropriate value of σ_x . By using Eq. (3) the "simulated" value of Y in centimeters was calculated and adjusted by e_y to its observed value of y, with e_y obtained in the same manner as e_x . The resulting 16 pairs of X, y data points were fitted by using the independent model.

B. Analysis of Simulated Data

The data were analyzed by using the F test of the ratios of percent error of the slope, by analysis of covariance, and by use of the normalized mean-square successive difference (u) test.

The percent error P_i of each observed slope was calculated:

$$P_i = 100(b_i - 0.16)/0.16 . \quad (4)$$

The observed standard deviation of the percent error of the slope for a set of 16 runs is

$$\text{obs \% S.D.} = [\sum (P_i - \bar{P})^2 / 15]^{1/2} . \quad (5)$$

The calculated standard deviation of the percent error of the slope for a set was obtained by averaging the 16 values of the variance of the slope and multiplying this result by 100:

$$\text{calc \% S.D.} = (100/0.16) [\sum \hat{V}(b_i) / 16]^{1/2} . \quad (6)$$

Ratios of the observed to calculated standard deviation of the percent error in the slope for the 19 sets are presented in part 1 of Table 2. The obs/calc variance ratios are F distributed with 15 and 224 degrees of freedom. The critical value for $F[\alpha = 0.05(15,224)]$ is 1.67.

The 16 calibration runs per set were grouped in the order they were generated into 4 quartets of 4 runs each, and each quartet was examined by the analysis of covariance described by Brownlee^{4a} and in Section VI of this report. Ratios of the mean squares for the variance between the individual slopes, S_2^2 , and for the deviation of the group means, S_3^2 , to the mean square about the individual lines S_1^2 , were calculated. The ratios, S_2^2/S_1^2 , are F distributed with 3 and 15 degrees of freedom. At a significance level of $\alpha = 0.05$, the number of quartets per set in which the hypothesis of consistency between the four runs was rejected was noted. The results are summarized in part 2 of Table 2.

The third test of the data was the normalized mean-square successive difference (u) test, the basis for which is explained in Section VII.

The statistic u is approximately $N(0,1)$ distributed for n as small as 10. The regions of interest are as follows:

- u > 2, which indicates that the y variate is not normally distributed (e.g., because of rounding or transcription error);
- 2 < u < 2, the region of nonrejection; and
- u < -2, which indicates that the dominant error is in the x variate (volume measurement) since $e_t = 0$.

The numbers of runs per set rejected on the basis of the above criteria are presented in part 3 of Table 2.

Table 2

Summary of Simulated Calibration

Set	σ_x	σ_y	Part 1 Percent of error of the slope		Variance ratio	Part 2 Analysis of covariance		Part 3 u	
			Percent std. dev.			S_2	S_3	<-2	>2
			Obs	Calc					
1	0.16	0.00	0.029431	0.008856	11.05*	4/4	4/4	8/16	0/16
2	0.08	0.00	0.014715	0.004428	11.05*	4/4	4/4	8/16	0/16
3	0.04	0.00	0.007358	0.002213	11.05*	4/4	4/4	8/16	0/16
4	0.16	0.02	0.030641	0.011566	7.02*	4/4	4/4	3/16	0/16
5	0.16	0.04	0.033171	0.017046	3.79*	3/4	4/4	2/16	0/16
6	0.08	0.02	0.016585	0.008522	3.79*	3/4	4/4	2/16	0/16
7	0.16	0.08	0.041101	0.030154	1.86*	2/4	2/4	0/16	0/16
8	0.08	0.04	0.020550	0.015078	1.86*	2/4	2/4	0/16	0/16
9	0.04	0.02	0.010270	0.007537	1.86*	2/4	2/4	0/16	0/16
10	0.16	0.16	0.062705	0.058027	1.17	0/4	1/4	0/16	0/16
11	0.08	0.08	0.031351	0.029008	1.17	0/4	1/4	0/16	0/16
12	0.04	0.04	0.015675	0.014506	1.17	0/4	1/4	0/16	0/16
13	0.08	0.16	0.056293	0.057368	0.96	0/4	0/4	0/16	0/16
14	0.04	0.08	0.028147	0.028684	0.96	0/4	0/4	0/16	0/16
15	0.04	0.16	0.054312	0.057166	0.90	0/4	0/4	0/16	0/16
16	0.00	0.16	0.053283	0.057048	0.87	0/4	0/4	0/16	0/16
17	0.00	0.08	0.026642	0.028424	0.87	0/4	0/4	0/16	0/16
18	0.00	0.04	0.013321	0.014261	0.87	0/4	0/4	0/16	0/16
19	0.00	0.02	0.006660	0.007129	0.87	0/4	0/4	0/16	0/16

*F[$\alpha = 0.05(15, 224)$] = 1.67.

C. Results of Analysis

This example illustrates the effects of cumulative error on the estimated variance of the slope. Table 2 shows that three tests for sets 1 through 6 are significant at the 5% level. In this example, the variance ratio test of percent error of the slope and the analysis of covariance are based on 14 and 224 degrees of freedom and is a more sensitive indicator of cumulative error in the independent variable than is the t test, which is the test applied to a single run having 14 degrees of freedom.

To the practitioner, Table 2 indicates that the use of precision provers in the calibration of tanks would permit use of the independent error model without resulting in serious underestimation of the variance of the slope. Typically the standard deviation, σ_x , of a 100-liter prover is in the range of 0.0075 liters. The standard deviation, σ_y , of the liquid-level sensor and response system is 0.02 centimeters.

The effect of σ_x and σ_y on the observed and calculated percent variances of the slope appear to be additive. From the data in part 1 of Table 2 the standard deviation for $\sigma_x = 0.16$, $\sigma_y = 0.16$ (set 10) can be estimated by adding the variance of sets 1 and 16. Since in most tank calibrations the value of σ_x is well known from calibration of the prover, the underestimation of the slope variance by the independent error model can be estimated.

The estimation procedure involves use of the calculated standard deviation of the slope obtained by the independent model and the σ_x value from calibration of the prover. For example, for $\sigma_x = 0.16$ and $\sigma_y = 0.04$, the calculated S.D.(b) is 0.017046. By using data from part 1 of Table 2, a reasonable estimate of the observed S.D.(b) = 0.033171 can be determined:

$$[(0.017046)^2 + (0.008856)^2 + (0.079431)^2]^{1/2} = 0.032838.$$

The above computations and evaluations are instructive and are suggested practice for the preliminary analysis of tank calibration data.

111. EXAMPLE 2 - PRESSURE GAUGE DATA

This example, from data supplied by Schooner and Houser,⁵ illustrates the case in which the measurement errors e_x and e_y in Eq. (2) are small compared with e_t , which is the lack of fit due to nonuniformity of the tank diameter or nonlinearity of the device used to measure the y variate.

The experiment was done with a right circular cylindrical tank and a Ruska XR-38 pressure counter. The calibration increments were measured with 30-gallon and 100-gallon provers. Data from the first 6 runs, made under wet-wall conditions, were fitted with straight lines by the independent error model. Data for the 6 runs along with the predicted values of y and residual y are listed in Table 3.

Also listed in Table 3 are values of R_n , the statistic used by Tietjen et al.⁶ to detect a single outlier; for a sample size of 9 and $\alpha = 0.05$ the critical value is 2.28. The values of predicted and residual x are useful for checking for data recording errors and for developing some idea of the magnitude of the error associated with the use of Eq. (1).

Graphical or statistical analysis of the residuals is indispensable to any situation--linear, polynomial, nonlinear, or analysis of variance--where a model is fitted. The examination of residuals is a necessary first step in testing the validity of the underlying least-squares model and is an important

Table 3

Linear least-Squares Analysis - Example 2

Pt. No.	Obs x	Obs y	Pred. y	Resid. y	Cusum resid.	R _n	Pred. x	Resid. x
Run 1								
1	113.534	2.531	2.600	.069	.069	1.558	112.751	-.782
2	492.136	36.062	36.008	-.053	.015	1.100	492.738	.602
3	870.744	69.387	69.418	.031	.047	.610	870.391	-.352
4	1249.298	102.840	102.822	-.017	.029	.331	1249.495	.197
5	1627.960	136.257	136.236	-.020	.009	.384	1628.190	.230
6	2006.570	169.662	169.646	-.015	-.006	.303	2006.750	.180
7	2385.145	203.134	203.052	-.081	-.088	1.600	2386.069	.924
8	2763.714	236.446	236.458	.012	-.075	.253	2763.575	-.138
9	3142.293	269.789	269.864	.075	.000	1.713	3141.432	-.860
Run 2								
1	113.601	2.540	2.614	.074	.074	1.601	112.756	-.844
2	492.144	36.070	36.016	-.053	.021	1.049	492.746	.602
3	870.714	69.390	69.421	.031	.052	.590	870.356	-.357
4	1249.289	102.848	102.826	-.021	.031	.385	1249.529	.240
5	1627.871	136.258	136.232	-.025	.006	.457	1628.159	.288
6	2006.624	169.671	169.653	-.017	-.011	.319	2006.823	.199
7	2385.277	203.147	203.065	-.081	-.092	1.524	2386.200	.923
8	2763.702	236.447	236.457	.010	-.082	.206	2763.583	-.118
9	3142.218	269.775	269.857	.082	.000	1.772	3141.284	-.933
Run 3								
1	113.592	2.537	2.607	.070	.070	1.569	112.792	-.799
2	492.217	36.072	36.018	-.053	.017	1.093	492.823	.606
3	870.771	69.393	69.423	.030	.047	.584	870.429	-.341
4	1249.325	102.846	102.827	-.018	.029	.341	1249.530	.205
5	1627.933	136.258	136.237	-.020	.008	.385	1628.167	.234
6	2007.418	169.650	169.635	-.014	-.005	.265	2006.577	.159
7	2384.969	203.123	203.040	-.082	-.088	1.601	2385.906	.937
8	2763.554	236.437	236.447	.010	-.077	.219	2763.432	-.121
9	3142.055	269.770	269.847	.077	.000	1.730	3141.173	-.881
Run 4								
1	113.553	2.534	2.609	.075	.075	1.638	112.702	-.850
2	492.086	36.065	36.009	-.055	.019	1.109	492.713	.627
3	870.689	69.388	69.416	.028	.047	.537	870.368	-.320
4	1249.254	102.838	102.819	-.018	.029	.338	1249.461	.207
5	1627.794	136.243	136.220	-.022	.007	.405	1628.045	.251
6	2006.368	169.642	169.624	-.017	-.009	.314	2006.561	.193
7	2384.997	203.115	203.033	-.081	-.090	1.538	2385.915	.918
8	2763.698	236.438	236.449	.011	-.079	.226	2763.569	-.128
9	3142.226	269.770	269.849	.079	.000	1.732	3141.326	-.899

Table 3 (cont'd)

Pt. No.	Obs x	Obs y	Pred. y	Resid. y	Cusum resid.	R _n	Pred. x	Resid. x
Run 5								
1	113.594	2.535	2.605	.070	.070	1.647	112.794	-.799
2	492.222	36.066	36.015	-.052	.018	1.119	492.814	.592
3	870.806	69.396	69.422	.026	.044	.528	870.511	-.292
4	1249.172	102.826	102.809	-.016	.027	.333	1249.363	.191
5	1627.667	136.227	136.207	-.019	.007	.380	1627.887	.220
6	2006.167	169.624	169.606	-.017	-.009	.347	2006.366	.199
7	2384.500	203.067	202.990	-.076	-.086	1.552	2385.366	.866
8	2763.548	236.424	236.437	.013	-.072	.295	2763.391	-.156
9	3142.045	269.764	269.836	.072	.000	1.690	3141.224	-.820
Run 6								
1	113.616	2.541	2.617	.076	.076	1.663	112.745	-.870
2	492.137	36.070	36.018	-.051	.025	1.023	492.721	.584
3	870.670	69.395	69.420	.025	.050	.473	870.385	-.284
4	1249.170	102.839	102.818	-.020	.030	.367	1249.397	.227
5	1627.721	136.247	136.222	-.024	.005	.449	1628.002	.281
6	2006.198	169.637	169.618	-.018	-.012	.330	2006.403	.205
7	2384.765	203.105	203.023	-.081	-.094	1.533	2385.687	.922
8	2763.309	236.414	236.426	.012	-.081	.242	2763.170	-.138
9	3141.909	269.752	269.833	.081	.000	1.770	3140.981	-.927

supplement to other diagnostic techniques in identifying causes of abnormalities in data.

The residuals for the 6 calibration runs were plotted against the volume values (Figure 2), and the plots were examined for trends and scatter. Two things are evident: (a) individual observations are closely grouped about 9 calibration points, and (b) the calibration points display erratic swings that are large compared with the measurement error. This indicates the presence of error associated with e_t defined in Eq. (2).

The estimates of the coefficients and the standard errors of the coefficients for the 6 runs are shown in Table 4. The estimates for the overall least-squares line are given in the line labeled "Sum." The u test statistic is defined in Section VIII.

In the analysis of covariance given in Table 5, the F ratios are all abnormally low. The F ratio of the mean square due to the differences of the individual slopes compared with the mean square of the points about the individual lines is significantly low at the $\alpha = 0.001$ level.

The plot of the residuals and the analysis of covariance suggest that, instead of six independent estimates of the regression line, the data consist of six replicate observations of y_{ji} at each of the nine $\bar{X}_i$ values. One method of treating such data is to adjust values of the dependent variable to take account of differences among the associated values of the independent variable. To eliminate the small effect of the observed differences in the X_{ji} values, the y_{ji} values in each i cluster were adjusted to the same $\bar{X}_i$ value by the method described in Section VII.

The $\bar{X}$ values and the adjusted y values (denoted by y') are listed in Table 6.

Figure 2. Plot of residuals - Example 2.

Table 4

Estimates of Coefficients and Standard Errors - Example 2

k	a	b	Std. dev. y resid.	Std. dev. a	Std. dev. b	u
1	-7.41842171	.08824237	.05620650	.03639405	.000019166	-.1924
2	-7.40958362	.08823926	.05894573	.03816810	.000020100	-.3050
3	-7.41615726	.08824285	.05695022	.03687755	.000019421	-.1931
4	-7.41048219	.08823676	.05809622	.03761699	.000019810	-.2567
5	-7.41799587	.08824010	.05429956	.03516053	.000018518	-.2485
6	-7.40760725	.08823979	.05857229	.03792713	.000019976	-.4000
Sum	-7.41337433	.08824019	.05156269	.01363050	.000007178	-.6461
$\bar{x}, \bar{y}$	-7.41337507	.08824019	.05714730	.03700392	.000019288	-.2672

Table 5

Analysis of Covariance

Source of variance	Sum of squares	Degrees of freedom	Mean square	F ratio
Between $\hat{b}$ and $\bar{b}$	.000000889	1	.000000889	.00027
Deviations of group means about their least-squares line	.000593510	4	.000148377	.04534
Between individual slopes	.000226000	5	.000045200	.01381
Deviations of individual points about their least-squares lines	.137433000	42	.003272214	
About over all line	.138254000	52	.002658730	
Due to overall line	401717.707846000	1	401717.707846000	

Table 6							
One-Way Analysis of Variance							
k	$\bar{X}$	Adjusted y' values					
		Run 1	Run 2	Run 3	Run 4	Run 5	Run 6
1	113.58166	2.5352	2.5382	2.5360	2.5365	2.5339	2.5379
2	492.15790	36.0638	36.0711	36.0667	36.0712	36.0622	36.0717
3	870.73233	69.3859	69.3916	69.3895	69.3918	69.3894	69.4005
4	1249.25133	102.8358	102.8446	102.8394	102.8377	102.8330	102.8461
5	1627.82433	136.2450	136.2538	136.2484	136.2456	136.2408	136.2561
6	2006.39083	169.6461	169.6504	169.6476	169.6440	169.6437	169.6540
7	2384.94216	203.1161	203.1174	203.1206	203.1101	203.1060	203.1206
8	2763.58750	236.4348	236.4368	236.4399	236.4282	236.4274	236.4385
9	3142.12433	269.7741	269.7667	269.7761	269.7610	269.7710	269.7710

Source	Sum of squares	Degrees of freedom	Mean square	F ratio
Slope	401717.700748000	1	401717.700748000	
Means	.137182000	7	.019597428	824.1909
Error	.001070000	45	.000023777	

The adjusted y'_{ji} values were then fitted to the $\bar{X}_i$ values by the method described by Brownlee^{4b} for linear regression with several observations on y at x_i . The residuals are plotted in Figure 3. The means squares are summarized in Table 7. The F ratio of the mean squares of the group means about their line to the mean square of the replicate y'_{ji} measurements is significant at the $\alpha = 0.001$ level; that is, there is a significant lack of fit of the group means to a straight line. The estimates of the coefficients of the linear least-squares fit based on the nine $\bar{X}, \bar{y}'$ points are given in the last line of Table 4. The preceding line gives the estimates of the coefficients of the linear least-squares fit based on the unadjusted 54 X, y pairs.

Figure 3. Plot of residuals about the $\bar{X}, \bar{Y}$ least-squares line.

Table 7		
Comparison of Methods of Analysis and Standard Deviation of the Slope		
Method used	Estimate of % standard deviation of slope	Degrees of freedom
Observed 6 calibrations	0.001026	5
Sum of all 54 points	0.008134	52
Nine $\bar{x}, \bar{y}'$ points	0.022085	7

Attempts to improve this linear fit by use of a quadratic or cubic polynomial fit were unsuccessful. Since the measurements involve just one pressure instrument and one tank, there is no basis, without additional information, for assigning the lack of fit either to nonlinearity of the instrument response or to imperfections in the tank. Subsequent data have shown that the major source of error is irregularity in the cross sectional area of the tank.

Since the investigation of higher-degree polynomials did not produce positive results, it is concluded that the "best" equation is the linear equation based on the nine $\bar{x}, \bar{y}'$ points. Before proceeding further the analyst should make sure that the standard deviation of the slope is not underestimated. A comparison of this value with the spread of the plotted residuals is useful. In comparing Figure 3 with Figure 2 it is seen that the adjustment of the y_{ji} values to $\bar{y}_i$ has increased the vertical spread of the residuals: this is a typical outcome.

A comparison of the estimates of the standard deviation of the slope due to the method of combining the 54 x_{ji}, y_{ji} observations is given in Table 7.

IV. EXAMPLE 3 - PROCESS TANK DATA I

This example, from data supplied by Wilson,⁷ illustrates the case in which (as in the preceding example) the replications of the calibration runs essentially comprise repeated measurements of a single run, and the major source of error is due to changes in the cross sectional area of the tank. In this example, however, e_t can be eliminated by use of a polynomial fit. With the effect of the diameter changes in the tank removed, a significant difference between the runs was apparent.

The data are for three repeated calibration runs of 10 increments of 20 liters each with the first increment of each run offset by 0.5 liter. In this analysis the data are coded by dividing each x datum by 100. This linear transformation has no effect on linearity of regression or on the analysis of variance and does not alter the estimate of the y intercept or the shape of the plot of the y residuals.

The results of fitting the calibration data by the independent linear model are shown in Tables 8 and 9, and the residuals from the combined runs are plotted in Figure 4. The value of u was < -2 in all three runs. The plot shows that the residuals are not randomly distributed but fall on a smooth curve resembling a parabola. In Table 10 the F ratio of the mean square due to the difference of the individual slopes compared with the mean square of the points about the individual lines is significantly low at the $\alpha = 0.01$ level. All these results indicate the presence of e_t as the major source of error. The pattern of the residuals in Figure 4 suggests that a polynomial fit may be appropriate.

One good feature of the calibration is that all increments, for all three runs, are of equal size, and therefore the data permit the use of orthogonal polynomial techniques.^{3b} Linear, quadratic, and cubic lines were fitted to

Table 8

Linear Least-Squares Analysis - Example 3

Pt. No.	Obs x	Obs y	Pred. y	Resid. y	Cusum resid.	R _n	Pred. x	Resid. x
Run 1								
1	.280	1.300	1.602	.302	.302	2.195	.144	-.135
2	.480	2.000	2.049	.049	.352	.332	.457	-.022
3	.680	2.580	2.495	-.084	.267	.546	.717	.037
4	.880	3.100	2.941	-.158	.109	.993	.950	.070
5	1.080	3.550	3.387	-.162	-.053	1.003	1.152	.072
6	1.280	3.980	3.834	-.145	-.198	.903	1.345	.065
7	1.480	4.350	4.280	-.069	-.268	.437	1.511	.031
8	1.680	4.750	4.726	-.023	-.291	.151	1.690	.010
9	1.880	5.100	5.172	.072	-.219	.492	1.847	-.032
10	2.080	5.400	5.619	.219	.000	1.587	1.981	-.098
Run 2								
1	.285	1.400	1.685	.285	.285	2.215	.157	-.127
2	.485	2.120	2.131	.011	.296	.081	.479	-.005
3	.685	2.600	2.577	-.022	.273	.158	.695	.010
4	.885	3.190	3.023	-.166	.106	1.122	.959	.074
5	1.085	3.630	3.469	-.160	-.054	1.067	1.157	.072
6	1.285	4.040	3.914	-.125	-.179	.829	1.341	.056
7	1.485	4.440	4.360	-.079	-.258	.531	1.520	.035
8	1.685	4.820	4.806	-.013	-.271	.091	1.690	.005
9	1.885	5.180	5.252	.072	-.198	.527	1.852	-.032
10	2.085	5.500	5.698	.198	.000	1.543	1.995	-.089
Run 3								
1	.290	1.400	1.697	.297	.297	2.221	.155	-.134
2	.490	2.090	2.141	.051	.348	.357	.466	-.023
3	.690	2.680	2.584	-.095	.253	.632	.732	.042
4	.890	3.180	3.028	-.151	.102	.979	.958	.068
5	1.090	3.640	3.472	-.167	-.065	1.070	1.165	.075
6	1.290	4.040	3.915	-.124	-.189	.791	1.345	.055
7	1.490	4.440	4.359	-.080	-.270	.520	1.526	.036
8	1.690	4.810	4.803	-.006	-.277	.045	1.693	.003
9	1.890	5.170	5.246	.076	-.200	.534	1.855	-.034
10	2.090	5.490	5.690	.200	.000	1.495	1.999	-.090

Table 9

Estimates of Coefficients and Standard Error - Example 3

k	a	b	Std. dev. y resid.	Std. dev. a	Std. dev. b	u
1	.97816969	2.23121212	.17054147	.12320932	.093879991	-2.4608
2	1.04980909	2.22969696	.15915782	.11537819	.087613497	-2.3125
3	1.05436363	2.21818181	.16558573	.12044825	.091151948	-2.4784
Sum	1.02706227	2.22667037	.15627978	.06540777	.049667720	-4.0184
$\bar{x}, \bar{y}'$	1.02742575	2.22636363	.16450967	.11925791	.090559597	-2.4507

Table 10

Analysis of Covariance

Source of variance	Sum of squares	Degrees of freedom	Mean square	F ratio
Between $\hat{b}$ and $\bar{b}$	.018443597	1	.018443597	.67613
Deviations of group means about their least-squares line	.010401667	1	.010401667	.38131
Between individual slopes	.000335151	2	.000167575	.00614
Deviations of individual points about their least-squares lines	.654673939	24	.027278080	
About overall line	.683854355	28	.024423369	
Due to overall line	49.087282311	1	49.087282311

Figure 4. Plot of residuals for overall linear line - Example 3.

the data by the method of orthogonal polynomials. These results are presented in Table 11.

Inspection of the residual y values shows the improvement of the cubic fit over the quadratic and linear fits. Other results are given in Table 12. The interpretation of the significant F tests is that, given the null hypothesis of no factor effect, the linear, quadratic and cubic coefficients are significant except for the cubic coefficient for run 2. The critical value for $F[\alpha=0.005(1,6)]$ is 18.635, and y residuals for the cubic fit in run 2 of Table 12 are only marginally better than that for the quadratic fit. The interpretation of the F test in run 2 is that the improvement of the cubic over the quadratic fit is not significant. In the overall line the critical F value for $F[\alpha=0.005(1,26)]$ is 9.41 and the cubic coefficient is highly significant.

The data from the three calibration runs were pooled and the cubic equation was fitted to the data. The plot of the residuals on the overall cubic fit are shown in Figure 5. The difference between the first run and the other two runs may be due to the tank being dry for run 1 and wet for runs 2 and 3, or it may be due to a zero offset on the manometer in run 1. The shapes of the three curves are in general agreement and provide a profile of the cross sectional area of the tank. The peak and valley formed by the second and third points of run 2 are self-canceling and are probably due to a data handling error or to misreading of the manometer. Unlike errors associated with the measurement of the increments, a misreading of the manometer is a recoverable error.

The residual data for the cubic fit was next examined by using the two-way crossed classification model for the analysis of variance. The results are presented in Table 13. The hypotheses that there are no run and point effects were both rejected. The run effect was not detected in the analysis of covari-

Table 11

Polynomial Least-Squares Analysis

Pt. No.	x	y	Predicted y			Residual y		
			Cubic	Quadratic	Linear	Cubic	Quadratic	Linear
Run 1								
1	.280	1.300	1.313	1.357	1.602	.013	.057	.302
2	.480	2.000	1.981	1.967	2.049	-.018	-.032	.049
3	.680	2.580	2.572	2.536	2.995	-.007	-.043	-.084
4	.880	3.100	3.096	3.064	2.941	-.003	-.035	-.158
5	1.080	3.550	3.564	3.551	3.387	.014	.001	-.162
6	1.280	3.980	3.985	3.997	3.834	.005	.017	-.145
7	1.480	4.350	4.370	4.403	4.280	.020	.053	-.069
8	1.680	4.750	4.731	4.767	4.726	-.018	.017	-.023
9	1.880	5.100	5.076	5.091	5.172	-.023	-.008	.072
10	2.080	5.400	5.417	5.373	5.619	.017	-.026	.219
Run 2								
1	.285	1.400	1.419	1.458	1.685	.019	.058	.285
2	.485	2.120	2.068	2.055	2.131	-.051	-.064	.011
3	.685	2.600	2.647	2.614	2.577	.047	.014	-.022
4	.885	3.190	3.165	3.136	3.023	-.024	-.053	-.166
5	1.085	3.630	3.631	3.620	3.469	.001	-.009	-.160
6	1.285	4.040	4.055	4.066	3.914	.015	.026	-.125
7	1.485	4.440	4.445	4.474	4.360	.005	.034	-.079
8	1.685	4.820	4.812	4.844	4.806	-.007	.024	-.013
9	1.885	5.180	5.164	5.177	5.252	-.015	-.002	.072
10	2.085	5.500	5.511	5.471	5.698	.011	-.028	.198
Run 3								
1	.290	1.400	1.410	1.460	1.697	.010	.060	.297
2	.490	2.090	2.079	2.062	2.141	-.010	-.027	.051
3	.690	2.680	2.666	2.624	2.584	-.013	-.053	-.095
4	.890	3.180	3.183	3.146	3.028	.003	-.033	-.151
5	1.090	3.640	3.644	3.630	3.472	.004	-.009	-.167
6	1.290	4.040	4.059	4.073	3.915	.019	.033	-.124
7	1.490	4.440	4.440	4.477	4.359	.000	.037	-.080
8	1.690	4.810	4.800	4.842	4.803	-.009	.032	-.006
9	1.890	5.170	5.151	5.167	5.246	-.018	-.002	.076
10	2.090	5.490	5.503	5.453	5.690	.013	-.036	.200

Table 12

Selected Results of Polynomial Analysis

Source of variance	Degrees of freedom	F ratio	Std. dev. about line	F ratio	Std. dev. about line	F ratio	Std. dev. about line
		Run 1		Run 2		Run 3	
Mean	1/9	70.4359	1.360599	73.8632	1.358461	74.6277	1.352217
Linear	1/8	564.8532	0.170541	647.6638	0.159157	592.1917	0.165585
Quadratic	1/7	131.4257	0.040998	94.2339	0.044741	104.9783	0.044258
Cubic	1/6	22.8040	0.020211	6.7930	0.033095	50.0842	0.015636
Overall Line							
Mean	1/29	234.8808	1.310055				
Linear	1/28	2009.8488	0.156279				
Quadratic	1/27	240.4441	0.050566				
Cubic	1/26	19.1939	0.039084				

Estimate of Coefficients for the Overall Line

	a	b ₁	b ₂	b ₃
Cubic	0.2826	4.1999	-1.2826	0.2223
Quadratic	0.4981	3.3938	-0.4925	
Linear	1.0271	2.2267

Figure 5. Plot of residuals for overall cubic line - Example 3.

Table 13

Two-Crossed Classification ANOVA of Cubic Residuals

Pt. No.	Run 1	Run 2	Run 3	Average
1	.0636	-.0187	-.0011	.0146
2	.0276	-.0767	-.0312	-.0267
3	.0350	.0288	-.0373	.0088
4	.0364	-.0413	-.0190	-.0079
5	.0522	-.0167	-.0157	.0066
6	.0431	-.0067	.0032	.0132
7	.0597	-.0209	-.0116	.0090
8	.0224	-.0387	-.0198	-.0120
9	.0218	-.0494	-.0307	-.0194
10	.0686	-.0226	-.0038	.0140
Average	.0430	-.0262	-.0167	.0000

Source	Sum of squares	Degrees of freedom	Mean square	F ratio
Between points	.006274315	6	.001045719	3.6683
Between runs	.028224948	2	.014112474	49.5056
P x R interaction	.005131218	18	.000285067	

ance of the linear least-squares fit (Table 10). The reason for this is that in the analysis of covariance the mean square of the deviations of the individual points about their least-squares lines includes the lack of fit to the linear line which may result in an overestimation of the mean square. Consequently all F ratios based on this mean square will be too low (see Section VI). The interaction mean square of Table 13 is the best estimate of the deviation of the individual points about their cubic line and thus of the measurement variance, s_y^2 .

An examination of the plot of the residuals from the overall cubic fit (Fig. 5) clearly shows that the difference between runs is due to a change in intercept and not due to variations in slope. The best estimate of the variance of the overall mean ($\bar{X}, \bar{y}$ point) is obtained by dividing the between run mean square of Table 13 by 30, the number of X,y pairs.

Because the hypothesis of no point effect was rejected, the technique in the previous example was used to adjust values of the dependent variable to take account of the small differences among the associated values of the independent variable. For polynomial relationships, correction of the neighboring pairs to the same $\bar{X}_i$ is made by using the relationship $y'_{ij} = y_{ij} - b'(X_{ij} - \bar{X}_i)$ where b' is the derivative of the cubic fit evaluated at $\bar{X}_i$ (see Section VII). In this particular example, since $X_{ij} - \bar{X}_i$ is -0.005 for all i in run 1, zero in run 2, and +0.005 for all i in run 3, the use of a simple average at each point would not introduce any error.

The results of the polynomial fits to the adjusted ten $\bar{X}, \bar{y}'$ points are presented in Table 14. While in this example the values for the coefficients are nearly the same as for the overall cubic line (Table 12), the adjusted data cubic line provides the valid estimates of the mean squares for the analysis of

Table 14

Analysis of Polynomial Fits to Adjusted Data

Pt. No.	x	y	Predicted y			Residual y		
			Cubic	Quadratic	Linear	Cubic	Quadratic	Linear
1	.285	1.366	1.381	1.425	1.661	.014	.058	.295
2	.485	2.070	2.043	2.028	2.107	-.026	-.041	.037
3	.685	2.620	2.628	2.591	2.552	.008	-.028	-.067
4	.885	3.156	3.148	3.115	2.997	-.007	-.040	-.158
5	1.085	3.606	3.613	3.600	3.443	.006	-.006	-.163
6	1.285	4.020	4.033	4.045	3.888	.013	.025	-.131
7	1.485	4.410	4.419	4.451	4.333	.009	.041	-.076
8	1.685	4.793	4.781	4.818	4.778	-.012	.024	-.014
9	1.885	5.150	5.130	5.145	5.224	-.019	-.004	.074
10	2.085	5.463	5.477	5.443	5.669	.014	-.030	.206

Estimates of Coefficients of Adjusted Data

	a	b ₁	b ₂	b ₃
Cubic	.2852	4.1912	-1.2745	.2200
Quadratic	.4985	3.3934	-.4924	
Linear	1.0274	2.2264		

variance presented in Table 15. The cubic coefficient is highly significant so the best calibration line is the cubic fit to the adjusted ten $\bar{X}, \bar{y}'$ points. Since Table 15 is based on a set of data where each point consists of the average of three X, y' pairs, it is not unexpected that the variance of the points about the line is roughly one-third of the between-points mean square in Table 13.

Table 15				
Analysis of Variance of Adjusted Data				
Source	Degrees of freedom	Mean square	F ratio	Std. dev. about line
Mean	1/9	134.371145548	72.9680	1.3570
Linear	1/8	16.357053561	604.3927	.1645
Quadratic	1/7	.204850060	122.9922	.0408
Cubic	1/6	.009566838	27.4379	.0186

In this example, additional runs would improve the reliability of the calibration equation whereas more points per run would not. This is the opposite of the case for the preceding example, in which more runs did not improve the reliability of the equation but more points per run would better define the tank.

V. EXAMPLE 4 - PROCESS TANK DATA II

Data on the calibration of a process tank, supplied by Anderson,⁸ were fitted by using the independent model, and the results were examined by analysis of covariance. The least-squares equations for the four runs are

$$\begin{aligned}
 Y_1 &= -1.408 + 9.561X_1, \\
 Y_2 &= -1.462 + 9.571X_2, \\
 Y_3 &= -1.483 + 9.567X_3, \\
 Y_4 &= -1.454 + 9.509X_4.
 \end{aligned}
 \tag{7}$$

Inspection of Eq. (7) suggests that runs 1 and 4 are different: the estimate is low in run 1 for the y intercept and in run 4 for the slope. Data for the four runs and the usual least-squares listings are given in Table 16. The

Table 16

Linear Least-Squares Analysis - Example 4

Pt. No.	Obs x	Obs y	Pred. y	Resid. y	Cusum resid.	R _n	Pred. x	Resid. x
Run 1								
1	.6818	5.160	5.110	-.049	-.049	1.237	.6869	.0051
2	.9103	7.230	7.295	.065	.015	1.555	.9034	-.0068
3	1.1350	9.440	9.443	.003	.019	.081	1.1346	-.0003
4	1.3636	11.650	11.629	-.020	-.001	.468	1.3657	.0021
5	1.5907	13.810	13.800	-.009	-.011	.212	1.5917	.0010
6	1.8182	16.020	15.975	-.044	-.055	.976	1.8228	.0046
7	2.0458	18.140	18.151	.011	-.044	.254	2.0445	-.0012
8	2.2734	20.270	20.327	.057	.013	1.275	2.2673	-.0060
9	2.5000	22.420	22.494	.074	.087	1.666	2.4922	-.0077
10	2.7267	24.720	24.661	-.058	.029	1.346	2.7328	.0061
11	2.9574	26.900	26.867	-.032	-.003	.781	2.9608	.0034
12	3.1777	28.970	28.973	.003	.000	.087	3.1773	-.0003
Run 2								
1	.5678	3.980	3.972	-.007	-.007	.206	.5686	.0008
2	.7940	6.190	6.137	-.052	-.060	1.334	.7995	.0055
3	1.2482	10.470	10.484	.014	-.046	.337	1.2467	-.0014
4	1.4753	12.680	12.657	-.022	-.068	.513	1.2467	-.0014
5	1.7024	14.750	14.831	.081	.012	1.863	1.6938	-.0085
6	1.9295	16.960	17.004	.044	.057	1.023	1.9248	-.0046
7	2.1576	19.170	19.188	.018	.076	.412	2.1557	-.0018
8	2.3847	21.390	21.361	-.028	.047	.650	2.3876	.0029
9	2.6118	23.600	23.535	-.064	-.017	1.509	2.6185	.0067
10	2.8389	25.660	25.708	.048	.031	1.163	2.8338	-.0050
11	3.0660	27.880	27.882	.002	.034	.057	3.0657	-.0002
12	3.2931	30.090	30.055	-.034	.000	.874	3.2966	.0035
Run 3								
1	.6789	5.010	5.011	.001	.001	.001	.039	-.0001
2	.9078	7.220	7.201	-.018	-.016	.364	.9097	.0019
3	1.1367	9.420	9.391	-.028	-.045	.548	1.1396	.0029
4	1.3656	11.490	11.581	.091	.046	1.707	1.3560	-.0095
5	1.5917	13.840	13.744	-.095	-.049	1.766	1.6017	.0100
6	1.8183	15.900	15.911	.011	-.037	.218	1.8170	-.0012
7	2.0440	18.110	18.071	-.038	-.076	.711	2.0480	.0040
8	2.2734	20.180	20.265	.085	.009	1.579	2.2644	-.0089
9	2.4995	22.380	22.428	.048	.057	.911	2.4944	-.0050
10	2.7266	24.590	24.601	.011	.068	.215	2.7254	-.0011
11	2.9537	26.800	26.773	-.026	.042	.521	2.9564	.0027
12	3.1830	29.010	28.967	-.042	.000	.889	3.1874	.0044

Table 16 (cont'd)

Pt. No.	Obs x	Obs y	Pred. y	Resid. y	Cusum resid.	R _n	Pred. x	Resid. x
Run 4								
1	.6832	5.100	5.042	-.057	-.057	1.441	.6892	.0060
2	.9094	7.200	7.193	-.006	-.064	.159	.9101	.0007
3	1.1367	9.370	9.354	-.015	-.079	.352	1.1383	.0016
4	1.3628	11.500	11.504	.004	-.074	.165	1.3623	-.0004
5	1.5908	13.690	13.672	-.017	-.092	.382	1.5926	.0018
6	1.8170	15.720	15.823	.103	.011	2.277	1.8060	-.0109
7	2.0450	17.970	17.991	.021	.033	.477	2.0427	-.0022
8	2.2711	20.110	20.141	.031	.064	.701	2.2677	-.0033
9	2.4982	22.270	22.301	.031	.096	.701	2.4949	-.0032
10	2.7257	24.470	24.464	-.005	.090	.127	2.7262	.0005
11	2.9504	26.620	26.601	-.018	.071	.450	2.0523	.0019
12	3.1738	28.840	28.768	-.071	.000	1.795	3.1858	.0075

y residuals are plotted in Figure 6. The estimates of the coefficients and their standard errors are given in Table 17.

The results obtained in the analysis of covariance are summarized in Table 18, and they indicate that the lines are parallel (hypothesis 1). However, hypothesis 2, that the four run means fall on a straight line, was rejected at the $\alpha = 0.001$ level. Hypothesis 3 is the assumption that there is no difference between $\hat{b}$ and $\bar{b}$.^{*} Hypothesis 3 was rejected at the $\alpha = 0.05$ level.

Inspection of the plot of the residuals (Figure 6) suggests that run 4 might be the source of the rejections of hypotheses 2 and 3. Data in the upper regions of the tank for calibration run 4 are substantially different from those for the other three calibration runs. Calculation of the overall line and the analysis of covariance were repeated with calibration run 4 omitted. These results are summarized in Tables 19 and 20. Hypothesis 2 was again rejected, this time at the $\alpha = 0.005$ level, which indicates that calibration run 4 was not the sole source of the lack of agreement between lines.

The analysis of covariance is relatively sensitive to changes in the data set means. A small shift in the y-intercept, or any other factor that results in a shift of the data set means, is indicated by a rejection of hypothesis 2 or 3 in the analysis of covariance.

With the rejection of hypothesis 2 further analysis based on the combination of the three calibration runs by summing all data points (Sum line in Table 19) is not valid. If possible, the physical cause of the differences in the group means should be determined and eliminated. The analyst at this point

^{*}These terms are defined in Table 24.

Figure 6. Plot of residuals for overall linear line - Example 4.

Table 17

Estimates of Coefficients and Standard Errors - Example 4

κ	a	b	Std. dev. y resid.	Std. dev. a	Std. dev. b	u
1	-1.40804573	9.56086334	.04760452	.03653035	.01752171	-.1091
2	-1.4621546	9.57094091	.04590323	.03425007	.01574595	-.1420
3	-1.48282038	9.56650119	.05713866	.04381574	.02101496	1.0453
4	-1.45412384	9.50896843	.04759061	.03654909	.01754210	-1.2884
Sum	-1.45447373	9.55338367	.07504452	.02855214	.01357905	-4.1138
$\bar{x}, \bar{y}$	-1.45237801	9.55255552	.02312145	.01762191	.00836399	-1.5563

Table 18

Analysis of Covariance

Source of variance	Sum of squares	Degrees of freedom	Mean square	F ratio
Between $\hat{b}$ and $\bar{b}$	.013061025	1	.013061025	5.27558
Deviations of group means about their least-squares line	.128163535	2	.064081767	25.88378
Between individual slopes	.018802750	3	.006267583	2.53159
Deviations of individual points about their least-squares lines	.099029990	40	.002475749	
About the overall line	.259057300	46	.005631680	
Due to the overall line	2800.323109400	1	2280.323109400	

Table 19

Estimates of Coefficients and Standard Errors, Runs 1-3

k	a	b	Std. dev. y resid.	Std. dev. a	Std. dev. b	u
1	-1.40804573	9.56086334	.04760452	.03653035	.01752171	- .1091
2	-1.46215402	9.57094091	.04590323	.03425007	.01574595	- .1420
3	-1.48282038	9.56650119	.05713866	.04381574	.02101496	1.0453
Sum	-1.45122851	9.56627625	.05458779	.02391444	.01130476	- .8534
$\bar{X}, \bar{y}$	-1.45187456	9.56687073	.01847333	.01407937	.00668257	-1.4011

Table 20

Analysis of Covariance, Runs 1-3

Source of variance	Sum of squares	Degrees of freedom	Mean square	F ratio
Between $\hat{b}$ and $\bar{b}$	.000041608	1	.000041608	.01634
Deviations of group means about their least-squares line	.024489767	1	.024489767	9.61875
Between the individual slopes	.000401440	2	.000200720	.07883
Deviations of individual points about their least-squares lines	.076381320	30	.002546044	
About the overall line	.101314140	34	.002979827	
Due to the overall line	2133.801741460	1	2133.801741460	

has two options; (a) to request more calibration runs, or (b) to accept the existing data and find the equation least likely to result in misrepresentation of the reliability of the measurements based on this calibration.

To obtain valid estimates of the coefficients and their standard errors, the analysis proceeded with adjustment of the data according to Section VII. The results of the least-squares fit of the twelve $\bar{X}, \bar{y}'$ points for all four data sets and for only the first three data sets are shown in Table 21 and 22, respectively.

The results in Table 23 indicate that the linear analysis of runs 1 to 3 provides the more acceptable results. If the overall line had not been rejected because of hypothesis 2, it might have been preferred to the adjusted line on the basis of the lower u value and a large estimate of the standard deviation of the slope.

A comparison of the estimate of the standard deviation of the slope for the adjusted data for the three runs (line labeled $\bar{X}, \bar{y}'$ in Table 19) with that of the estimate of the standard deviation of the slope obtained by summing all data points (line labeled Sum in Table 19) indicates that these estimates differ by approximately the factor square root of k, the number of calibration runs.

$$(0.011305)/\sqrt{3} = 0.006527$$

$$\sim 0.006716$$

Examples 2 and 4 illustrate the hazard of uncritically combining data sets into an overall line and the importance of examining the curves fitted to the adjusted data. Additional discussions of these examples are given in parts A and B of Section VII.

Table 21

One-way Analysis of Variance, Runs 1-4

k	$\bar{X}$	Adjusted y' values			
		Run 1	Run 2	Run 3	Run 4
1	.6529	4.8841	4.7932	4.7618	4.8107
2	.8803	6.9441	7.0151	6.9579	6.9227
3	1.1641	9.7184	9.6670	9.6822	9.6322
4	1.3918	11.9196	11.8825	11.7405	11.7772
5	1.6189	14.0794	13.9522	14.0998	13.9584
6	1.8457	16.2831	16.1599	16.1622	15.9946
7	2.0731	18.4008	18.3627	18.3880	18.2384
8	2.3006	20.5303	20.5870	20.4403	20.3923
9	2.5273	22.6815	22.7934	22.6463	22.5487
10	2.7544	24.9853	24.8534	24.8563	24.7448
11	2.9818	27.1338	27.0763	27.0691	26.9206
12	3.2080	29.2597	29.2772	29.2490	29.1239

Source	Sum of squares	Degrees of freedom	Mean square	F ratio
Slope	2789.332544120	1	2789.332544120	423078.2456
Means	.021568580	10	.002156858	.3271
Error	.237346100	36	.006592947	

Table 22

One-way Analysis of Variance, Runs 1-3

k	$\bar{X}$	Adjusted y' values		
		Run 1	Run 2	Run 3
1	.6529	4.8841	4.7932	4.7618
2	.8803	6.9441	7.0151	6.9579
3	1.1641	9.7184	9.6670	9.6822
4	1.3918	11.9196	11.8825	11.7405
5	1.6189	14.0794	13.9522	14.0998
6	1.8457	16.2831	16.1599	16.1622
7	2.0731	18.4008	18.3627	18.3880
8	2.3006	20.5303	20.5870	20.4403
9	2.5273	22.6815	22.7934	22.6463
10	2.7544	24.9853	24.8534	24.8563
11	2.9818	27.1338	27.0763	27.0691
12	3.2080	29.2597	29.2772	29.2490

Source	Sum of squares	Degrees of freedom	Mean square	F ratio
Slope	2098.294577030	1	2098.294577030	555452.1553
Means	.010345470	10	.001034547	.2738
Error	.090663200	24	.003777633	

Table 23

Summary of Results of Tests on Example 4

	Std. dev. of slope	u	Analysis of covariance	Analysis of variance
Runs 1-4, linear analysis	<u>from Table 17</u>		<u>from Table 18</u>	<u>from Table 21</u>
a) Individual lines	0.009029		--	--
b) Overall line	0.013579	-4.11*	hypotheses 2 & 3 rejected	--
c) Adjusted line	0.008864	-1.56	--	Means
Runs 1-3, linear analysis	<u>from Table 19</u>		<u>from Table 20</u>	<u>from Table 22</u>
a) Individual lines	0.010523	--	--	--
b) Overall line	0.011305	-0.85	hypothesis 2 rejected	--
c) Adjusted line	0.006683	-1.40	--	Means

*Hypothesis of no effect was rejected.

VI. ANALYSIS OF COVARIANCE

The statistical notation and approach to the analysis of covariance used in this report are those given by Brownlee.⁴ The particular formulation of the analysis of covariance of interest is that of evaluating the consistency between k least-squares equations. By testing an ordered set of hypotheses the experimenter can determine whether or not all data points may be considered to lie on a single overall line. The notation and definitions used in the test statistics are shown in Table 24.

The analysis of covariance involves the partitioning of the total sum of squares about the overall line into the independent component parts associated with slopes and group means that are then tested for significance relative to the variation due to unassignable causes. In this case the unassignable variation represents the variance of the points about their individual lines.

The test of hypothesis tested is summarized in Table 25. The following hypotheses are tested:

- $H_1: S_2^2/S_1^2$, the least-squares equations for each of
the data sets can be regarded as parallel.
- $H_2: S_3^2/S_1^2$, the data run means $(\bar{y}, \bar{x})$ are not significantly different
from the least-squares equation through them.
- $H_3: S_4^2/S_1^2$, the slope, $\bar{b}$, of the composite least-squares equation is
not significantly different from the slope, $\hat{b}$, resulting
from H_2 .

*No attempt is made here to develop the general method of analysis of covariance (ANOCOVA) but only to present techniques for the interpretation of such results in problems involving volume calibrations. For a complete treatment of ANOCOVA see Chapter 11 of Brownlee.⁴

Table 24

Algebraic Notation

Pt. No. $j = 1, 2, \dots k$ (number of runs)	Data points $i = 1, 2, \dots n_k$ (number of points in run k)	Least-squares (l.s.) equations*
1	(X_{1i}, y_{1i})	$Y_1 = \bar{y}_{1.} + b_1(X - \bar{X}_{1.})$
2	(X_{2i}, y_{2i})	$Y_2 = \bar{y}_{2.} + b_2(X - \bar{X}_{2.})$
.	.	...
j	(X_{ji}, y_{ji})	$Y_j = \bar{y}_{j.} + b_j(X - \bar{X}_{j.})$
.	.	...
k	(X_{ki}, y_{ki})	$Y_k = \bar{y}_{k.} + b_k(X - \bar{X}_{k.})$

Equations from which the test statistics are derived:

b_i	The k individual lines	$Y = \bar{y}_{.i} + b_i(X - \bar{X}_{.i})$
$\bar{b}$	The k parallel lines with average slope, $\bar{b}$	$Y = \bar{y}_{.i} + \bar{b}(X - \bar{X}_{.i})$
$\hat{b}$	The l.s. line for the group means $(\bar{X}_{j.}, \bar{y}_{j.})$	$Y = \bar{y}_{..} + \hat{b}(X - \bar{X}_{..})$
b	The overall l.s. line	$Y = \bar{y}_{..} + b(X - \bar{X}_{..})$

*The conventional dot notation is used to indicate the subscript summed over, e.g., the symbols $\bar{x}_{..}$, $\bar{y}_{..}$ denote overall means.

Table 25

Brownlee's Covariance Table

Source of variance	Sum of squares	Degrees of freedom	Mean square	F ratio
Between $\hat{b}$ and $\bar{b}$	SS_4	1	S_4^2	S_4^2/S_1^2
Deviations of group means about their least-squares line	SS_3	$k - 2$	S_3^2	S_3^2/S_1^2
Between the individual slopes	SS_2	$k - 1$	S_2^2	S_2^2/S_1^2
Deviations of individual points about their least-squares lines	SS_1	$kn_k - 2k$	S_1^2	
About the overall line	SS_{about}	$kn_k - 2$		
Due to the overall line	$SS_{\text{due to}}$	1		

Hypothesis H_1 should be a two-tailed test. The F ratio of H_1 can be significantly low. A low F ratio indicates that the observations are all locally bound and the k estimates of the intercept and slope are not independent.

The plot of the residuals of all runs on a single graph will usually indicate the cause. Two conditions that may result in a low F ratio are (1) erratic variations due to nonlinearity of the instrument or imperfections in the tank with all calibration points falling at about the same volume measurement values, and (2) smooth changes in the cross sectional area so that a quadratic or cubic calibration equation is required. These two cases are discussed above (Examples 2 and 3).

A significantly high value of F for the ratio S_2^2/S_1^2 indicates either that the lines are not parallel or that the pooled residual variance (S_1^2) about the individual lines is underestimated. Comparison of the k graphs of the lines may indicate which set is different. The presence of a cumulative error in the volume measurements resulting in serial correlation will cause underestimation of the residual variance.

High critical F values may imply that the relation between X and y is poorly defined. Skew lines may be caused by the lack of good calibration procedure or because of departure from the calibration procedure in one or more of the calibration runs.

The second hypothesis tested is that the $\bar{X}, \bar{y}$ points of the k sets are linearly related. If the F ratio of H_2 is significantly large this hypothesis is rejected. Probable causes of poor agreement between the data sets' means are either appreciable cumulative error in the volume measurements or variability in the tank heels.

The third hypothesis tested is that there is no significant difference between the two slopes estimated, namely, the average slope of the k lines and the slope of the linear relationship between the k $\bar{x}, \bar{y}$ points. If the F ratio is significantly large this hypothesis is rejected, and the plot of the residuals should be examined to determine the source. This test is strongly dependent on the magnitude of the differences between the volume measurements of the k sets. If the same volume measurements are repeated in each run, the value of S_4^2 will approach zero and the analysis of variance on adjusted data is recommended. Example 2 above is a case of this type.

If a hypothesis is rejected at any step of the analysis, further testing of hypothesis should be abandoned, and the data, procedures, and calibration equipment should be examined for conditions that may result in critical F values. A discussion on several conditions is given below.

When none of the hypotheses is rejected, the data may be pooled into a single overall line and a , b , $V(a)$, $V(b)$, and covariance (a , b) calculated from the pooled data.

VII. ADJUSTMENT OF DATA AND THE ANALYSIS OF VARIANCE

Rejection of a hypothesis in the analysis of covariance indicates that at least one of the assumptions on which the analysis is based has been violated and therefore the model assumed is not applicable. The experimenter must then formulate a new model consistent with the distribution of the observed error. In Section III, Examples 2 and 3 were used to show how adjustment of the data and the analysis of variance might be used in the diagnosis of anomalous calibration data. Before the analysis is continued, Bartlett's test of the homogeneity of the variance of the residuals should be made (Brownlee^{4d}).

In appraising the characteristics of anomalous data in Section III, values of the dependent variable were adjusted to take into account differences among the associated values of the independent variable. The average value of the volumes, $\bar{X}_i$, for each cluster of points was calculated, and the difference between this value and the observed value for each point i and data set j was used to calculate y'_{ji} , a new value for y_{ji} .

The transformation is defined by

$$Y'_{ji} = Y_{ji} - b(X_{ji} - \bar{X}_i) \quad (8)$$

where b = the overall slope defined in Table 24. A graphic illustration of the transformed data would show the k values of y'_i appearing on a line erected vertically at each $\bar{X}_i$.

The form of the data is now

$$(\bar{X}_i, y'_{1i}, y'_{2i}, \dots y'_{kj}) \quad (9)$$

In the parlance of analysis of variance, the $\bar{X}_i$ are treatment levels and the y'_{ji} are replicate determinations in a one-way classification. The component of variance model (Model II) is used to estimate the mean and variance for each treatment $\bar{X}_{.i}$. The model is

$$y'_{ji} = \mu_i + T_i + \epsilon_{ji} \quad (10)$$

where

$$\mu_i = \text{true magnitude of } y'_i,$$

T_i = an effect due to the adjustment of the y'_i at point i , and

ϵ_{ji} = random measurement error common to all observations.

The term μ_i is constant for all measurements for a given point i . The term T_i is a calculated value which is the difference, due to changes in X_{ji} , of the particular value y_{ji} from the average of the i th point. It is assumed that each class i is a sample of k from a normal distribution with mean $\mu + T_i$ and variance σ^2 .

The random measurement error, ϵ_{ji} , often referred to as the experimental error, accounts for all uncontrolled effects not related to the treatments (in this case the adjustments of y'_i). As such, the experimental error is the combined effect of many random variables independent of the treatment effect. The ϵ_{ji} 's are assumed to be normally distributed with expected value zero and variance σ_c^2 .

A. Additional Discussion of Example 2

In Example 2, abnormally low F ratios were observed in the analysis of covariance in testing for parallel lines or linear relationships between the means. Under these conditions the k runs were considered replicates of a single calibration. This required the removal of the effect, T_i , due to differences between the volume measurements of the k sets. Eq. (8) was used to adjust the data. The adjusted data for Example 2 are shown in Table 6.

In the analysis of variance the F ratio for the variation of the nine group means about the line compared with the variation of the points from the six runs about their mean was significant at $\alpha = 0.001$. Thus, the variability between the six runs is much less than the error associated with the fitting of the linear least-squares line.

A one-way analysis of variance was made with n cells each having $k - 1$ degrees of freedom. The hypothesis of homogeneity of within-cell variance was tested by Bartlett's test. Since the variances were found to be homogeneous, the within-cell variances were pooled and the variance of $\bar{y}'_1$ was calculated. These and other intermediate calculations are given in Table 26.

The results of the least-squares fit of $\bar{X}, \bar{y}'$ shown in Table 4 are given in more detail in Table 26. The residual variance about the least-squares line should not be significantly greater than the pooled variance of $\bar{y}'$. However, in the case of Example 2, the standard deviation of the $\bar{y}$ residual is 0.037004 (see Table 4) and the pooled standard deviation of $\bar{y}$ is 0.001963 in Table 26. Although this still shows a significant lack of fit to a straight line, it is concluded that the model based on the adjusted data provides reasonable (i.e., correct) estimates of the variance of the slope, $V(b)$, and of the residual variance, $V(y)$.

B. Additional Discussion of Example 4

In Example 4, hypotheses 2 and 3 in the analysis of covariance were rejected because of high F values in testing the four data runs. Data run 4 was then eliminated, and a second analysis of covariance was run on the remaining three data runs. Hypothesis 2 was again rejected because of a high F value.

Although the F ratio of the mean square due to differences of the individual slopes compared with the mean square of the points about their lines (hypothesis 1) was not significantly low, the possibility of changes in the cross sectional area of the tank could not be ignored in view of the indication of considerable measurement error as evidenced by the deviation of the group means. The data for calibration runs 1 through 3 were adjusted by using Eq. (8) and were examined in a one-way analysis of variance (the discussion from

Table 26

Computer Worksheet - Example 2

$\bar{x}$	y'			$\bar{y}'$	Degrees of Freedom	Variance y'	Standard Deviation $\bar{y}'$
113.5816	2.5352 2.5365	2.5382 2.5339	2.5360 2.5379	2.53633	5	.00000275	.000677
492.1570	36.0638 36.0712	36.0711 36.0622	36.0667 36.0717	36.06783	5	.00001729	.001697
870.7323	69.3859 69.3918	69.3916 69.3894	69.3895 69.4005	69.39149	5	.00002390	.001995
1249.2513	102.8358 102.8377	102.8446 102.8330	102.8394 102.8461	102.83949	5	.00002600	.002081
1627.8243	136.2450 136.2456	136.2538 136.2408	136.2484 136.2561	136.24833	5	.00003280	.002338
2006.3908	169.6461 169.6440	169.6504 169.6437	169.6476 169.6540	169.64766	5	.00001600	.001632
2384.9421	203.1161 203.1101	203.1174 203.1060	203.1206 203.1206	203.11516	5	.00003620	.002456
2763.5875	236.4348 236.4282	236.4368 236.4274	236.4399 236.4385	236.43433	5	.00002880	.002190
3142.1243	269.7741 269.7610	269.7667 269.7710	269.7761 269.7710	269.76999	5	.00003120	.002280
Bartlett's Test Ratio = 6.1245					Pooled Standard Deviation $\bar{y}'$ = .001963		

$$y = b_0 + b_1x$$

	Value	Std. dev.	t
b0	-7.413374535	.037003917	-200.34026
b1	.088240194	.000019488	4527.81961
	Std. Dev.	mssd	u
Res. y	.057147303	.054740515	-.26721

Table 26 (cont'd)

Point	$\bar{x}$	$\bar{y}'$	Pred. $\bar{y}'$	Diff.	Sum	Rn	Pred. $\bar{x}$	Diff.
1	113.581	2.536	2.609	.072	.072	1.614	112.757	-.824
2	492.157	36.067	36.014	-.053	.019	1.082	492.759	.602
3	870.732	69.391	69.420	.028	.048	.554	870.406	-.325
4	1249.251	102.839	102.820	-.018	.029	.350	1249.463	.211
5	1627.824	136.248	136.226	-.022	.007	.411	1628.075	.251
6	2006.390	169.647	169.630	-.016	-.009	.313	2006.580	.189
7	2384.942	203.115	203.034	-.080	-.090	1.558	2385.857	.915
8	2763.587	236.434	236.446	.011	-.078	.240	2763.453	-.133
9	3142.124	269.770	269.848	.078	.000	1.736	3141.237	-.887

here on relates to data with run 4 deleted). The adjusted data for Example 4 and the results of the analysis of variance are presented in Table 27, which shows the twelve $\bar{X}, \bar{y}'$ points and the least-squares fit. The observed standard deviation of the twelve $\bar{X}, \bar{y}'$ points about their least-squares line (0.018473) is smaller than the observed standard deviation of $\bar{y}'$ (0.035485), which indicates that the effect due to changes in the cross sectional area of the tank is negligible. In Example 4 an effect was observed that is the opposite of that in Example 2: the pooled standard deviation of the average y' is greater than the standard deviation of the $\bar{y}$ residuals. In Example 2 the major error source is in the departure of the data from a straight line; in Example 4 the major error source is in the y data.

The conclusion drawn regarding these calibration data is that the major source of error is in the manometer measurements. In particular, it appears that the sensitivity of the manometer is the limiting feature of the measurement system. The least-squares fit of the individual data points for the overall line is judged to provide a less valid estimate of the variance of the slope than the least-squares fit of the twelve $\bar{X}, \bar{y}'$ points (adjusted data).

VIII. MEAN-SQUARE SUCCESSIVE DIFFERENCE

If the least-squares model for fitting the data is correct, the residuals will be randomly distributed about a mean of zero. The lack of randomness of the residuals is a strong indication that the data were fitted by an incorrect method. The mean-square successive difference (u) test is sensitive in many instances to nonrandom fluctuations. In particular, it is sensitive in detecting serial correlations and trends, either periodic or otherwise. The statistic

Table 27

Computer Worksheet - Example 4 for k = 3

$\bar{x}$	Adjusted y'			$\bar{y}'$	Degrees of Freedom	Variance y'	Standard Deviation $\bar{y}'$
	1	2	3				
.6529	4.8841	4.7932	4.7618	4.8130	2	.004034	.036671
.8803	6.9441	7.0151	6.9579	6.9723	2	.001417	.021734
1.1641	9.7184	9.6670	9.6822	9.6892	2	.000697	.015245
1.3918	11.9196	11.8825	11.7405	11.8475	2	.008936	.054577
1.6189	14.0794	13.9522	14.0998	14.0438	2	.006396	.046177
1.8457	16.2831	16.1599	16.1622	16.2017	2	.004966	.040688
2.0731	18.4008	18.3627	18.3880	18.3838	2	.000375	.011194
2.3006	20.5303	20.5870	20.4403	20.5192	2	.005472	.042710
2.5272	22.6815	22.7934	22.6463	22.7070	2	.005899	.044346
2.7544	24.9853	24.8534	24.8563	24.8983	2	.005674	.043491
2.9818	27.1338	27.0763	27.0691	27.0930	2	.001257	.020472
3.2080	29.2597	29.2772	29.2490	29.2619	2	.000202	.008219

Bartlett's Test Ratio = 10.2535

Pooled Standard Deviation $\bar{y}' = .035485$

$$y = b_0 + b_1x$$

	Value	Std. Dev.	t
b0	-1.451874563	.014079368	-103.12071
b1	9.566870726	.006682572	1431.61494
	Std. Dev.	mssd	u
Res. y	.018473326	.014502215	-1.40114

Point	$\bar{x}$	$\bar{y}'$	Pred. $\bar{y}'$	Diff.	Sum	Rn	Pred. $\bar{x}$	Diff.
1	.6529	4.8131	4.7943	-.0187	-.0187	1.217	.6548	.0019
2	.8803	6.9723	6.9698	-.0024	-.0212	.151	.8805	.0002
3	1.1641	9.6892	9.6849	-.0042	-.0255	.253	1.1645	.0004
4	1.3918	11.8475	11.8632	.0157	-.0097	.913	1.3901	-.0016
5	1.6189	14.0438	14.0359	-.0078	-.0175	.448	1.6197	.0008
6	1.8457	16.2017	16.2056	.0039	-.0135	.226	1.8452	-.0004
7	2.0731	18.3838	18.3812	-.0025	-.0161	.146	2.0733	.0002
8	2.3006	20.5192	20.5576	.0384	.0222	2.194	2.2965	-.0040
9	2.5272	22.7070	22.7255	.0185	.0408	1.073	2.5252	-.0019
10	2.7544	24.8983	24.8991	.0008	.0416	.048	2.7543	-.0000
11	2.9818	27.0930	27.0746	-.0183	.0232	1.128	2.9837	.0019
12	3.2080	29.2619	29.2386	-.0238	.0000	1.494	3.2104	.0024

is obtained by first calculating d^2 , the mean of the squares of the $n - 1$ successive differences between the residuals, R_i ,*:

$$d^2 = \frac{\sum (R_{i+1} - R_i)^2}{n - 2} \quad (11)$$

where

$$R_i = (y_i - \hat{y}_i),$$

y_i = observed y , and

$\hat{y}_i$ = predicted y .

The $d^2/2$ is compared with estimated variance about the least-squares line, s^2 :

$$s^2 = \sum R_i^2 / (n - 2) . \quad (12)$$

The test statistic, u , is defined by

$$u = \left[\frac{d^2}{2s^2} - 1 \right] / \left[\frac{n - 3}{(n - 1)^2 - 1} \right]^{1/2} , \quad (13)$$

and u is approximately normally distributed with mean zero and unit standard deviation.

The test of randomness is based on the agreement (or disparity) between $d^2/2$ and s^2 . In the case of long trends the ratio $d^2/2s^2$ will be less than one since the value of $d^2/2$ will not be increased by the trend to as great an extent

*The degrees of freedom that form the denominator in the equations defining d^2 and u for n values of observed data are $n - 1$.^{4c} For use of these equations on residual data, n is replaced by $n - 1$ in Eqs. (11) to (13).

as s^2 . For short oscillations both $d^2/2$ and s will increase, but the increase in $d^2/2$ will usually be proportionally larger.

Long trends are associated with high negative values of u ($u \leq -2$) and short oscillations with high positive values ($u \geq 2$). Thus, high negative values of u indicate that the dominant error is due either to serial correlation (Example 1) or to changes in the cross sectional area of the tank (Examples 2 and 3). High positive values of u indicate that the dominant error is in the y variate, which may be due to manometer reading error.

A. Additional Discussion of Example 3

The data in Example 3 were judged to be cubic rather than quadratic. The plot of the overall quadratic line is shown in Figure 7. From visual comparison of this with the cubic line (Figure 5), it is difficult to determine whether the cubic provides a significant improvement, but the F test (Table 12) indicates that it does.

The mean-square successive differences, u values, for all these data sets in Example 3 were < -2 , and are summarized in Table 9. This test and inspection of Figures 4, 5, and 7 identified the major error as being due to changes in the cross sectional area of the tank. Because the u test is applied to data within individual runs, the degrees of freedom will always be fewer than in tests based on combined run data, and hence it is a less sensitive test.

IX. SUMMARY

The calibration model is

$$y + e_y = a + b(x + e_x) + e_t$$

Figure 7. Plot of residuals for overall quadratic line - Example 3.

where a and b are the intercept and slope, respectively, and

x = "true" volume (mass)

y = "true" liquid level,

e_x = random error associated with the increment measurements,

e_y = random error associated with the liquid level measurements, and

e_t = fixed error due to tank imperfections or instrument nonlinearity.

In Example 1, e_t is set at zero and simulated calibration runs are examined to determine under what conditions it is appropriate to use the independent least-squares model and at what combination of volume-level errors to switch from the independent error model to the cumulative error model.

The typical standard deviation on a 100-liter precision prover is 0.0075 liters and for an electromanometer, 0.02 centimeters. It is shown that if a precision prover and an electromanometer are used, the independent error model is permitted, with negligible effect due to underestimation of the variance of the slope. The examination of process data suggests that many other factors are more troublesome and will normally present a greater challenge to the analyst.

The plotting and close examination of the residual y data was found to be the single most useful thing an analyst can do. In this report it is shown how six different statistical tests can be applied to the calibration data. These tests are important and are an integral part of the analysis, but they seldom reveal why a hypothesis is rejected or what would be a useful next step in the analysis. This information is usually provided in the plot of the y residuals. The statistical tests on the fit of the independent error model to the data are as follows:

1. The R_n test for detection of a single outlier.
2. The u test for serial correlations and trends.

3. The analysis of covariance for the comparison of several linear equations.
4. Bartlett's test for homogeneity of the variance of the residuals.
5. One-way analysis of variance on the adjusted data.
6. Cross-classification model of the analysis of variance to test run versus data point effects.

In addition, the method of orthogonal polynomials is used to fit equations of higher degree where the data warrant.

Example 2 provides an almost classic example of the case in which e_t is the major source of error. The example is used to introduce the technique for adjusting data. The method is discussed at length in Section VII. This technique depends on having the points clustered, i.e., repeating the points in succeeding runs. This was a useful exercise in every one of the examples studied in this report. The condition of having the data points repeated as closely as possible in succeeding runs is contrary to the procedure suggested in ANSI N15.19.⁹

On the basis of the examples studied in this report and other data, it is conjectured that very few tanks are perfect. In general, the analyst will need all the ingenuity and resourcefulness he can muster for the analysis of calibration data, and he should be prepared to make compromises for nonuniformity of the tank. That is, the residuals will not display a nice random pattern in such a case; however, this does not invalidate the result of the linear least-squares fit.

If the tank is nonuniform and many data points are taken, one can be assured that a test on runs of residual values of like sign will be statistically significant as will all tests based on the assumption of random error.

Example 2 also illustrates the case of an overabundance of runs and not enough points. If the principle source of error is the nonuniformity of the tank, additional points would help to plot the tank's profile. If points are being repeated, as they were here, small advantage is gained by having more than four runs.

In Example 3, the opposite effect is seen. The number of points is more than adequate but an additional run would have been helpful. It is possible that too many calibration points can result in underestimation of the variance of the slope.

In Example 3, the lack of fit of the linear least-squares line due to large e_t errors is examined. A cubic equation is used to eliminate the e_t effect.

The effects of the presence of all three error terms on a linear calibration equation are illustrated in Example 4.

Having established the principal source of error, the analyst must then decide how to treat the data in such a way that the final product is an acceptable calibration curve. At this stage it is beneficial to know how the tank is to be used and the required degree of accuracy. It may often be possible to accept the adjusted line as in Example 2 even though the line is not a perfect fit. Often, it need not be a perfect fit, but it should be sufficient for the intended use.

The techniques emphasized in this report are not intended to give minimum variance of the slope. The objective is to determine the variance that best describes the actual variability associated with the measurements made, on the basis of the calibration curve. The goal here is to obtain an estimate of the

variance of the slope such that the limit of error on the measurements truly reflects the uncertainty in the data.

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