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U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Measurement Assurance for Dimensional Measurements on Integrated-Circuit Photomasks

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Measurement Assurance for Dimensional Measurements on Integrated-Circuit Photomasks

NBS Technical Note
1112

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FOREWORD

This document has evolved from material presented as part of an on-going series of NBS training seminars on linewidth measurements for integrated-circuit photomasks and wafers sponsored by the Semiconductor Materials and Processes Division of the Center for Electronics and Electrical Engineering in cooperation with the Statistical Engineering Division of the Center for Applied Mathematics. The seminars are conducted under the NBS Semiconductor Technology Program which serves to focus NBS research on improved measurement technology for the semiconductor device community in specifying materials, equipment, and devices in national and international commerce, and in monitoring and controlling device fabrication and assembly. This research leads to carefully evaluated, well-documented test procedures and associated technology which, when applied by the industry, are expected to contribute to higher yields, lower cost, and higher reliability of semiconductor devices and to provide a basis for controlled improvements in fabrication processes and device performance.

The document is intended both as an instructional aid for future NBS training seminars on linewidth measurements and as a procedural guide for the calibration of an optical-microscope linewidth-measurement system using an NBS Optical Microscope Linewidth-Measurement Standard (SRM-474 or SRM-475) or any properly calibrated linewidth standard. It also serves as the basis for a standard method for calibration of an optical microscope for linewidth or line-spacing measurement which is currently being prepared under the auspices of the American Society for Testing and Materials (ASTM) Subcommittee F-1 on Electronics.

Special acknowledgment is gratefully made to the following NBS staff: Dr. Diana Nyyssonen and Mr. John Jerke for their helpful suggestions and discussions; Dr. James Filliben for modifications to the software package DATAPLOT which produced the graphs and tables; Dr. Clifford Spiegelman for advice and consultation particularly in regard to the derivation of uncertainties; Dr. Lee Kieffer for suggestions for clarification on many points; and Mrs. Janet Couch for her expertise and patience in the preparation of the manuscript. Special acknowledgement is also made to the Office of Measurement Services of the National Bureau of Standards which supported this work.

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Measurement Assurance for Dimensional
Measurements on Integrated-Circuit Photomasks

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Optical Microscope Linewidth-Measurement Standards, SRM-474 and SRM-475, have been developed by NBS for optical imaging systems capable of making line-spacing and linewidth measurements in the $0.5\mu\text{m}$ - $12\mu\text{m}$ regime on IC photomasks. Each artifact affords a means of reducing systematic errors via a calibration curve and keeping the optical system in statistical control. Procedures are given for accomplishing these goals along with a discussion of the uncertainty of the calibrated values.

Key Words: IC photomask; linear calibration curve; line-spacing; linewidth; measurement assurance; photomask; SRM; statistical control of measurement process; tests for systematic error; uncertainty.

1. Introduction

Optical imaging systems which use an optical microscope fitted with some type of measurement attachment are commonly used for line-spacing and linewidth measurements in the $0.5\mu\text{m}$ - $12\mu\text{m}$ regime on integrated circuit (IC) photomasks. In an effort to provide the microelectronics industry with calibration standards for determining and reducing systematic errors in line-spacing and linewidth measurements, the National Bureau of Standards undertook to develop a dimensional artifact and procedures for calibrating a variety of measurement systems. References [1], [2], and [3] describe this effort.

Past attempts to improve the accuracy of linewidth measurements have been hampered by the lack of a suitable linewidth artifact and misconceptions about the nature of line-spacing and linewidth measurements. Linewidth determination is a more difficult measurement than line-spacing determination. Line-spacing measures the displacement between two objects; it can be a left-edge to left-edge, right-edge to right-edge, or center to center measurement in which the errors in detecting the line edges cancel each other. Linewidth measures the width of a physical object; it is basically a left-edge to right-edge measurement in which the errors in detecting the edges are additive. Therefore, systematic errors in linewidth measurements are fundamentally related to edge detection [4] and to a lesser extent may also depend upon the inherent metric or line-spacing calibration of the system. Theory of the optical microscope in relation to linewidth measurements is discussed in references [5] and [6].

In the absence of a linewidth standard, the IC industry has sometimes relied on line-spacing calibration to resolve the linewidth measurement problem. This relationship was investigated in an NBS sponsored interlaboratory study involving ten IC companies [2]. Of the twenty systems analyzed in the study, only seven had substantial systematic line-spacing errors while seventeen of the systems had substantial systematic linewidth errors. A line-spacing calibration curve created for each of the seven systems with line-spacing offsets from NBS was used to correct linewidth data for those systems. In no case did the line-spacing correction eliminate the linewidth errors although it did reduce some substantially. In at least one case the offset was worse after correction. As far as systems in the study are representative of optical systems available in the industry at this time, it does not seem reasonable to expect a correction for metric to solve the linewidth measurement problem.

A solution to the linewidth measurement problem is made possible by the development of SRM-474 and SRM-475. The two artifacts are essentially the same except that the former contains four rows of lines, alternately opaque and clear, which are calibrated for linewidth; the latter contains only two rows of lines. Each row contains ten randomly arranged lines in the regime from 0.5 μ m to 12 μ m. In addition, both SRMs contain a row of ten randomly arranged spacings in the regime 0.5 μ m to 12 μ m.

The procedures in this document serve as a guide for establishing a measurement assurance program for linewidth or line-spacing measurements. The measurement assurance concept as set forth in reference [7] requires that a measurement system be tied to a defined unit of measurement or to a single measurement system such as the NBS system. The line-scale of an optical system is tied to the defined unit of length (wavelength of radiation of krypton 86) [8] by a calibration curve derived from measurements made on the calibrated spacings on the SRM. The linewidth capability of an optical system is tied to the unit of length via an NBS photometric system in which the image profile of the calibrated lines on the SRM are made to agree with theoretical profiles within the reported uncertainty [9]. This is accomplished by adherence to NBS recommended procedures for adjusting the microscope [10] and by a calibrated curve derived from measurements made on the calibrated lines on the SRM.

The measurement assurance concept also requires that the measurement system be maintained in a state of control, thus assuring that the uncertainty ascribed to the output of the system is valid at all times. Methods suggested for maintaining control on the values reported by the calibrated system and for computing the associated total uncertainty are applicable to either a linewidth or line-spacing discipline.

Although the procedures outlined in this document specify the use of SRM-474 or SRM-475, they are valid for any properly calibrated dimensional artifact as long as it is understood that several substantial issues such as the proper form of the calibration curve and possible sources of error should be addressed before attempting to adapt this analysis to a particular situation. Attention should be given to the discussions in section 4 concerning between-day differences in the calibration curve, the linearity assumption, and operator differences, keeping in mind that any problems in these areas must be resolved for a valid application of the calibration curve.

All analyses are illustrated using measurements made on an artifact similar to SRM-474 by a participant in the interlaboratory study. The raw data from the interlaboratory study are reported to two decimal places. Calculations were made via a computer, and the results are rounded to four decimal places in most of the tables. Consequently, values calculated from these rounded values may vary slightly from those reported in this document.

As a final note, the term system as used in this publication should not be thought of as only the microscope and measurement attachment per se but as the total configuration of operator, environment and apparatus that go into producing a measurement. It is recommended that a minimum of four sets of measurements be made of the lines or spacings on the SRM in order to establish a calibration curve for an optical system. The calibration curve will be valid for the system only insofar as the initial measurements that produce the calibration curve are representative of operating conditions and only as long as the system response remains in a state of control.

2. What exactly is a calibration curve?

Calibration is a process of intercomparing an unknown with a standard and assigning a value to the unknown based on the accepted value of the standard. The intercomparison can also be accomplished by treating the standard as if it were an unknown in the user's measurement process, thus eliminating any offset that may exist between the user's measurement system and the measurement system used by the laboratory such as NBS, which measured the standard. When the intercomparisons are extended over a measurement regime of interest and a functional relationship is shown to exist between the user's measurements and the accepted values for the standard, the assignment of values to the user's system is based on this functional relationship.

For the present exercise, the NBS dimensional artifact covering the $0.5\mu\text{m}$ - $12\mu\text{m}$ regime is the standard, and measurements made on the individual lines on the artifact with an optical imaging system form the basis for establishing a functional relationship between the two systems. A least-squares technique is employed to derive a best fitting straight line to the measured linewidths as a function of the NBS values. This empirical fit is called the calibration curve.

In Figure 1, each optical measurement is plotted against the corresponding NBS value, and the calibration curve fitted to all the measurements is shown by the solid line. The offset between the user's system and the NBS system is reduced by relating any future linewidth value back to the NBS value. Schematically, for a future linewidth value $y(T)$ as shown on the y-axis, a dotted line is drawn through $y(T)$ parallel to the x-axis. At the point where it intersects the calibration curve another dotted line is drawn parallel to the y-axis, and its point of intersection on the x-axis, $x(T)$, is the corresponding calibrated value relative to NBS.

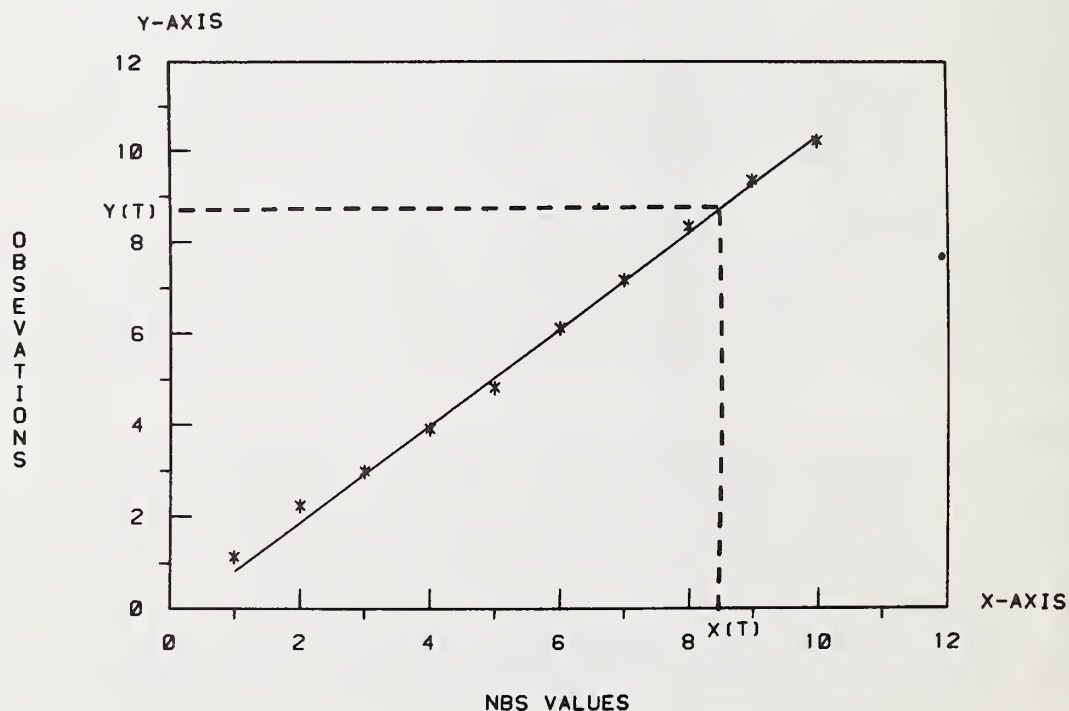


Figure 1. Schematic Diagram of Calibration Curve

3. Is the optical system in need of calibration and, if so, how is this accomplished?

The question of whether or not a calibration is needed for an optical imaging system can be resolved by applying the procedures in this section to measurements made on the NBS artifact. If a calibration is indicated, the same measurements are used to construct a calibration curve which in turn is used to correct all future measurements on the optical systems.

The determination of systematic error is made independently for line-spacing and linewidth measurements. A line-spacing calibration curve for reducing systematic line-spacing errors is derived from measurements on the ten line-spacings on the SRM. Linewidth calibration curves for reducing systematic linewidth errors for opaque and clear lines are derived from measurements on the ten opaque lines and the ten clear lines on the SRM. Separate calibration curves are needed for each polarity because it is expected that offsets for clear lines will be in the opposite direction from offsets for opaque lines.

A minimum of four repetitions of the measurements on the ten lines or spacings on the SRM are needed in order to establish a calibration curve. These repetitions should be spaced over roughly a two-week interval to ensure independence among the measurements, and it is reemphasized that the efficacy of the calibration curve as a device for reducing systematic errors is dependent upon the repetitions being truly representative of operating conditions in the laboratory. The user is further cautioned that different operators may produce measurements with systematic errors that are substantially different in sign or in magnitude, thereby necessitating separate calibration curves for each operator.

The procedures and analyses that are set forth in this publication are intended for either line-spacing or linewidth measurements. Because it is anticipated that the majority of users will be primarily concerned with linewidth determination, the text and examples are in terms of linewidth, but the total content including equations and tests are equally applicable to line-spacing measurements. Examples of the derivation of both a line-spacing calibration curve and a linewidth calibration curve are included at the end of this section.

To return to the calibration question, it is noted that an optical imaging system with negligible linewidth errors will exhibit the following characteristics. The observed linewidths will be a linear function of the nominal linewidth values assigned to the artifact by NBS. This linear function will have a slope $b=1$ and an intercept $a=0$, and the scatter about the fitted line will be sufficiently small for the system.

The thrust of the remainder of this section is to examine the relationship between the NBS values for the linewidths and the user's observations on the lines. This is accomplished by fitting the observed linewidths to a linear function of the NBS linewidth values and testing the resulting estimates of the slope and intercept for the characteristics cited above. If these tests show that linewidth errors are not negligible, thus indicating a need for correction to the optical system, the fitted curve becomes the calibration curve for the system.

Before undertaking this analysis on the data, it is suggested that the differences between the observed linewidths and the corresponding NBS values be plotted against the NBS values. Any truly aberrant or oscillatory behavior of the system will show up on the plot, and such behavior obviously precludes using such data to calibrate the system.

A plot of the differences will also make it easier to identify any outliers that are present in the data set. For the purpose of this exercise an outlier is defined as any measurement that is obviously inconsistent with the other measurements in the data set whether it be an individual point or a group of points representing a single repetition. Any such discordant points should be deleted from the data set because even a single isolated outlier can seriously perturb the calibration curve. An outlier can be particularly disruptive if it happens to lie near one of the endpoints of the calibration interval.

Formal tests for outliers are not given in this publication, but they are discussed in detail in reference [11]. A careful study of the difference plot should be adequate for spotting outliers. When one is satisfied that the data set is free of any discordant points and that it consists of measurements that are representative of the diversity of conditions affecting the measurement system, then the following analysis is performed.

Given linewidth measurements z_{ij} and corresponding NBS linewidth values w_{ij} ($i=1, \dots, k$; $j=1, \dots, 10$) where i denotes the repetition and j denotes the linewidth, least-squares estimates are obtained for a linear function of the form

$$(3.1) \quad z_{ij} = a + b w_{ij} \quad \begin{matrix} i=1, \dots, k \\ j=1, \dots, 10 \end{matrix}$$

The equations for estimating the parameters are as follows:

$$\hat{b} = \frac{\sum_i \sum_j (w_{ij} - \bar{w})(z_{ij} - \bar{z})}{\sum_i \sum_j (w_{ij} - \bar{w})^2}$$

$$\hat{a} = \bar{z} - \hat{b} \bar{w}$$

$$\text{where } \bar{w} = \frac{\sum_i \sum_j w_{ij}}{n} \quad \text{and} \quad \bar{z} = \frac{\sum_i \sum_j z_{ij}}{n}$$

The deviations from the linear fit are

$$\delta_{ij} = z_{ij} - \hat{a} - \hat{b} w_{ij}$$

and the residual standard deviation of the linear fit is

$$s = \left(\frac{\sum_i \sum_j \delta_{ij}^2}{n - 2} \right)^{1/2}$$

where n is the total number of observations, $n = 10k$.

Before proceeding with the tests for the slope and intercept, one should be satisfied that a linear function is indeed a good characterization of the system response as a function of the assigned values of the lines. A formal statistical test for deciding the appropriateness of a linear function is given in the appendix. A plot of the deviations from the linear fit provides considerable insight into this question.

The deviations are perhaps the best diagnostic tool for deciding whether or not the linear fit is a proper representation of the relationship between the NBS values and the user's system. The deviations, when plotted against the NBS values, should scatter randomly about a line drawn horizontal to the x-axis at zero with approximately an equal number of points falling above and below the line. Any apparent cyclic behavior or obvious clustering of the deviations is evidence that a linear fit is not appropriate. The NBS artifact is designed only for systems with linear responses and probably cannot be used if a higher order curve is indicated because of the limited number of lines on the artifact.

Test statistics, t_1 and t_2 , for testing if the intercept $a=0$ and if the slope $b=1$, respectively, are as follows [12]:

$$t_1 = \frac{\hat{a}}{s_a} \quad t_2 = \frac{\hat{1-b}}{s_b}$$

where

$$s_a = s \left(\frac{\sum_i \sum_j w_{ij}^2}{n \sum_i \sum_j (w_{ij} - \bar{w})^2} \right)^{1/2} \quad \text{and} \quad s_b = \left(\frac{s}{\sum_i \sum_j (w_{ij} - \bar{w})^2} \right)^{1/2}$$

A value of $|t_1| > t_{\alpha/2}(n-2)$ indicates that the intercept a is significantly different from zero, and a value of $|t_2| > t_{\alpha/2}(n-2)$ indicates that the slope b is significantly different from one.¹ The parameters a and b are tested separately so that the nature of the systematic errors can be ascertained. If only the intercept is significantly different than assumed, the errors are constant, and a constant correction would be sufficient to reduce this source of error. If the slope is significantly different than assumed, the errors are related to width of the lines, and a calibration curve is used to correct for

¹ For probability level $1-\alpha$ the critical value $t_{\alpha/2}(v)$ can be found in a table of Student's t distribution where v is the number of degrees of freedom in the residual standard deviation of the fit. Critical values for $\alpha = 0.05$ are tabulated in Table 1 for $v = 30$ (2) 120.

systematic errors. A significant value for either of these parameters implies that there are systematic linewidth errors and, as a practical matter, any future linewidth measurement z should be corrected by

(3.2)

$$z^{**} = \frac{z - \hat{a}}{\hat{b}}$$

Calculations for a line-spacing calibration curve based on measurements that were made by a participant in the interlaboratory study on an artifact similar to SRM-474 are illustrated in Worksheet A. The calculations utilize four repetitions on the spacings, and the total number of observations is $n=40$.

Worksheet A lists the NBS assigned values of the line-spacings, the participant's observed values, and the differences between the observed and NBS assigned values. The differences are plotted against the NBS values in Figure 2, and for the system to be properly adjusted for scale, these differences should be scattered randomly about the zero line drawn horizontal to the x-axis. It is obvious from Figure 2 that the line-spacing scale of the system is offset from the NBS system and that the magnitude of this offset is related to the size of the spacing. This judgment is confirmed by the t-tests for the slope and intercept of the linear function that has been fit to the data. Both t-tests are significant.

The deviations from the linear fit are plotted in Figure 3. They are sufficiently random to conclude that a linear function is appropriate for the data. Because the t-tests for the intercept and slope are significant, all future line-spacing measurements should be corrected by the calibration curve as defined in equation 3.2.

Calculations for a linewidth calibration curve based on measurements by the same participant in the interlaboratory study on the ten opaque lines on the same artifact are illustrated in Worksheet B. The calculation utilizes four repetitions on the opaque lines, and the total number of observations is $n=40$.

Worksheet B lists the NBS assigned values of the lines, the participant's observed values, and the differences between the participant's values and the NBS assigned values. The differences are plotted against the NBS values in Figure 4. It is obvious from Figure 4 that the linewidth calibration of the system is also offset from the NBS system and that the magnitude of this offset is related to the width of the lines. This is confirmed by the t-tests for the intercept and slope as shown in the worksheet.

The deviations from the linear fit are plotted in Figure 5. In this case, the deviations are randomly distributed about the zero line indicating that the linear fit is a good characterization of the data and that the calibration curve will adequately correct the system. Because the t-tests for the intercept and the slope of this linear function are significant, all future linewidth measurements should be corrected by equation 3.2.

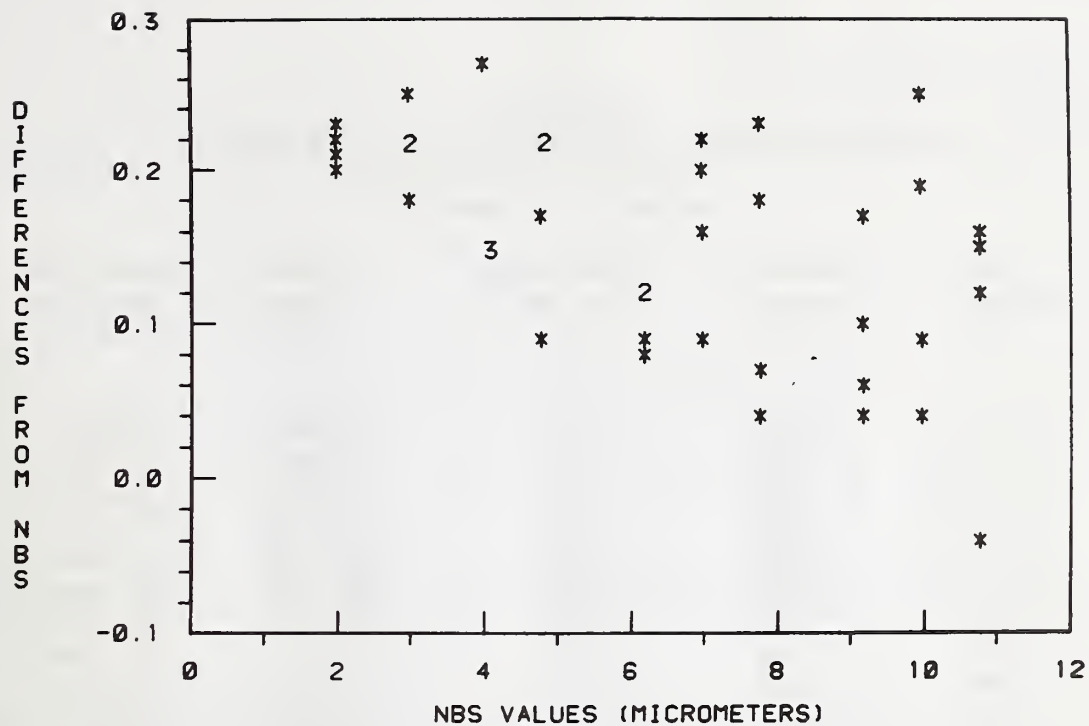


Figure 2: Differences between line-spacing measurements and NBS values plotted against NBS values.

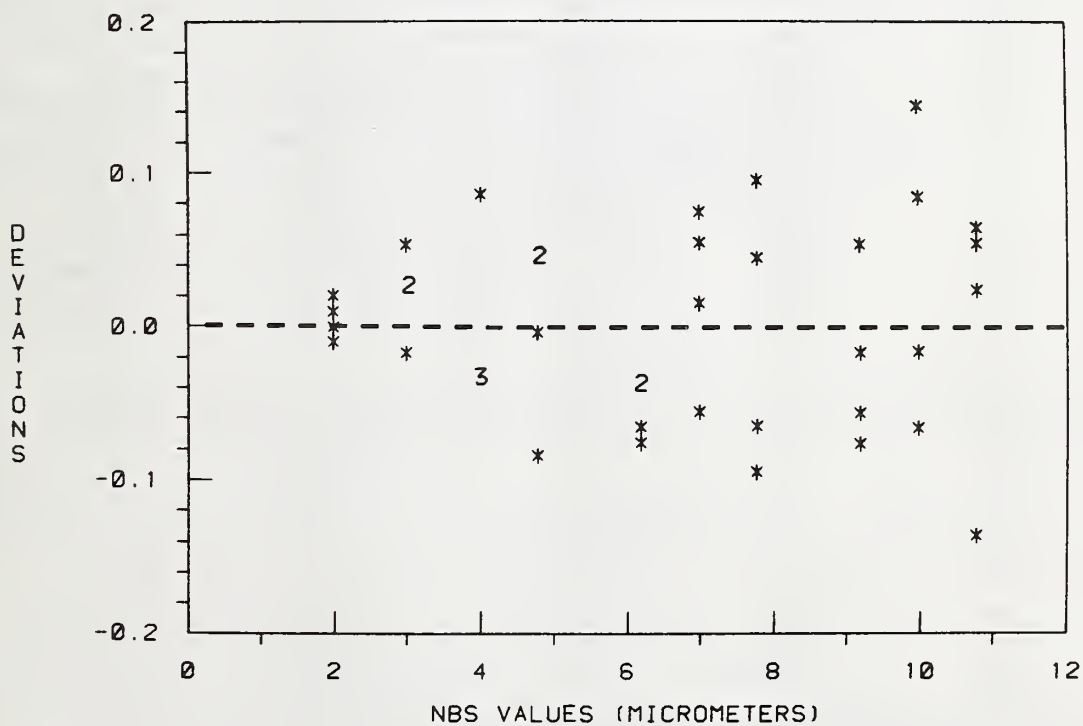


Figure 3: Deviations of line-spacing measurements from a linear calibration curve plotted against NBS values.

Worksheet A
Calculations for Line-spacing Calibration Curve
Line-Spacing Measurements
Values in Micrometers

NBS Values w	Observed Values z	Differences From NBS d	Fitted Value z'	Deviations From Fit δ
6.19	6.31	0.12	6.3455	-0.0355
9.17	9.27	0.10	9.2869	-0.0169
1.99	2.21	0.22	2.2000	0.0100
7.77	8.00	0.23	7.9050	0.0950
4.00	4.27	0.27	4.1839	0.0861
10.77	10.93	0.16	10.8662	0.0638
4.78	4.95	0.17	4.9538	-0.0038
2.99	3.24	0.25	3.1870	0.0530
6.98	7.14	0.16	7.1253	0.0147
9.98	10.23	0.25	10.0864	0.1436
6.19	6.27	0.08	6.3455	-0.0755
9.17	9.21	0.04	9.2869	-0.0769
1.99	2.19	0.20	2.2000	-0.0100
7.77	7.81	0.04	7.9050	-0.0950
4.00	4.15	0.15	4.1839	-0.0339
10.77	10.73	-0.04	10.8662	-0.1362
4.78	4.87	0.09	4.9538	-0.0838
2.99	3.17	0.18	3.1870	-0.0170
6.98	7.07	0.09	7.1253	-0.0553
9.98	10.02	0.04	10.0864	-0.0664
6.19	6.31	0.12	6.3455	-0.0355
9.17	9.34	0.17	9.2869	0.0531
1.99	2.22	0.23	2.2000	0.0200
7.77	7.95	0.18	7.9050	0.0450
4.00	4.15	0.15	4.1839	-0.0339
10.77	10.92	0.15	10.8662	0.0538
4.78	5.00	0.22	4.9538	0.0462
2.99	3.21	0.22	3.1870	0.0230
6.98	7.18	0.20	7.1253	0.0547
9.98	10.07	0.09	10.0864	-0.0164
6.19	6.28	0.09	6.3455	-0.0655
9.17	9.23	0.06	9.2869	-0.0569
1.99	2.20	0.21	2.2000	0.0000
7.77	7.84	0.07	7.9050	-0.0650
4.00	4.15	0.15	4.1839	-0.0339
10.77	10.89	0.12	10.8662	0.0238
4.78	5.00	0.22	4.9538	0.0462
2.99	3.21	0.22	3.1870	0.0230
6.98	7.20	0.22	7.1253	0.0747
9.98	10.17	0.19	10.0864	0.0836

Calculations for finding a spacing calibration curve such that $z=a + bw$ for n measurements:

$$n = 10k = 40$$

Differences from NBS

$$d = z - w$$

$$\bar{w} = \Sigma \Sigma w / n = 6.462$$

$$\bar{z} = \Sigma \Sigma z / n = 6.614$$

Estimate of slope

$$\hat{b} = \Sigma \Sigma (w - \bar{w})(z - \bar{z}) / \Sigma \Sigma (w - \bar{w})^2 = 0.9870$$

Estimate of intercept

$$\hat{a} = \bar{z} - \hat{b} \bar{w} = 0.2358$$

Deviations from linear fit

$$\delta = z - z' \quad \text{where} \quad z' = \hat{a} + \hat{b}w$$

$$\text{Standard deviation from linear fit } s = \sqrt{\Sigma \delta^2 / (n-2)} = 0.06203$$

with degrees of freedom $n-2 = 38$

Student's t-test for intercept:

$$\text{Standard deviation of } \hat{a} \quad s_a = s \sqrt{\frac{\Sigma \Sigma w^2}{n \Sigma \Sigma (w - \bar{w})^2}} = 0.02430$$

$$\text{T statistic for intercept } t_1 = \hat{a} / s_a = 9.7$$

$$\text{Critical value } t_{.025} (38) = 2.0$$

$t_1 > t_{.025}$ implies a is significantly different than zero

Student's t-test for slope:

$$\text{Standard deviation of } \hat{b} \quad s_b = s / \sqrt{\Sigma \Sigma (w - \bar{w})^2} = 0.00344$$

$$\text{T statistic for slope } t_2 = (\hat{b} - b) / s_b = 3.8$$

$$\text{Critical value } t_{.025} (38) = 2.0$$

$t_2 > t_{.025}$ implies b is significantly different than one

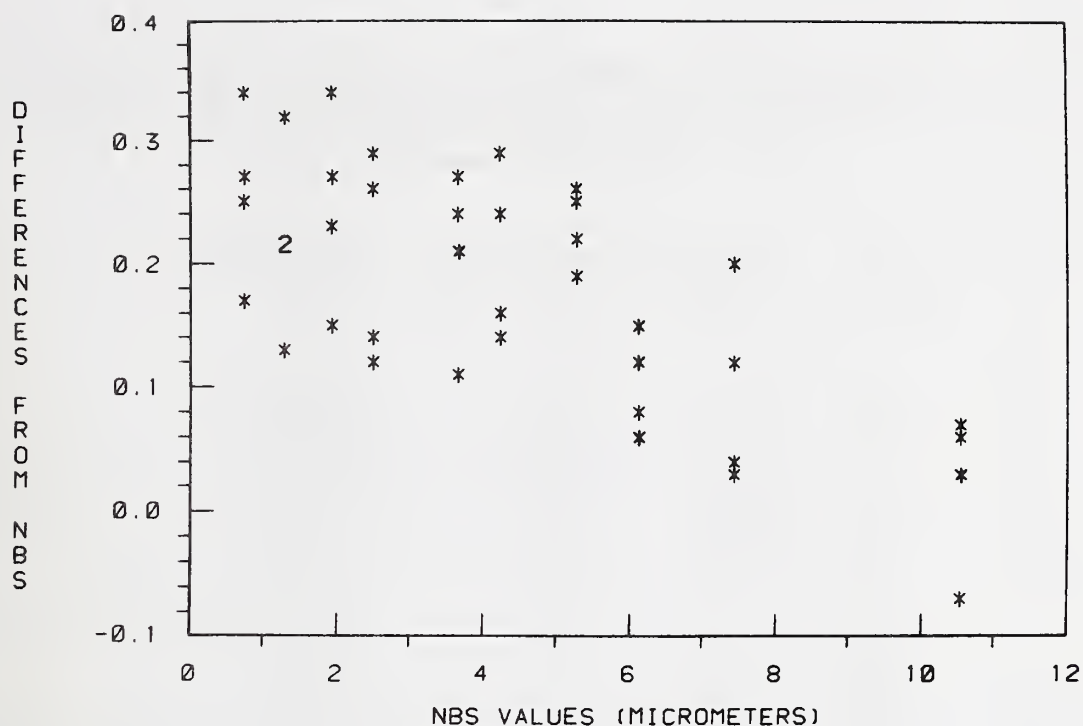


Figure 4: Differences between linewidth measurements and corresponding NBS values plotted against NBS values.

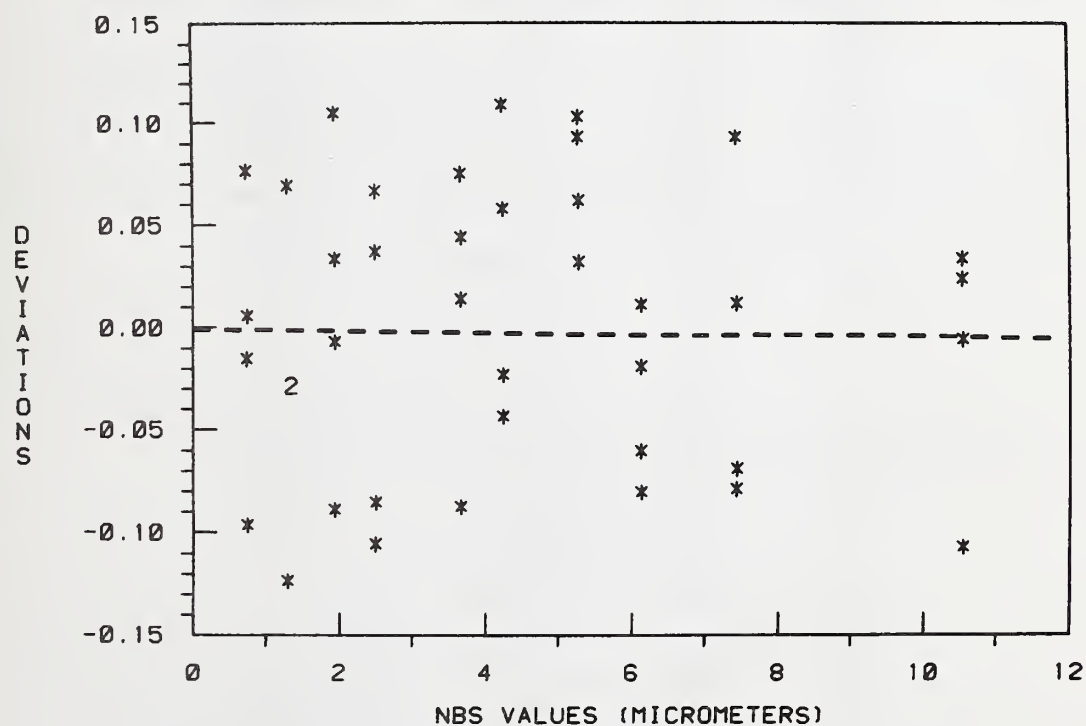


Figure 5: Deviations of observed linewidths from linear calibration curve plotted against NBS values.

Worksheet B
Calculations for Linewidth Calibration Curve
Linewidth Measurements on Opaque Lines
Values in Micrometers

NBS Values w	Observed Values z	Differences from NBS d	Deviations From Fit δ
2.50	2.62	0.12	-0.1036
1.94	2.17	0.23	-0.0066
.74	.99	0.25	-0.0145
4.25	4.39	0.14	-0.0429
10.56	10.63	0.07	0.0339
5.29	5.54	0.25	0.0913
3.67	3.94	0.27	0.0736
7.45	7.57	0.12	0.0116
1.30	1.43	0.13	-0.1215
6.14	6.26	0.12	-0.0189
2.50	2.76	0.26	0.0364
1.94	2.28	0.34	0.1034
.74	1.01	0.27	0.0055
4.25	4.54	0.29	0.1071
10.56	10.49	-0.07	-0.1061
5.29	5.48	0.19	0.0313
3.67	3.88	0.21	0.0136
7.45	7.48	0.03	-0.0784
1.30	1.52	0.22	-0.0315
6.14	6.20	0.06	-0.0789
2.50	2.64	0.14	-0.0836
1.94	2.09	0.15	-0.0866
.74	.91	0.17	-0.0945
4.25	4.41	0.16	-0.0229
10.56	10.59	0.03	-0.0061
5.29	5.51	0.22	0.0613
3.67	3.78	0.11	-0.0864
7.45	7.49	0.04	-0.0684
1.30	1.52	0.22	-0.0315
6.14	6.22	0.08	-0.0589
2.50	2.79	0.29	0.0664
1.94	2.21	0.27	0.0334
.74	1.08	0.34	0.0755
4.25	4.49	0.24	0.0571
10.56	10.62	0.06	0.0239
5.29	5.55	0.26	0.1013
3.67	3.91	0.24	0.0436
7.45	7.65	0.20	0.0916
1.30	1.62	0.32	0.0685
6.14	6.29	0.15	0.0111

Calculations for finding a linewidth calibration curve such that $z = a + bw$ for n measurements:

$$n = 10k = 40$$

$$d = z - w$$

$$\bar{w} = \Sigma \Sigma w / n = 4.384$$

$$\bar{z} = \Sigma \Sigma z / n = 4.564$$

$$\text{Estimate of slope } \hat{b} = \Sigma \Sigma (z - \bar{z})(w - \bar{w}) / \Sigma \Sigma (w - \bar{w})^2 = 0.9767$$

$$\text{Estimate of intercept } \hat{a} = \bar{z} - \hat{b} \bar{w} = 0.2817$$

$$\text{Deviations from linear fit } \delta = z - z'' \text{ where } z'' = \hat{a} + \hat{b}w$$

$$\text{Standard deviation of linear fit } s = \sqrt{\Sigma \Sigma \delta^2 / (n-2)} = 0.06826$$

with degrees of freedom $n-2 = 38$

Student's t-test for intercept:

$$\text{Standard deviation of } \hat{a} \quad s_a = s \sqrt{\frac{\Sigma \Sigma w^2}{n \Sigma \Sigma (w - \bar{w})^2}} = 0.01955$$

$$t \text{ statistic for intercept } t_1 = \hat{a} / s_a = 14.4$$

$$\text{Critical value } t_{.025} (38) = 2.0$$

$t_1 > t_{.025}$ implies that a is significantly different than zero.

Student's t-test for slope:

$$\text{Standard deviation of } \hat{b} \quad s_b = s / \sqrt{\Sigma \Sigma (w - \bar{w})^2} = 0.00372$$

$$t \text{ statistic for slope } t_2 = \hat{b} / s_b = 6.3$$

$$\text{Critical value } t_{.025} (38) = 2.0$$

$t_2 > t_{.025}$ implies that b is significantly different than one.

4. What can go wrong with the calibration procedure?

Specifically what can go wrong with the calibration procedure so that values corrected by the calibration curve do not have the desirable property of negligible offset from NBS? Possible sources of error that could affect this accuracy, some of which have already been discussed, are summarized in this section. A thorough analysis of the initial calibration data should be undertaken before the calibration program is put into operation, and if significant sources of errors are identified by this analysis, such problems should be rectified, and the initial calibration experiment should be repeated.

i) Lack of precision.

The inability to repeat measurements adequately with the system operating over a range of diverse conditions affects the accuracy of the results. Such lack of precision as indicated by a process standard deviation that is large relative to the accuracy goal of the program, is a key statistic alerting one that unidentified sources of error are afflicting the system. There is nothing intrinsic to the statistical treatment that will improve precision. The calibration curve is a device for reducing systematic error, and improvements in precision will have to come from adherence to the NBS recommended procedures for adjusting the microscope and from changes in operating conditions in the laboratory. It is important to realize that measurement assurance, while making abundant use of statistical methodology, is not achieved by statistics alone, but by the totality of procedures that relate the system to the NBS photometric system.

ii) Outliers in the initial calibration data.

Outliers in the initial calibration data can seriously distort the calibration curve especially if they lie near one of the endpoints of the calibration interval. Formal tests for the types of outliers encountered in the interlaboratory study are not given in this document because they require considerable computing capability, but they can be found in reference [2].

iii) Non-linearity of the calibration curve.

The assumption of linearity is crucial in justifying the validity of the results for the entire calibration interval. The NBS artifact is designed for systems with linear responses relative to the values assigned to the lines on the artifact. Modelling a process that is non-linear in a given region of the calibration interval would require an artifact with lines closer together over that region. A statistical test for linearity is included in the appendix.

iv) Differences among operators.

It is possible for different operators to produce measurements that have offsets that are different in sign and in magnitude. If the measurement system includes several operators, one may accept this source of error as part of the imprecision of the process by including several operators in the initial calibration experiment. This would certainly require a total of more than four repetitions. If the measurement system includes very few operators who consistently produce significantly different offsets, it may be worthwhile to establish separate calibration curves for each operator.

v) Lack of system control.

The calibration procedures outlined in this publication are dependent upon a calibration curve which is assumed to be valid for the system in its future operation. This assumption must be checked continuously because if the system response drifts or occasionally takes unpredictable excursions, the resulting values will not be properly corrected for offset. A procedure for exercising control over the calibration curve is discussed in section 6.

vi) Differences among repetitions in the initial calibration data.

The calibration curve will not produce satisfactory results if there is an essential difference in the response of the system from repetition to repetition; i.e., if the slope and intercept of the calibration curve do not correctly characterize the individual repetitions. This source of error is checked by plotting the differences between the observed linewidths and the NBS values for the linewidths against the NBS values. Connecting the points for a single repetition enables one to see the type of variation that exists among repetitions. If the slopes and intercepts of linear functions fitted to the data for each individual repetition are changing in sign or are significantly different in magnitude, the calibration curve, which is based on the averages of the repetitions, will not properly correct the system at any given time.

Comparison of slopes and intercepts can be done graphically as in Figure 6 in which the responses for each repetition are connected by solid lines with the corresponding fitted linear functions shown by dashed lines. This example illustrates that even where the overall cluster of calibration data appears to indicate a linear response, examination of the finer structure of the individual repetitions reveals very disparate responses including changes in the sign of the slope. A formal statistical test is given in appendix II.

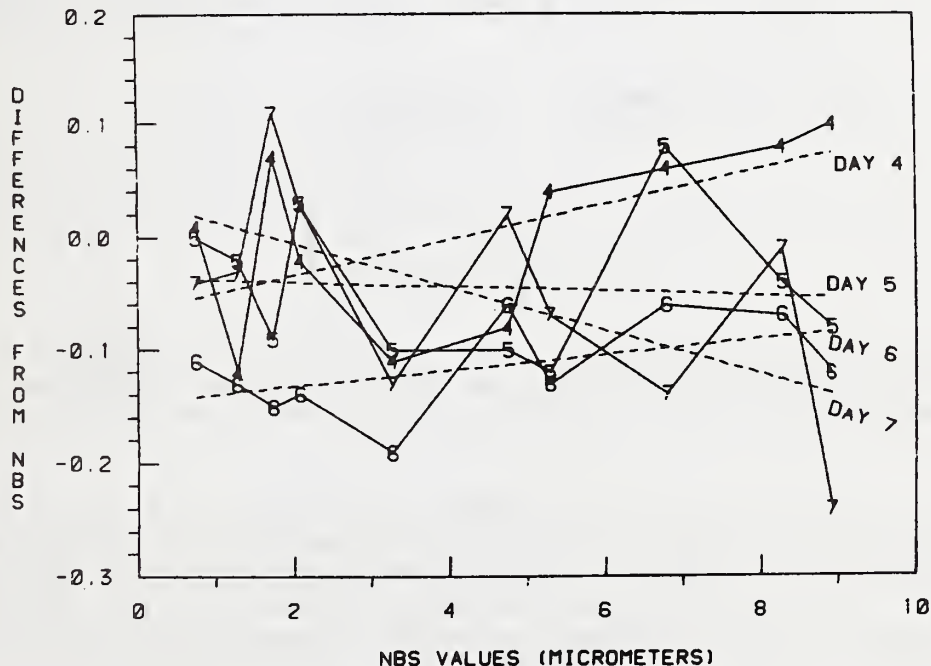


Figure 6: Daily changes in calibration curve. Differences for one day are connected by solid lines, and the corresponding calibration curve is represented by a dashed line.

5. What is the precision of the optical system and how is it estimated?

The term system precision refers to the ability to repeat measurements on the optical system from day to day or from occasion to occasion. It is based on the fact that repeated measurements on the same physical object such as an opaque line will not be identical, and the variation or scatter inherent in such measurements is referred to as the system precision. It is usually estimated by a standard deviation.

The standard deviation is computed from repeated measurements of the linewidths, and the repetitions must include all conditions for which the precision is assumed to be valid; i.e., they should include all the normal operating conditions in the laboratory. The repetitions should be sufficiently separated in time so as to adequately characterize the system. They should also span the range of linewidths of interest.

The standard deviation from the linear fit for the linewidth calibration curve is the usual estimate of the system precision. It is a proper estimate as long as the mathematical model, in this case we have assumed a linear relationship, is valid.

If there are a sufficient number of repeated measurements at each line over the entire regime of $0.5 \mu\text{m}$ - $12 \mu\text{m}$, then the standard deviation of each line is an estimate of the system precision, and the pooled standard deviation over all lines is also an estimate of this precision. For example, for linewidths z_{ij} ($i=1, \dots, k$; $j=1, \dots, 10$) where i denotes the repetition and j denotes the line, the standard deviation for the j th line is

$$(5.1) \quad s_j = \left(\frac{\sum_{i=1}^k (z_{ij} - \bar{z}_j)^2}{k-1} \right)^{1/2} \quad \text{where} \quad \bar{z}_j = \frac{\sum_{i=1}^k z_{ij}}{k}$$

The pooled standard deviation is

$$(5.2) \quad s_p = \left(\frac{\sum_{j=1}^{10} (k-1) s_j^2}{10(k-1)} \right)^{1/2}$$

Each of the standard deviations $s_1, s_2, \dots, s_{10}$ has $(k-1)$ degrees of freedom, and the pooled standard deviation s_p has $10(k-1)$ degrees of freedom. These calculations are illustrated for data on opaque lines from the interlaboratory study in Worksheet C.

The question arises, "Should the standard deviation from the linear fit or the pooled standard deviation be quoted as the estimate of process precision?" The preferred one is usually the one with the larger number of degrees of freedom, i.e., the standard deviation from the linear fit, but there are occasions when the pooled standard deviation is the more useful as described in section 7.

Worksheet C
 Calculations for a Pooled Standard Deviation
 Linewidth Measurements on Opaque Lines
 Values in Micrometers

Lines	Repetitions				Averages $\bar{z}$	Std Devs s_j
	1st z_1	2nd z_2	3rd z_3	4th z_4		
1	2.42	2.56	2.44	2.59	2.502	0.0850
2	1.96	2.07	1.88	2.00	1.978	0.0793
3	0.76	0.78	0.68	0.86	0.770	0.0739
4	4.21	4.36	4.23	4.31	4.278	0.0699
5	10.53	10.39	10.49	10.52	10.482	0.0640
6	5.37	5.31	5.38	5.38	5.360	0.0337
7	3.75	3.69	3.59	3.72	3.688	0.0695
8	7.43	7.34	7.75	7.51	7.408	0.0793
9	1.21	1.30	1.30	1.40	1.302	0.0776
10	6.10	6.04	6.06	6.13	6.082	0.0403

Standard deviation for each line $s_j = \sqrt{\frac{4}{3} \sum_{i=1}^4 (z_i - \bar{z})^2}$, $j=1, \dots, 10$

Calculation of pooled standard deviation

$$s_p = \sqrt{\frac{\sum (k-1)s_j^2}{10(k-1)}} = 0.0692$$

Degrees of freedom = $10(k-1) = 30$

6. How long will the derived calibration curve be adequate for correcting dimensional measurements?

Establishment of a calibration curve based on the recommended number of repetitions (a minimum of four sets of measurements on ten lines) constitutes the first step in what can become a full fledged measurement assurance program for linewidth measurements. The measurement assurance concept requires continual surveillance on the output of the calibrated system to ensure that the system is behaving accordingly; otherwise, the applied corrections based on the established calibration curve may degrade instead of improve the accuracy of the resulting value. That is, it must be demonstrated that the system is responding as presumed by utilizing a control procedure that is capable of detecting significant changes.

This can be accomplished by exercising quality control on the calibration curve itself in much the same way that quality control is exercised on individual components in a process. A test built into the operational procedure at given intervals can be used to see if the values corrected by the calibration curve meet the requirement of having negligible offset from NBS. The method for controlling the output of the system as corrected by the calibration curve is in keeping with a control chart approach.

This approach requires making one set of measurements on the ten lines on the artifact at least at the start-up of the measurement sequence or as often as experience dictates and testing for out-of-control conditions after each set of measurements. If ten measurements impose too great a burden on the workload, one should consider measuring only three of the lines--making sure that two of the lines are near the two endpoints of the calibration interval and that one is near the center. In fact, fewer control measurements at frequent intervals may be preferable to all ten measurements at infrequent intervals. One could alternate lines from occasion to occasion. The application of the control test will be easier, however, if the same number of lines are checked each time.

In order to conform to our previous notation, let z_i^{**} ($i=1, \dots, m$) denote the corrected linewidth measurements (see equation 3.2) and w_i denote the assigned values² for the respective lines where $m \leq 10$. If the optical system is perfectly calibrated by the calibration curve, the quantities v_i , referred to as the control values, have expected values of zero where

$$v_i = z_i^{**} - w_i \quad i=1, \dots, m$$

² The quantities w_i refer to the NBS assigned values if the calibrated pattern on the SRM is used for control purposes. In order to minimize contamination on the SRM, it is preferable to use a secondary standard for control purposes, in which case the quantities w_i refer to the values assigned to the secondary standard according to the procedures in section 3.

The control values should be evenly distributed about zero and fall within the appropriate upper and lower control limits. It is recommended that all control values be plotted as a function of time as in Figure 7. This will result in m values at each interval. If one or more values falls outside of the control limits, the system is out-of-control at that time. Such a finding precludes using the calibration curve to correct the system. The control measurements and statistical test for control should be repeated at that point, the workload being discontinued until the system is brought back into control. Continued failures should trigger an investigation into the cause and may indicate a permanent change, in which case the initial calibration measurements should be repeated and the calibration curve reestablished.

The calculation of the upper and lower control limits depends on s the estimate of system precision from the linear fit, ν the number of degrees of freedom associated with s , and m the number of control values. For probability level $1-\alpha$, the upper and lower limits should be chosen as follows:

$$\begin{aligned}\text{Upper control limit } \ell_1 &= s_c t_{\alpha/2}^*(\nu) \\ \text{Lower control limit } \ell_2 &= -s_c t_{\alpha/2}^*(\nu)\end{aligned}$$

where the standard deviation of the control is $s_c = \frac{s}{\hat{b}}$

and the critical values of the t^* distribution with ν degrees of freedom are tabulated for $m = 10$ and $m = 3$ for $\alpha = .05$ in Table 2.1 and for $\alpha = 0.01$ in Table 2.2.

Statistical control in the context of a calibration curve infers not only that measurements on a given line are repeatable within certain limits but also that the system response remains linear. Because the test is sensitive to departures from linearity, control limits based on $\alpha = 0.05$ will produce stringent control on the calibrated values; less stringent control can be achieved with control limits based on $\alpha = 0.01$.

One example of calculations for control purposes for $m=3$ and $\alpha = 0.05$ is shown in Worksheet D, and the control values are plotted in Figure 7. Note that the lines defining the lower endpoint, mid-point, and upper endpoint of the calibration curve are denoted by L , M and U respectively in the graph. This convention enables one to identify consistently non-linear behavior.

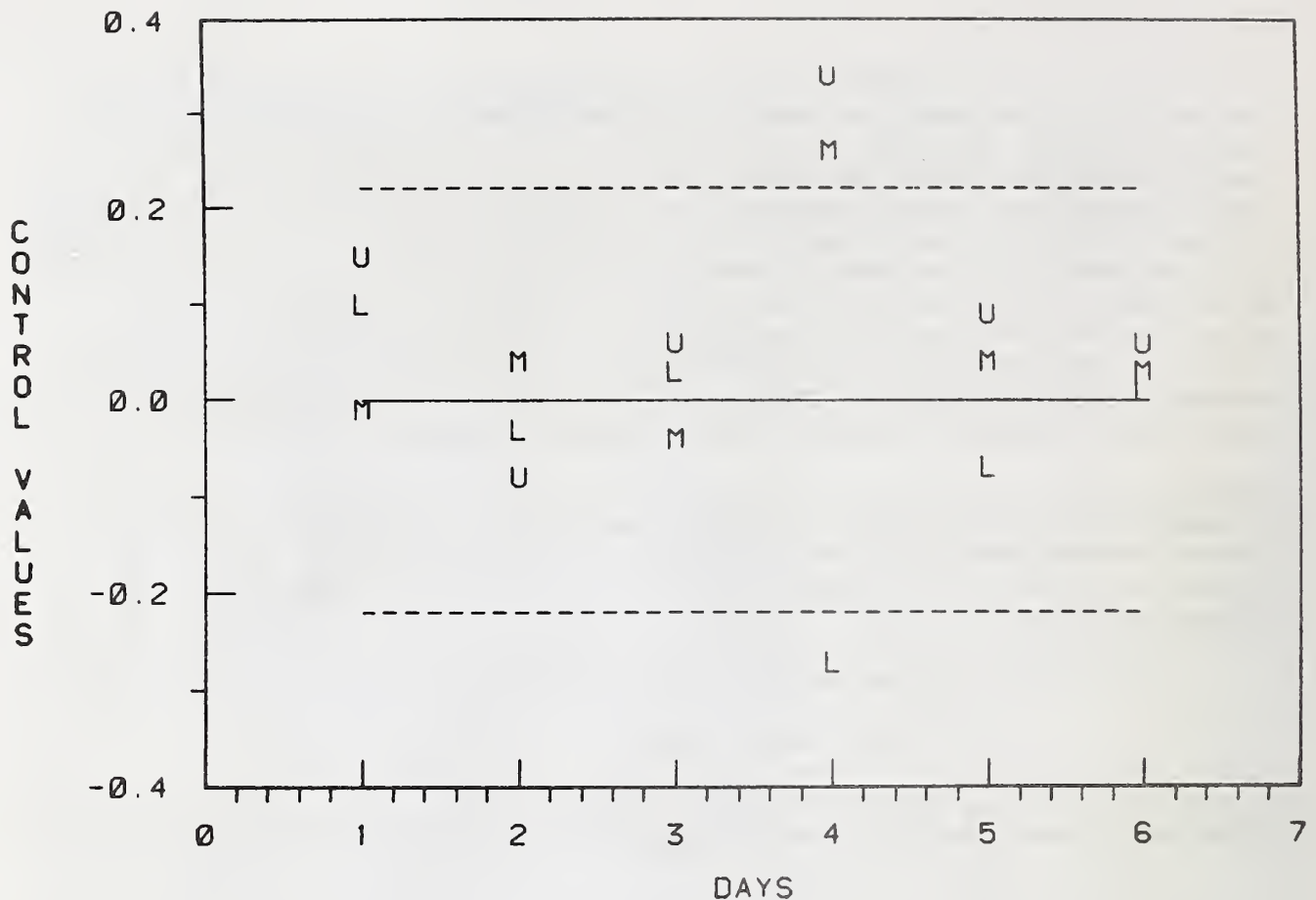


Figure 7: Control chart for opaque linewidths for data in Worksheet D. Control limits computed for significance level $\alpha = 0.05$.

The graphical method of looking for control values that are outside of the control limits is synonymous with testing each control value in the following way:

$$\text{If } v_i > \ell_1 \text{ or if } v_i < \ell_2 \text{ for any } i \quad i=1, \dots, m$$

the system is judged out-of-control. This test is easily implemented on a computer or even by hand, but the benefit derived from plotting the control data periodically should not be overlooked. The test is capable of detecting only large changes in the behavior of the system, and the plots provide additional information alerting one to a system that is gradually changing over time and providing information about the precision of the process.

Worksheet D
 Calculations for Control of Measurement Process
 at the $\alpha = 0.05$ Significance Level
 Linewidth Measurements on Opaque Lines at $m = 3$ Points
 Values in Micrometers

Repetition	NBS Value w	Observed Value z	Corrected Value z^{**}	Control v	Upper Limit ℓ_1	Lower Limit ℓ_2
1	0.76	1.12	0.86	0.10	0.17	-0.17
	3.29	3.49	3.28	-0.01	0.17	-0.17
	8.89	9.11	9.04	0.15	0.17	-0.17
2	0.76	0.99	0.73	-0.03	0.17	-0.17
	3.29	3.53	3.33	0.04	0.17	-0.17
	8.89	8.89	8.81	-0.08	0.17	-0.17
3	0.76	1.05	0.79	0.03	0.17	-0.17
	3.29	3.46	3.25	-0.04	0.17	-0.17
	8.89	9.02	8.95	0.06	0.17	-0.17
4	0.76	0.76	0.49	-0.27✓	0.17	-0.17
	3.29	3.75	3.55	0.26✓	0.17	-0.17
	8.89	9.30	9.23	0.34✓	0.17	-0.17
5	0.76	0.96	0.69	-0.07	0.17	-0.17
	3.29	3.53	3.33	0.04	0.17	-0.17
	8.89	9.05	8.98	0.09	0.17	-0.17
6	0.76	1.03	0.77	0.01	0.17	-0.17
	3.29	3.52	3.32	0.03	0.17	-0.17
	8.89	9.02	8.95	0.06	0.17	-0.17

✓ Indicates that control value is either $> \ell_1$ or $< \ell_2$.

Linewidth calibration from Worksheet B

$$\hat{a} = 0.2817 \quad \hat{b} = 0.9767$$

$$s = 0.06826$$

Correction
$$z^{**} = \frac{1}{\hat{b}} (z - \hat{a})$$

Control Values
$$v = z^{**} - w$$

Upper Control Limit
$$\ell_1 = \frac{s}{\hat{b}} t^*_{.025(38)} \quad t^*_{.025(38)} = 2.498 \text{ from Table 2.1}$$

Lower Control Limit
$$\ell_2 = -\ell_1$$

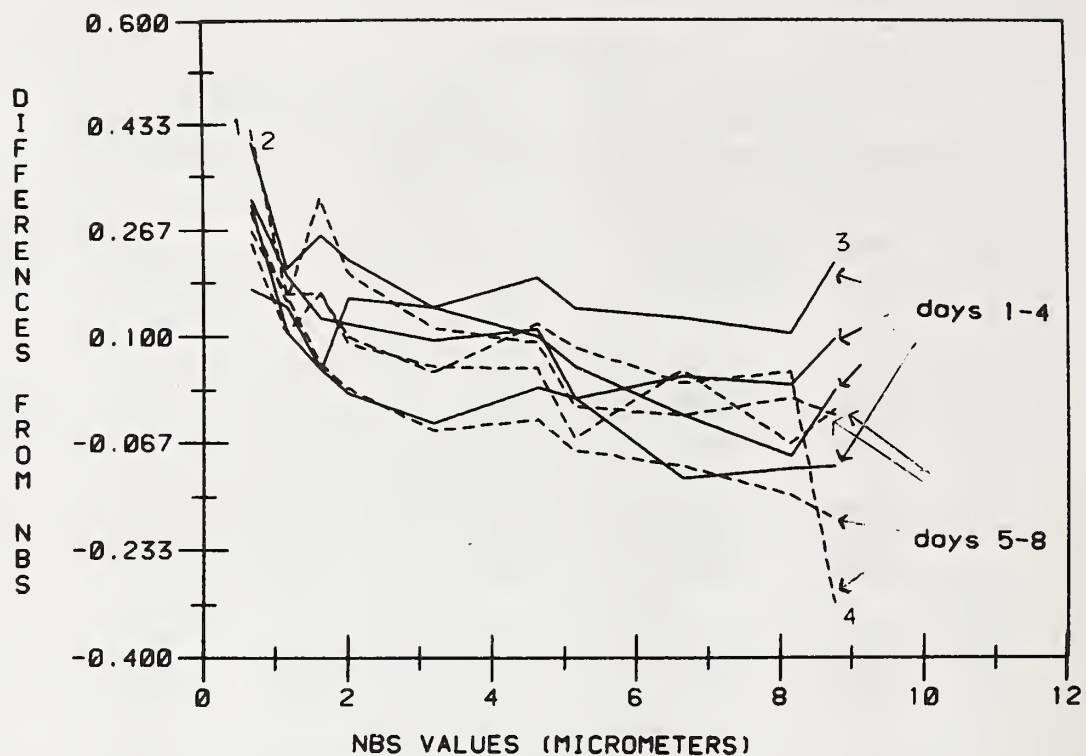


Figure 8: Differences of observed linewidths from NBS values plotted against NBS values. Differences for days 1-4 are connected by solid lines, and differences for days 5-8 are connected by dashed lines.

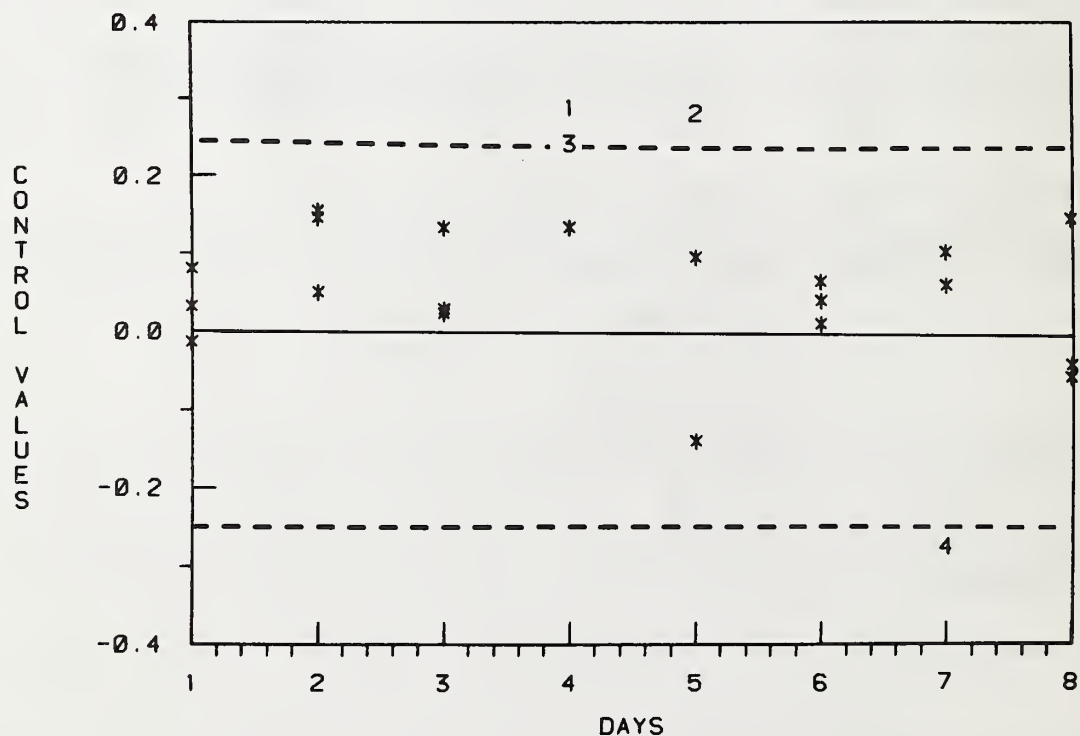


Figure 9: Control values for the lower, mid and upper endpoints of data in Figure 7. Control limits are for significance level $\alpha = 0.05$.

Another example is presented to clarify some of the ideas presented in this section. A linear calibration curve was established for an optical system using the NBS SRM, and the subsequent control measurements taken over eight days are plotted in Figure 8; measurements for each day are connected in order to demonstrate the strengths and weaknesses of a control procedure based on $m=3$ control points. Control values for the lower, mid, and upper endpoints and associated control limits were calculated for the existing calibration curve and are plotted in Figure 9.

The four data points that are outside of the control limits in Figure 9 are marked accordingly in Figure 8. Notice that the endpoints are the most sensitive to change and that point #4 is unquestionably out-of-control. But what about the other three data points that are outside of the control limits? Is it as apparent that they represent a significant change in the system? One fact is apparent from a perusal of Figure 9 that cannot be gleaned from Figure 8; i.e., almost all of the control values are above the zero line indicating that the system has drifted since the initial calibration curve was established.

Furthermore, the curves in Figure 8 raise a basic question about whether or not a linear calibration curve is adequate for this system. Significant findings for points #1 and #2 reflect a combination of non-linearity in the lower end of the system and overall drift. The question about the linearity assumption cannot be answered directly by the control procedure, but the out-of-control points flag a deficiency in the system; i.e., that the values corrected by the linear calibration curve will not meet the requirement of having negligible offset from NBS. That is precisely the question in which the user should be interested.

Finally, the system drift and multiple out-of-control conditions that are documented by the control procedure provide sufficient evidence that the derived calibration curve no longer is adequate for correcting linewidth measurements. The calibration curve should be reestablished from more recent measurements on the system, and this new calibration curve should be monitored at frequent intervals until one is satisfied that the system is no longer drifting and is operating consistently in control.

7. Can the control data be used to update the calibration curve?

If all ten lines are being measured as part of the control process, it is advantageous to keep track of the individual measured values, and periodically, at convenient intervals, to add the accumulated control data to the calibration data. If a smaller number of control measurements are made regularly, the entire calibration curve should be repeated occasionally so that the parameters can be updated.

The slope and intercept and associated estimate of random error should be revised based on this updated data set. This can be done simply by using the updated data as in Worksheet B to estimate the necessary parameters which in turn become the accepted parameters for the system and are used to correct the linewidth values until the next update. Assuming the system remains in statistical control, this will result in a smaller uncertainty for the corrected values as the calibration curve is tied down by a larger data base.

However, for some computer automated systems with limited storage capacities, it may be more convenient to save the averages of k repetitions made on each line during the calibration sequence rather than the individual measurements and to update this data base after every K succeeding control sequences. In this case the pooled standard deviations from each sequence should be combined to form the estimate of process precision. Note that a calibration curve based on averages will yield the same estimates of the slope and intercept as one based on the raw data as long as each average contains the same number of data points, but the residual standard deviation, in this case, is not an estimate of process precision.

For example, let $\bar{z}_j$ ($j=1, \dots, 10$) be the averages of k repetitions on each line and s_p the corresponding pooled standard deviation. Also let

$\bar{Z}_j$ ($j=1, \dots, 10$) be the averages of K succeeding repetitions on each line with pooled standard deviation S_p . (See equations (5.1) and (5.2) in the last section for pooled standard deviations.)

Thus the averages

$$z'_j = \frac{k\bar{z}_j + K\bar{Z}_j}{k + K} \quad j=1, \dots, 10$$

form a data base for updating the calibration curve given the model

$$(7.1) \quad z'_j = a' + b'w_j \quad j=1, \dots, 10$$

The estimates of the updated parameters (denoted by $\hat{\cdot}$) are:

$$(7.2) \quad \hat{b}' = \frac{\sum_j (z'_j - \bar{z}') (w_j - \bar{w})}{\sum_j (w_j - \bar{w})^2}$$

$$(7.3) \quad \hat{a}' = y' - \hat{b}' \bar{w}$$

The residual standard deviation of the fit with 8 degrees of freedom is

$$s' = \left(\frac{\sum_j \left(\frac{z'_j - \hat{a}' - \hat{b}' w_j}{8} \right)^2}{1} \right)^{\frac{1}{2}}$$

Although s' can be used to estimate the system precision s where

$$(7.4) \quad s = s' \sqrt{k+K}$$

the pooled standard deviation

$$s'_p = \left(\frac{10(k-1) s_p^2 + 10(K-1) S_p^2}{10(k+K-2)} \right)^{\frac{1}{2}}$$

with $10(k+K-2)$ degrees of freedom is a better estimate of process precision. These calculations are illustrated in Worksheet E.

Obviously the data base can be updated after each control sequence; i.e., for $K = 1$, but no additional information is available for computing s'_p . In this case, the only way to get an updated estimate of the process precision is via equation (7.4).

There are several possible variations on the schemes outlined above for updating the data base. The mechanics of updating the data base are usually dependent on the degree of automation that exists in the laboratory and should be carefully thought out before implementation.

Worksheet E
 Calculations for Updating the Calibration Curve
 Linewidth Measurements on Opaque Lines
 Values in Micrometers

Lines	NBS Values w	Averages of Linewidth Measurements		
		From Calibration	From Control	Updated
		$\frac{\text{Runs}}{z}$	$\frac{\text{Runs}}{Z}$	Values z'
1	2.50	2.502	2.498	2.499
2	1.94	1.978	1.983	1.981
3	0.74	0.770	0.775	0.773
4	4.25	4.278	4.277	4.277
5	10.56	10.482	10.478	10.479
6	5.29	5.360	5.360	5.360
7	3.67	3.688	3.688	3.688
8	7.45	7.408	7.409	7.409
9	1.30	1.302	1.298	1.299
10	6.14	6.082	6.077	6.079

Values $\bar{z}'$ are averages of $k=4$ calibrations from Worksheet C.

Values $\bar{Z}'$ are averages of $K=8$ control runs.

Pooled standard deviation for calibration data is

$$s_p = 0.0692 \text{ from Worksheet C}$$

Pooled standard deviation for control data is

$$S_p = 0.0610$$

Updated data base:

$$z' = \frac{k\bar{z} + K\bar{Z}}{k + K}$$

$$\bar{z}' = \Sigma z' / 10 = 4.3844 \quad \bar{w} = \Sigma w / 10 = 4.384$$

Updated calibration curve:

$$\hat{b}' = \Sigma(z' - \bar{z}') (w - \bar{w}) / \Sigma(w - \bar{w})^2 = 0.9893$$

$$\hat{a}' = \bar{z}' - \hat{b}' \bar{w} = 0.0473$$

Updated pooled standard deviation

$$s'_p = \sqrt{\frac{10(k-1)s_p^2 + 10(K-1)S_p^2}{10(k + K-2)}} = 0.0636$$

with $\gamma = 10(k + K-2) = 100$ degrees of freedom.

8. What is the uncertainty of the values corrected by the calibration curve?

Because the functional relationship between the user's system and the NBS system is not known exactly but is estimated by a series of repeated measurements, the calibration curve itself has some imprecision associated with it. Also if the measurement on a line is repeated, the resulting value will not be exactly the same; i.e., the optical system has some imprecision associated with it. This combination creates some difficulty in finding the uncertainty appropriate for the calibrated values. The difficulty arises from the fact that the initial set of measurements used to derive the calibration curve is used over and over again, and limits to error must be computed for all future corrected linewidth values based on this calibration curve.³

Tracking system performance with control data and consistently revising the linewidth calibration curve with the same control data leads to either refinement of the calibration curve or its periodic reestablishment. In either case, the control data is a connecting thread that characterizes the output of the system.

The standard deviation of a calibrated value will be largest for lines near the endpoints of the calibration interval. Therefore, it is suggested that a standard deviation be computed for the endpoints based on the control data and that it be used as a maximum for all intervening lines. Assume that there have been p control sequences each of which produced m acceptable control values, $v_{j1}, \dots, v_{jm}$ ($j=1, \dots, p$). Then the control values for the endpoints, namely v_{j1} and v_{jm} ($j=1, \dots, p$) can be used to compute

$$s_{\text{CAL}} = \left(\frac{\sum_j v_{j1}^2 + \sum_j v_{jm}^2}{2p} \right)^{1/2}$$

with $2p$ degrees of freedom. For a given line with calibrated value z^{**} , the usual confidence interval statement that $(1-\alpha)$ percent of all calibrations for that line are contained in the interval $z^{**} \pm L$ where

$$L = s_{\text{CAL}} t_{\alpha/2}(2p)$$

is approximately true for the endpoints and conservatively true for all other lines.

When sufficient data does not exist for this analysis, a tolerance type interval, called a Scheffe' interval, based on the initial calibration curve can be constructed. This calculation, although cumbersome, should be used at least for the initial uncertainty statement and can be revised after several updates of the calibration curve have been completed.

³ In cases where the calibration curve is being utilized only once, the reader is referred to a paper by C. Eisenhart (1939) "The interpretation of certain regression methods and their use in biological and industrial research," Annals of Math Stat, 10, pp. 162-202.

The approach developed by Scheffe' [14] guarantees that a large percentage of statements that are made about future calibrations are correct. The statements refer to limits to error which are appropriate for the corrected values. For example, these limits L can be constructed such that the probability is > 0.99 that 95 percent of all intervals $z^{**} \mp L$ so constructed contain the linewidth value that is properly related to NBS where z^{**} is a corrected linewidth measurement.

The Scheffe' procedure is conservative and will yield fairly wide intervals depending on the choice of α and δ . The statement that the probability is $1-\delta$ that $(1-\alpha)$ percent of the intervals so constructed are correct should be defined according to the user's requirements. Scheffe' suggests that δ be chosen smaller than α , and a combination of $\alpha = 0.10$ and $\delta = 0.05$ may be appropriate for most situations. More conservative limits are given by a choice of $\alpha = 0.05$ and $\delta = 0.01$. Tables are provided to accommodate both sets of statements.

The calculation of the limits produces upper and lower curves bounding the calibration curve resulting in upper and lower limits for each nominal linewidth. The upper and lower limits are not necessarily symmetrical, but were sufficiently close for the data in the interlaboratory study that only the procedure for calculating the upper limits is given here. For example, the following limits are calculated for the linewidth calibration curve derived in Worksheet C for $\alpha = 0.05$ and $\delta = 0.01$.

Nominal Linewidth	Values in Micrometers	
	Limits to Error	
	Upper L	Lower L
1	0.25	-0.26
2	0.24	-0.25
3	0.24	-0.24
4	0.23	-0.23
5	0.23	-0.23
6	0.24	-0.24
7	0.25	-0.24
8	0.26	-0.25
9	0.27	-0.26
10	0.28	-0.27

The maximum of these limits to error occurs at one of the endpoints of the calibration interval and is appropriate for use in the uncertainty statement. The steps necessary for computing the maximum limit to error L based on reference [14] are given on the next page, and it is noted that the calculation should not be extended to linewidths outside of the calibration interval. The steps outlined assume that the calibration curve is linear and that the estimate of precision has $n-2$ degrees of freedom.

Choose probabilities α and δ .

Look up $Z_{\alpha/2}$ the upper $\alpha/2$ percentage point of the normal distribution. $Z_{\alpha/2} = 1.96$ for $\alpha = 0.05$
 $Z_{\alpha/2} = 1.6449$ for $\alpha = 0.10$

$\chi^2_{1-\delta}(v)$ The lower δ -percentage point of the chi-square distribution with v degrees of freedom. Table 3.1 gives values of $\chi^2_{1-\delta}(v)$ for $\delta = .01$ and $v = 30(2)120$.
 and Table 3.2 gives values for $\delta = 0.05$.

$F_{\delta}(2, v)$ is the upper δ -percentage point of the F distribution with 2 degrees of freedom in the numerator and v degrees of freedom in denominator ($v = n-2$). Table 4.1 gives values of $F_{\delta}(2, v)$ for $\delta = .01$ and $v = 30(2)120$, and Table 4.2 gives values for $\delta = 0.05$.

Compute

$$C_1 = C_3 Z_{\alpha/2} \sqrt{v} \left(\chi^2_{1-\delta}(v) \right)^{-1/2} \quad C_2 = C_3 \sqrt{2 F_{\delta}(p, v)}$$

The value of C_3 depends on several parameters and ranges between 0.95 and 1.05 for $\delta=.01$ and $n=40$. The exact value of C_3 which is appropriate for any data set can be found using methods given in reference [5]. The calculation of L is not overly sensitive to C_3 . For the interlaboratory study C_3 was taken to be 1.05, and this value is suggested as a conservative estimate of C_3 and is used in the sample calculation in Worksheet G.

Find the least-squares estimates $\hat{a}$, $\hat{b}$ and s for the calibration curve

$$z_{ij} = a + bw_{ij} \quad i=1, \dots, k; j=1, \dots, 10$$

and choose $w_{\max}$ to be the maximum linewidth of interest.

Compute

$$C = \hat{b}^2 - (C_2 s_b)^2$$

$$D = w_{\max} - \hat{a} - \hat{b}\bar{w} + C_1 s$$

where $\bar{w}$ is the average of the NBS values. The limit L is given by

$$L = \bar{w} - w' + \frac{\hat{b}D + C_2 \left(\frac{s^2 C}{n} + D^2 s_b^2 \right)^{1/2}}{C} \quad \text{where } w' = \frac{w_{\max} - \hat{a}}{\hat{b}}$$

The computations for the linewidth calibration curve derived in Worksheet C for $\alpha = .05$ and $\delta = .01$ are shown in Worksheet F.

Finally, the uncertainty that is appropriate for a corrected linewidth value is $\pm U$ where $U = L + |\Delta_{\text{NBS}}|$ and Δ_{NBS} is the systematic error from the NBS system.

Worksheet F
Calculation of Limits to Error L for Opaque Linewidth Values
Corrected by a Linear Calibration Curve
Values in Micrometers

Intercept $\hat{a}$	0.2817
Slope $\hat{b}$	0.9767
Standard deviation of intercept s_a	0.01955
Standard deviation of slope s_b	0.003717
Standard deviation of linear fit s	0.06826
Degrees of freedom v	38
Mean of NBS values $\bar{w}$	4.384

Choose $\alpha=.05$, $\delta=.01$
Look up in tables:

$Z_{.025} = 1.96$ the upper 2.5% point of the normal distribution

$\chi^2_{.99}(v) = 20.6914$ the lower 1% point of the χ^2 distribution
with v degrees of freedom

$F_{.01}(2, v) = 5.2112$ the upper 1% point of the F distribution with 2
and v degrees of freedom

Compute:

$$C_1 = 1.05 Z_{.025} \sqrt{v} / \sqrt{\chi^2_{.99}(v)} = 2.795 \quad C_2 = 1.05 \sqrt{2F_{.01}(2, v)} = 3.3898$$

Choose $w_{\max} = 10$ and compute

$$C = \hat{b}^2 - (C_2 s_b)^2 = 0.9794$$

$$D = w_{\max} - \hat{a} - \hat{b} \bar{w} + C_1 s = 5.8144$$

$$w' = \frac{w_{\max} - \hat{a}}{\hat{b}} = 10.1520$$

$$\bar{w} = \sum w_i / n = 4.384$$

$$L = \bar{w} - w' + \frac{\hat{b} D + C_2 \sqrt{\frac{C s^2}{n} + D^2 s_b^2}}{C} = 0.28$$

The maximum limit to error that is appropriate for any corrected opaque linewidth in the 1-10 μ m interval is $L = 0.28\mu$ m.

Table 1
Critical Values of Student's t Distribution
for a Two-Sided Test
Significance Level $\alpha = 0.05$

ν	$t_{.025}(\nu)$	ν	$t_{.025}(\nu)$
30	2.042	76	1.992
32	2.037	78	1.991
34	2.032	80	1.990
36	2.028	82	1.989
38	2.024	84	1.989
40	2.021	86	1.988
42	2.018	88	1.987
44	2.015	90	1.987
46	2.013	92	1.986
48	2.011	94	1.985
50	2.009	96	1.985
52	2.007	98	1.985
54	2.005	100	1.984
56	2.003	102	1.984
58	2.002	104	1.983
60	2.000	106	1.983
62	1.999	108	1.982
64	1.998	110	1.982
66	1.997	112	1.981
68	1.995	114	1.981
70	1.994	116	1.981
72	1.993	118	1.980
74	1.993	120	1.980

ν is the number of degrees of freedom in the estimate of process precision.

Table 2.1
Critical Values of t^* distribution
for controlling the output of a calibration curve
at m points
Significance level $\alpha = 0.05$

ν	$t_{.025}^*(\nu)$		γ	$t_{.025}^*(\nu)$	
	$m=3$	$m=10$		$m=3$	$m=10$
30	2.528	3.021	76	2.442	2.883
32	2.519	3.006	78	2.440	2.881
34	2.511	2.993	80	2.439	2.879
36	2.504	2.982	82	2.437	2.877
38	2.498	2.972	84	2.436	2.875
40	2.492	2.963	86	2.435	2.873
42	2.487	2.954	88	2.434	2.872
44	2.482	2.947	90	2.433	2.870
46	2.478	2.940	92	2.432	2.868
48	2.474	2.934	94	2.431	2.867
50	2.470	2.929	96	2.430	2.866
52	2.467	2.923	98	2.429	2.864
54	2.464	2.919	100	2.428	2.863
56	2.461	2.914	102	2.428	2.861
58	2.459	2.910	104	2.427	2.860
60	2.456	2.906	106	2.426	2.859
62	2.454	2.903	108	2.425	2.858
64	2.452	2.900	110	2.425	2.857
66	2.450	2.896	112	2.424	2.856
68	2.448	2.893	114	2.423	2.855
70	2.446	2.891	116	2.423	2.854
72	2.444	2.888	118	2.422	2.853
74	2.443	2.886	120	2.421	2.852

Critical values $t_{\alpha/2}^*(\nu)$ correspond to the upper ζ percent point of student's t distribution where

$$\zeta = 1/2 \left\{ 1 - \exp \left(\frac{\ln(1-\alpha)}{m} \right) \right\}$$

and ν is the number of degrees of freedom in the estimate of process precision.

Table 2.2
Critical values of t^* distribution
for controlling the output of a calibration curve
at m points
Significance level $\alpha = 0.01$

ν	$t_{.005(\nu)}^*$		ν	$t_{.005(\nu)}^*$	
	$m=3$	$m=10$		$m=3$	$m=10$
30	3.187	3.644	76	3.030	3.422
32	3.171	3.620	78	3.027	3.418
34	3.156	3.599	80	3.025	3.415
36	3.143	3.581	82	3.023	3.412
38	3.131	3.564	84	3.020	3.409
40	3.121	3.549	86	3.018	3.406
42	3.111	3.536	88	3.016	3.403
44	3.103	3.524	90	3.014	3.400
46	3.095	3.513	92	3.013	3.398
48	3.088	3.504	94	3.011	3.396
50	3.082	3.495	96	3.009	3.393
52	3.076	3.486	98	3.008	3.391
54	3.070	3.479	100	3.006	3.389
56	3.065	3.471	102	3.005	3.387
58	3.060	3.465	104	3.003	3.385
60	3.056	3.459	106	3.002	3.383
62	3.052	3.453	108	3.001	3.382
64	3.048	3.448	110	3.000	3.380
66	3.045	3.443	112	2.998	3.378
68	3.041	3.438	114	2.997	3.377
70	3.038	3.434	116	2.996	3.375
72	3.035	3.429	118	2.995	3.373
74	3.032	3.426	120	2.994	3.372

Critical values $t_{\alpha/2(\nu)}^*$ correspond to the upper ζ percent point of student's t distribution where

$$\zeta = 1/2 \left\{ 1 - \exp \left(\frac{\ln(1-\alpha)}{m} \right) \right\}$$

and ν is the number of degrees of freedom in the estimate of process precision.

Table 3.1
Percentage points of the χ^2 distribution
Lower 1% points

ν	$\chi^2_{.99}(\nu)$	ν	$\chi^2_{.99}(\nu)$
30	14.953	76	50.286
32	16.362	78	51.910
34	17.789	80	53.540
36	19.233	82	55.174
38	20.691	84	56.813
40	22.164	86	58.456
42	23.650	88	60.103
44	25.148	90	61.754
46	26.657	92	63.409
48	28.177	94	65.068
50	29.707	96	66.730
52	31.246	98	68.396
54	32.793	100	70.065
56	34.350	102	71.737
58	35.913	104	73.413
60	37.485	106	75.092
62	39.063	108	76.774
64	40.649	110	78.458
66	42.240	112	80.146
68	43.838	114	81.836
70	45.442	116	83.529
72	47.051	118	85.225
74	48.666	120	86.923

ν is the number of degrees of freedom in the estimate of process precision.

Table 3.2
Percentage points of the χ^2 distribution
Lower 5% points

ν	$\chi^2_{.95}(\nu)$	ν	$\chi^2_{.95}(\nu)$
30	18.493	76	56.920
32	20.072	78	58.654
34	21.664	80	60.391
36	23.269	82	62.132
38	24.884	84	63.876
40	26.509	86	65.623
42	28.144	88	67.373
44	29.787	90	69.126
46	31.439	92	70.882
48	33.098	94	72.640
50	34.764	96	74.401
52	36.437	98	76.164
54	38.116	100	77.929
56	39.801	102	79.697
58	41.492	104	81.468
60	43.188	106	83.240
62	44.889	108	85.015
64	46.595	110	86.792
66	48.305	112	88.570
68	50.020	114	90.351
70	51.739	116	92.134
72	53.462	118	93.918
74	55.189	120	95.705

ν is the number of degrees of freedom in the estimate of process precision.

Table 4.1
Percentage points of the F distribution
Upper 1% points

ν	$F_{.01}(2, \nu)$	ν	$F_{.01}(2, \nu)$
30	5.390	76	4.896
32	5.336	78	4.888
34	5.289	80	4.881
36	5.248	82	4.874
38	5.211	84	4.867
40	5.178	86	4.861
42	5.149	88	4.855
44	5.123	90	4.849
46	5.099	92	4.844
48	5.077	94	4.838
50	5.057	96	4.833
52	5.038	98	4.828
54	5.021	100	4.824
56	5.006	102	4.820
58	4.991	104	4.815
60	4.977	106	4.811
62	4.965	108	4.807
64	4.953	110	4.803
66	4.942	112	4.800
68	4.932	114	4.796
70	4.922	116	4.793
72	4.913	118	4.790
74	4.904	120	4.786

ν is the number of degrees of freedom in the estimate of process precision.

Table 4.2
Percentage points of the F distribution
Upper 5% points

ν	$F_{.05}(2, \nu)$	ν	$F_{.05}(2, \nu)$
30	3.316	76	3.117
32	3.294	78	3.114
34	3.276	80	3.111
36	3.259	82	3.108
38	3.245	84	3.105
40	3.232	86	3.103
42	3.220	88	3.100
44	3.209	90	3.098
46	3.200	92	3.095
48	3.191	94	3.093
50	3.183	96	3.091
52	3.175	98	3.089
54	3.168	100	3.087
56	3.162	102	3.086
58	3.156	104	3.084
60	3.150	106	3.082
62	3.145	108	3.080
64	3.140	110	3.079
66	3.136	112	3.077
68	3.132	114	3.076
70	3.128	116	3.074
72	3.124	118	3.073
74	3.120	120	3.072

ν is the number of degrees of freedom in the estimate of process precision.

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APPENDIX

I. Test of Linearity Assumption.

The test of whether or not a linear function of the measurements constitutes an appropriate calibration curve is based on the information in the repetitions [15]. The fitted value for each line

$$\hat{z}_j = \hat{a} + b\hat{w}_j \quad j=1, \dots, 10$$

is compared to the average of the repetitions for each line; namely,

$$\bar{z}_j = \frac{\sum_{i=1}^k z_{ij}}{k} \quad j=1, \dots, 10$$

where k is the number of repetitions of each linewidth.

Close agreement between the fitted curve and these averages is evidence that the hypothesis is correct. The test is based on an F statistic, and the assumption of linearity is rejected if

$$F = \frac{10(k-1)}{8} \cdot \frac{SSL - SSR}{SSR} > F_{.01}(8, 10(k-1))$$

where

$$SSL = \sum_{j=1}^{10} \sum_{i=1}^k (z_{ij} - \hat{z}_j)^2$$

$$SSR = \sum_{j=1}^{10} \sum_{i=1}^k (z_{ij} - \bar{z}_j)^2$$

and $F_{.01}(8, 10(k-1))$ is the upper one percent point of the F distribution with eight degrees of freedom in the numerator and $10(k-1)$ degrees of freedom in the denominator.

II. Test for Differences Among Repetitions.

The test of whether or not a system with linear response is changing from repetition to repetition is based on the following model; namely,

$$z_{ij} = \alpha_i + \beta_i x_{ij} \quad \begin{matrix} i=1, \dots, k \\ j=1, \dots, 10 \end{matrix}$$

where

$$x_{ij} = w_{ij} - \bar{w}_i$$

and

$$\bar{w}_i = \frac{\sum_j w_{ij}}{10}$$

Estimates of the intercepts α_i and the slopes β_i and corresponding standard deviations are computed for each repetition.

$$\hat{\beta}_i = \frac{\sum_j (x_{ij} - \bar{x}_i)(z_{ij} - \bar{z}_i)}{\sum_j (x_{ij} - \bar{x}_i)^2}, \quad i=1, \dots, k$$

$$\hat{\alpha}_i = \bar{z}_i - \hat{\beta}_i \bar{x}_i$$

$$\bar{x}_i = \frac{\sum_j x_{ij}}{10} \quad \bar{z}_i = \frac{\sum_j z_{ij}}{10}$$

$$s_{\alpha_i} = s \left(\frac{\frac{\sum_j x_{ij}^2}{10}}{\sum_j (x_{ij} - \bar{x}_i)^2} \right)^{1/2}$$

$$s_{\beta_i} = \frac{s}{\left(\sum_j (x_{ij} - \bar{x}_i)^2 \right)^{1/2}}$$

and

$$s_1 = \left(\frac{\sum_j (z_{ij} - \hat{\alpha}_i - \hat{\beta}_i x_{ij})^2}{8} \right)^{1/2}$$

The test statistic is

$$F = \frac{1}{2(k-1)} \left\{ \frac{\sum_i (\hat{\alpha}_i - \bar{\alpha})^2}{\frac{1}{k} \sum_i s_{\alpha_i}^2} + \frac{\sum_i (\hat{\beta}_i - \bar{\beta})^2}{\frac{1}{k} \sum_i s_{\beta_i}^2} \right\}$$

where

$$\bar{\alpha} = \frac{1}{k} \sum_i \hat{\alpha}_i \quad \bar{\beta} = \frac{1}{k} \sum_i \hat{\beta}_i$$

It is concluded that the system is not consistent from repetition to repetition if

$$F > F_{.01} (2(k-1), 8k)$$

where $F_{.01} (2(k-1), 8k)$ is the upper one percent point of the F distribution with $2(k-1)$ degrees of freedom in the numerator and $8k$ degrees of freedom in the denominator.

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National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NBS under the authority of the National Standard Data Act (Public Law 90-396).

NOTE: The principal publication outlet for the foregoing data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

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