



Designation: F 398 – 92 (Reapproved 2002)

Standard Test Method for Majority Carrier Concentration in Semiconductors by Measurement of Wavenumber or Wavelength of the Plasma Resonance Minimum¹

This standard is issued under the fixed designation F 398; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

As originally published, this test method was written for infrared instruments with output in units of wavelength. Calibration data, as included, were based on infrared measurements in units of wavelength and on a combination of concentration scale methods including Hall effect, neutron activation analysis, and four-probe resistivity. These latter measurements were converted, using the empirical data of Irvin, from resistivity to “impurity concentration.” The result of this procedure is calibration data that is a mixture of carrier concentration and impurity concentration values. While the differences between them is large only at very high concentrations, there are values in the original calibration plots for silicon (Fig. A1.1 and Fig. A1.2) that are above currently accepted solid-solubility limit values.

Common practice current infrared spectrophotometers is to give output in units of wavenumbers (cm^{-1}). In order to relate this test method and its original calibration to current infrared units without introducing numerical error (the calibration relation is not analytically invertible to wavenumbers), the direct substitution of $10\,000/\text{wavenumber}$ for wavelength is given in the analysis equation and in the tables of coefficients.

This test method is not believed to have wide current use in the semiconductor industry. However, because this test method may be useful for some applications and because the calibration data contained herein is believed to be available nowhere else in the archival literature, this test method is being retained as a standard.

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1. Scope

1.1 This test method covers determination of the wavenumber of the plasma resonance minimum in the infrared reflectance of a doped semiconductor specimen, from which the majority carrier concentration can be obtained.

1.2 This test method of determination of the wavenumber minimum is nondestructive and contactless. It is applicable to *n*- and *p*-type silicon, *n*- and *p*-type gallium arsenide, and *n*-type germanium.

1.3 This test method gives a relative measurement in that the relation between the wavenumber of the plasma resonance minimum and the majority carrier concentration is empirical. Such relations have been established for the several cases summarized in Annex A1. These relations are based upon

determinations of the plasma resonance minimum by the procedure of this method and determinations of the Hall coefficient according to Test Methods F 76 (Section 2) or resistivity according to Test Methods F 43 or Test Method F 84 (Section 2) as appropriate.

1.4 These relations have been established over a majority carrier concentration range from 1.5×10^{18} to 1.5×10^{21} cm^{-3} for *n*-type silicon, from 3×10^{18} to 5×10^{20} for *p*-type silicon, from 3×10^{18} to 7×10^{19} for *n*-type germanium, from 1.5×10^{17} to 1×10^{19} for *n*-type gallium arsenide, and from 2.6×10^{18} to 1.3×10^{20} cm^{-3} for *p*-type gallium arsenide.

1.5 These relations can be extended or developed for other materials by measuring the wavelength of the plasma resonance minimum according to this procedure on specimens whose majority carrier concentration has been determined by other means.

1.6 This test method is applicable to both bulk and diffused material. However, since there is some controversy over the effects of variations of junction depth on the measurement, it should be applied to surface concentration measurements on

¹ This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

Current edition approved May 15, 1992. Published July 1992. Originally published as F 398 – 74 T. Last previous edition F 398 – 87 ^{ε1}.

shallow (1 μm or less) diffusions only on a relative basis unless there is experimental corroboration of the results under the conditions of interest.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers²
- F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials³
- F 43 Test Methods for Resistivity of Semiconductor Materials³
- F 76 Test Methods for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Single-Crystal Semiconductors³
- F 84 Test Method for Measuring Resistivity of Silicon Slices with an In-Line Four-Point Probe³

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *plasma resonance*—plasma resonance is the transition between low-frequency and high-frequency reflectivity conditions in an extrinsic semiconductor in which the mobile carriers and the immobile ionized impurities can be considered as a plasma. At high frequencies or short wavelengths, the fields are varying too fast for the plasma to respond and the reflectivity approaches that of intrinsic silicon. At low frequencies or long wavelengths, the fields vary slowly so that the plasma may respond and the reflectivity is high.

3.1.2 *plasma resonance minimum*—this is the minimum in reflectivity as a function of wavenumber associated with plasma resonance.

4. Summary of Test Method

4.1 The majority carrier concentration of a semiconductor is measured by determining the reflectivity of the semiconductor as a function of wavenumber in the infrared region of the spectrum. This reflectivity has a high-wavenumber value which is almost independent of carrier concentration and is characteristic of the intrinsic semiconductor lattice. As the wavenumber decreases, the reflectivity decreases to a minimum at a wavenumber, ν_{PR} which is characteristic of the number of free carriers in the semiconductor. For decreasing wavenumbers, beyond ν_{PR} , the reflectivity increases rapidly, approaching a value of unity.

4.2 Carrier concentration in a specimen of unknown doping level but known type and composition, for example, *n*-type silicon, can be determined by measuring ν_{PR} and relating this to the carrier concentration through an appropriate empirical relationship.

5. Significance and Use

5.1 The measurement of the carrier concentration of a semiconductor is a vital part of the development and manufacture of semiconductor devices and integrated circuits.

5.2 An important use of this technique is the measurement of the surface concentration of diffused layers. There is, however, some controversy about the effect of variation of junction depth on the correspondence between the concentration measured by this technique and the true concentration, particularly for very shallow (1 μm or less) junctions.

5.3 This technique also may be applied to the measurement of bulk materials and to the study of nonuniformities across the surface of those materials. For such application the user should be certain that the surfaces are free of the films which normally appear on an as-grown crystal.

5.4 It must be noted that the reflectivity minimum measured by this procedure and associated with the plasma resonance occurs at a larger wavenumber value than that associated with the actual plasma resonance. The latter occurs at a wavenumber value which can be calculated from the equation:

$$\left(\frac{1}{\nu_p}\right)^2 = \lambda_p^2 = (2\pi c)^2 m^* \epsilon / N e^2$$

where:

- ν_p = wavenumber value of plasma resonance, m^{-1} ,
- λ_p = wavelength of plasma resonance, m ,
- c = speed of light, m/s ,
- m^* = effective mass of free carriers, kg ,
- ϵ = low frequency dielectric constant, F/m ,
- N = density of free carriers, m^{-3} , as measured by this method, and
- e = electronic charge, C .

6. Interferences

6.1 Highly polished surfaces can be obtained over highly damaged regions; this can cause misleading results. Users of this method should, therefore, assure themselves that the polishing technique that they are using yields damage-free surfaces in order to be certain that the measurement obtained using this technique represents the bulk properties of the materials.

NOTE 1—Pending establishment of a method of test for surface damage, the user may check for surface damage by successively measuring the reflectivity spectrum of the specimen and chemically removing a layer from the surface using a non-preferential etchant until the plasma resonance readings from successive measurements agree.

6.2 Since specimens may be inhomogeneous, users of this technique for a referee method should agree on the location on each specimen where the measurement is to be taken.

6.3 Anomalously low mobility material may yield a measurement of concentration which does not agree with that obtained by other techniques.

6.4 GaAs has a sharp reflection minimum due to lattice reflection (Reststrahlen Band) centered at 302 cm^{-1} . Therefore measurements of *p* and *n*-type GaAs near the lower limit of this test method should be interpreted carefully so that the Reststrahlen minimum is not mistaken for a plasma resonance minimum.

² Annual Book of ASTM Standards, Vol 03.06.

³ Annual Book of ASTM Standards, Vol 10.05.

7. Apparatus

7.1 *Spectrophotometer, Dual-Beam*, with either wavelength or a wavenumber presentation, under continuous dry-nitrogen or dry-air purge, and with the following additional characteristics:

7.1.1 Wavenumber range from 5000 cm^{-1} to 25 cm^{-1} . Wavelength range from 2 to 40 μm . If the range of values is smaller, the range of impurity concentrations covered by the method is reduced from that stated in 1.2.

7.1.2 Provision for up to a ten-times scale expansion of the reflectance measurement can be necessary for some materials.

7.1.3 Wavelength reproducibility of at least $\pm 0.05\text{ }\mu\text{m}$ as determined according to Practice E 275.

7.1.4 Spectral resolution of 2 cm^{-1} or smaller at 1000 cm^{-1} .

7.2 *Reflectance Accessory*, compatible with the spectrophotometer. This accessory must provide an angle of incidence upon the specimen of 30° or less. Additionally, there shall be either an identical or symmetric accessory for the reference beam providing equal path lengths in both beams, or an attenuator for the reference beam. Whether a reflectance attachment or an attenuator is used in the reference beam, the combination of specimen reflectance attachment and reference beam attachment must provide a 100% trace varying from a straight line by not more than 8% of a full scale (see 9.2).

NOTE 2—While good optical practice would require the use of reflectance accessories in each beam so designed and installed as to maintain the identical path length nature of a dual beam spectrophotometer, the data from the round robin indicate that the use of an attenuator in the reference beam does not deteriorate the interlaboratory precision of the measurement. Therefore the use of a matched pair of reflectance accessories is not required but is recommended.

7.3 *Aluminum Mirror*, optical quality, scratch-free, front-surface, for each reflectance attachment to be used.

8. Test Specimens

8.1 The surface of the test specimen must be smooth and highly reflecting, giving the appearance of a mirror, as is normally used for semiconductor processing.

8.2 The polishing technique that is used to prepare the surface of the test specimen must minimize subsurface damage (Note 1).

9. Calibration

9.1 *Spectrophotometer*:

9.1.1 Refer to the instruction manual for the details of operation of the instrument to be used.

9.1.2 Determine that the wavelength accuracy and repeatability are within the specifications for the instrument in accordance with Practice E 275.

NOTE 3—It is advised that a service representative of the manufacturer be contacted if the accuracy and repeatability determined above are outside the manufacturer's specifications.

9.2 *Reflectance Accessory*:

9.2.1 With the accessories to be used for reflectance measurements installed in each beam and with a front surface mirror on each reflectance attachment used, displace the scale towards zero by adjusting the zero control or some other control which does not change the 0 to 100% span. Run a double-beam 100% reflectance trace (Fig. 1).

9.2.2 The condition of the instrument is not acceptable for this method if this trace varies by more than 8 percentage points, peak to peak.

9.2.3 If the trace varies by more than this amount run a 100% trace in double-beam transmission operation.

9.2.4 If this trace varies by more than an acceptable value as stated by the manufacturer of the spectrometer, the spectrometer is not operating properly and must be readjusted.

9.2.5 If this trace is within the manufacturer's specification the reflectance attachments are the cause of the error and either they should be carefully aligned or the manufacturer contacted.

10. Procedure

10.1 If the conductivity type of the specimen is unknown determine it at a convenient time before interpretation of the measurement, according to Test Methods F 76 or F 42.

10.2 Measure the reflectance of the specimen as a function of wavenumber (Fig. 2, Trace 1). Scan speed must be slow

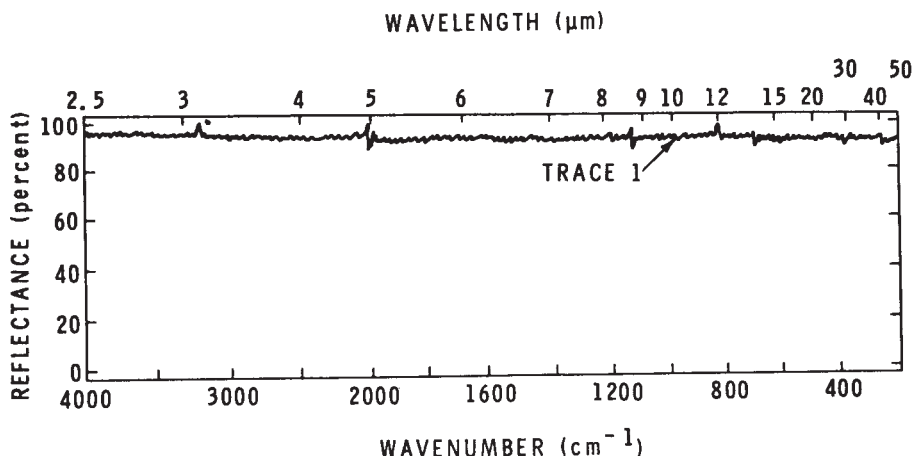
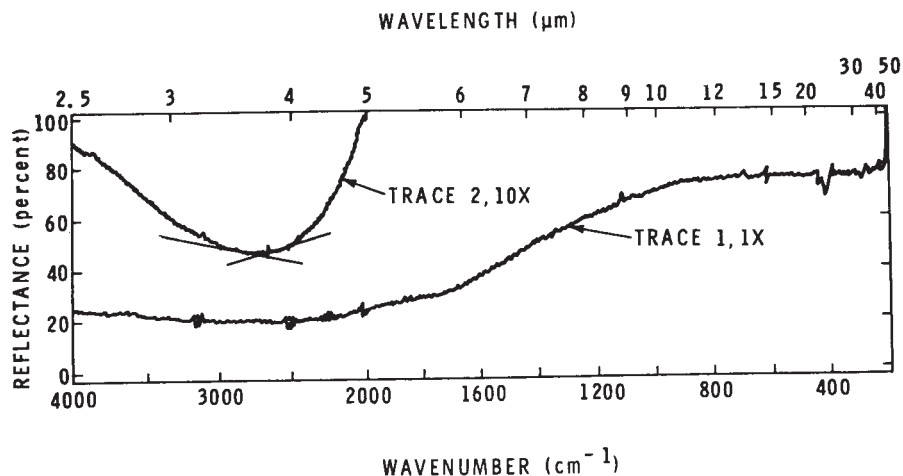


FIG. 1 Displaced 100% Double Beam Reflectance Curve with Front Surface Aluminum Mirrors on the Reflectance Attachments in Each Beam

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NOTE 1—The instrument on which this spectrum was obtained provided a bilinear presentation in wavenumber, with a scale change at 2000 cm^{-1} .

FIG. 2 Reflectance as a Function of Wavenumber for an *n*-type Silicon Specimen with a Carrier Concentration of $2.7 \times 10^{20}\text{ cm}^{-3}$

enough to allow the instrument to meet the requirements of 7.1.3 and 7.1.4. If a minimum is not observed, the specimen is not within the range of applicability of this test method.

10.3 Expand the reflectance scale if the difference between the reflectance at the minimum and the greatest reflectance on either side of the minimum is less than 10 percentage points in the reflection scale, and remeasure the reflectance as a function of wavenumber (Fig. 2).

NOTE 4—Care should be taken in aligning the instrument to minimize discontinuities in regions which would effect the determination of the minimum. These usually occur at filter or grating changes, and are amplified by scale expansion. Such a discontinuity is shown in Fig. 3, Trace 2.

10.4 Using a transparent straightedge, fit the short-wavelength portion of the reflectivity curve with a straight line whose nearest point of tangency or intersection to the reflectivity curve is no farther than 100 cm^{-1} from the approximate position of the reflectivity minimum. Use as much of the reflectivity curve as possible and still meet this condition (Fig. 2, Trace 2).

10.5 Using a transparent straightedge, fit the low wavenumber portion of the reflectivity curve with a straight line whose slope is the negative of that of the line in 10.4 (Fig. 2, Trace 2).

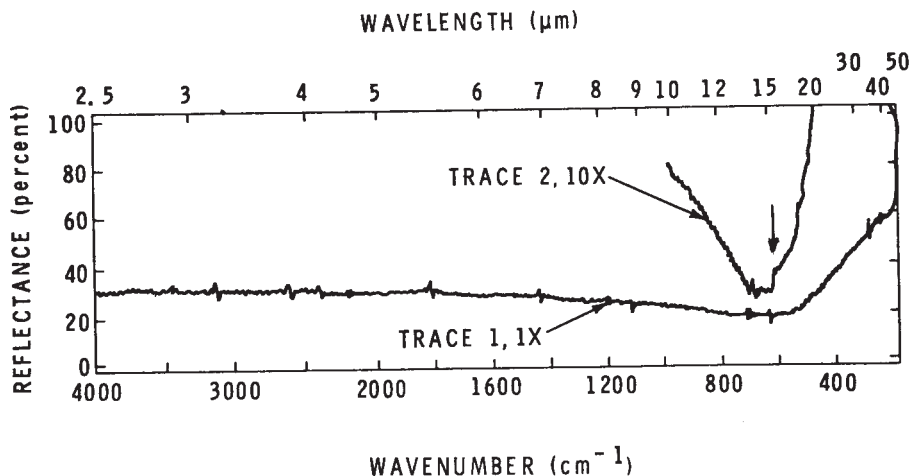
10.6 Determine the position of the minimum in reflectivity at the intersection of these two straight lines.

11. Calculation

11.1 Calculation


11.1.2 Calculate the impurity concentration from the measured wavelength minimum according to the equation:

$$N = [A \lambda_{PR} + C]^B = [10\ 000 A/v_{PR} + C]^B$$



NOTE 1—Traces showing sharp discontinuities must not be analyzed (Note 3).

FIG. 3 Reflectance as a Function of Wavenumber for an *n*-type Silicon Specimen, Showing a Sharp Discontinuity in Trace 2 due to a Grating Change (arrow)

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where:

- ν_{PR} = wavenumber value of plasma resonance minimum (cm^{-1}),
- N = majority carrier concentration, cm^{-3} ,
- λ_{PR} = wavelength of plasma resonance minimum, μm , and
- A, B, C = constants listed in Table 1.

Use the constant appropriate to the conductivity type and material of the specimens .

12. Report

12.1 Report the following information:

- 12.1.1 Material,
- 12.1.2 Conductivity type,
- 12.1.3 Wavenumber position of reflectivity minimum,
- 12.1.4 Carrier concentration,
- 12.1.5 Instrument used, and
- 12.1.6 Diagram showing location on specimen at which measurement was taken.

13. Precision and Bias

13.1 The relative two-sigma precision (R2S) of this measurement when used on an interlaboratory basis on *n*- and *p*-type silicon and on *n*-type gallium arsenide is given in Figs. 4-6. These precisions are based upon results reported by 10 laboratories for measurements taken on each of 10 samples of *n*-type silicon and 10 samples of *p*-type silicon, and on results reported by 7 laboratories for measurements taken on each of 8 samples of *n*-type gallium arsenide. Both wavelength and wavenumber presentations were represented in the test. The wavelength precisions were obtained from a least squares fit to the reported wavelength of the plasma resonance minimum, λ_{PR} , in micrometres. The concentration precisions were computed from a least squares fit to the deviations in concentration

above and below a nominal value of concentration, in cm^{-3} , computed from wavelengths two standard deviations below and above, respectively, the nominal wavelength values, using the appropriate equation from Table 1. The deviations in concentration above and below a nominal value for identical deviations in wavelength below and above, respectively, the corresponding value of wavelength differ because of nonlinear relationship between concentration wavelength. While the method has not been tested for *n*-type germanium or for other semiconductor materials, the precision for those materials is expected to be comparable to that obtained on the tested materials for concentration ranges yielding spectra comparable in terms of magnitude and sharpness of the reflectivity minimum to those of the materials tested.

13.2 The single instrument relative two-sigma precision reported by one laboratory was $\pm 1.91\%$ and a $\pm 1.31\%$ for the determination of the wavelength of the minimum, or $\pm 2.50\%$ for the determination of the concentration, for *n*- and *p*-type silicon, respectively. There appeared to be only a slight concentration dependence on these values. These precisions are based on four measurements on each of the 10 specimens of each conductivity type of silicon used to establish the interlaboratory precision stated in 13.1, hence the range of validity is the same as that of the interlaboratory precision. Corresponding data for the other materials to which this document applies have not been obtained, but are expected to be comparable for the determination of the position of the minimum.

14. Keywords

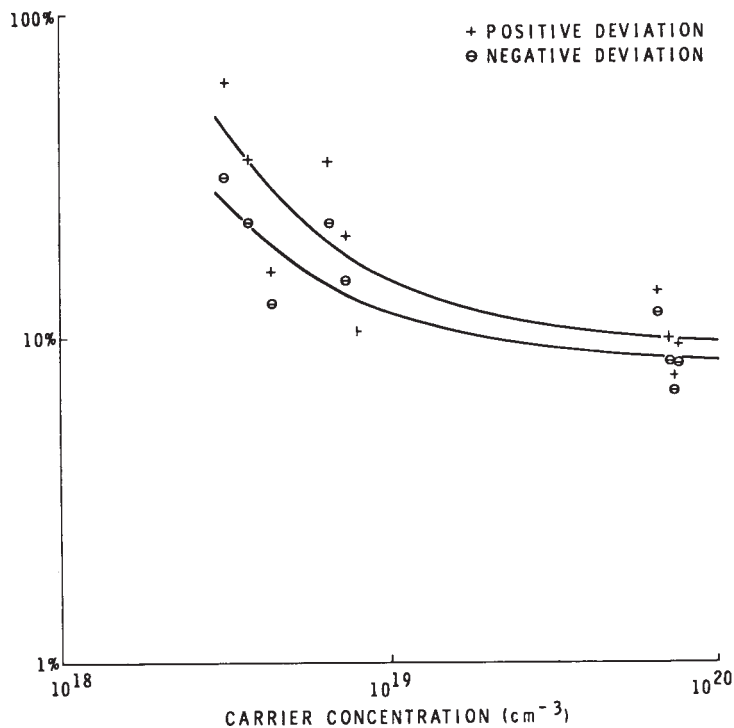
14.1 gallium arsenide; germanium; infrared reflectance; majority carrier concentration; plasma resonance; semiconductors; silicon

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TABLE 1 Constants for Calculating Carrier Concentration from the Wavelength of the Reflectivity Minimum

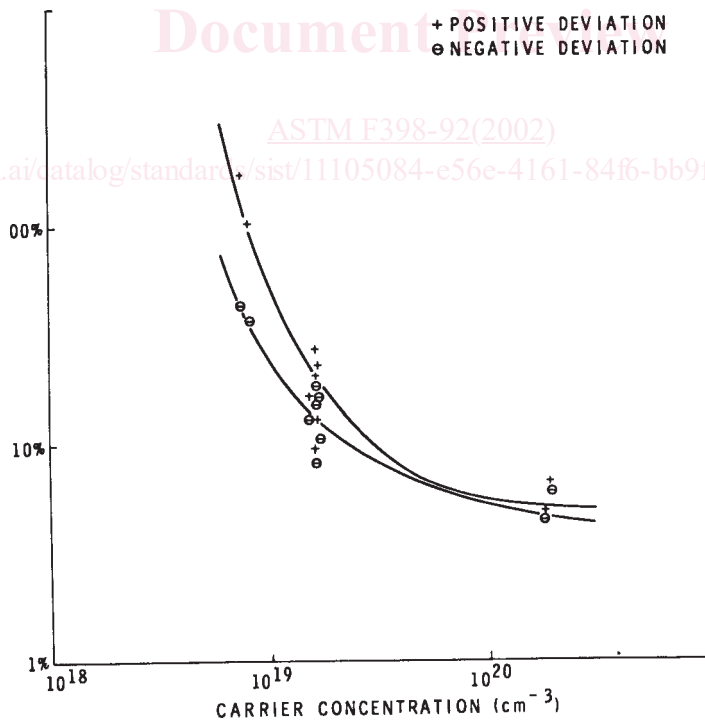
Material	Type	Applicable Range		A	B	C
		Wavelength, μm	Wavenumber, cm^{-1}			
Silicon	<i>n</i>	2.8–42.5	235–3570	3.039×10^{-12}	-1.835	-5.516×10^{-12}
Silicon	<i>p</i>	2.5–5.4	1850–4000	4.097×10^{-11}	-2.071	0
		5.4–32.4	310–1850	8.247×10^{-16}	-1.357	-2.626×10^{-15}
Gallium arsenide	<i>n</i>	9.4–18.5	540–1065	5.803×10^{-11}	-2.051	0
		18.5–30.4	330–540	2.405×10^{-8}	-2.898	0
		30.4–33.9	295–330	1.188×10^{-3}	-12.308	0
		3.7–30.0	330–2700	5.566×10^{-12}	-1.884	0

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NOTE 1—The data points are the concentration values calculated for a given specimen using the calibration curve and wavelength values two standard deviations above (+) and below (–) the interlaboratory mean wavelength for that specimen. The upper curve was fitted to the points (+) and the lower to the points (–).

FIG. 4 Least Squares Fit to Calculated Concentration Precision Data for n-type Silicon



NOTE 1—Fits were obtained as is described for Fig. 4.

FIG. 5 Least Squares Fit to Calculated Concentration Precision Data for p-type Silicon