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Precision Determination of Lattice Constants with a Geiger-Counter X-Ray Diffractometer*

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It is shown that the Geiger-counter x-ray diffractometer can be used for precision determination of lattice constants. The zero point of the goniometer (determined by two methods) and the position of the diffraction lines (2θ) were obtained with an accuracy of ±0.001°. The temperature was kept constant by a special unit within 0.05°C. An investigation of the systematic errors was made, which shows that the extrapolation of lattice constants *vs* $\cos^2\theta$, according to Wilson's equation, is permissible and the vertical divergence error, as computed by Eastabrook, lies within our tolerances for 2θ up to 165° and may be neglected. The lattice constants of Al, Ag, Ge, Si, CaF_2, CsI, TlCl, and TlBr, determined by extrapolation, are in good agreement with published data. The limiting factor in the accuracy of the lattice constant determination is not in the error of the diffractometer, but in the x-ray wavelength.

INTRODUCTION

FOR the precision determination of lattice constants, the following methods are in use: Straumanis' asymmetrical method,[1] the Kossel-van Bergen compensation method,[2] and symmetrical focusing method.[3] Lattice constants are obtained by these with a precision of a few units in the fifth decimal place. The discrepancy between the published data, however, is frequently ten times higher. The spectrometer method should give the same precision,[4] provided the zero position of the goniometer is located with a proper accuracy and a precise extrapolation of the lattice constant to $2\theta=180°$ can be made. This has not been possible thus far, and the spectrometer method was therefore not recommended for high precision.

Preliminary results with the Geiger-counter x-ray diffractometer[5] encouraged us to use the spectrometer method. Our experience with this instrument and the lattice constants of eight cubic crystals are reported in this paper.

GEIGER-COUNTER X-RAY DIFFRACTOMETER

The instrument itself has been described extensively by Parrish and Hamacher[6,7] and recently by Klug and Alexander.[8]

The accuracy of the instrument depends to a high degree on its alignment.[7] Of particular importance is an accurate determination of the zero position of the receiving slit. For this purpose a zero-setting slit (Fig. 1), as recommended by the manufacturer,[7] was placed in the center of the goniometer axis and the intensity distribution determined by counting in steps of 0.01° of 2θ (Fig. 2). Using the same intensity on the right and the left side of the maximum, the center line was determined. The intersection of the center line with the intensity curve gives one zero position. In order to eliminate the eccentricity of the setting slit, the same measurement was repeated, turning the slit 180° and the second zero position determined. The mean value of both gives the true zero position within 0.001°.

This method has a disadvantage, however, in that the axis of the sample holder has to be exchanged for a special axis; thus an unknown error might be introduced. As an alternative method we used therefore a shielding wedge,[9] placed directly on the sample-holder axis (Fig. 3). Rotating this wedge from +0.05° to −0.05° in steps of 0.01°, the x-ray intensity was determined by counting for two wedge positions differing by 180° (Fig. 4). The intersection of the two curves gives the zero point position. We obtained again an accuracy of 0.001° of 2θ in good agreement with the previous method. The result of three determinations by each method are given in the Table I.

The zero position of the sample holder (2:1 setting)

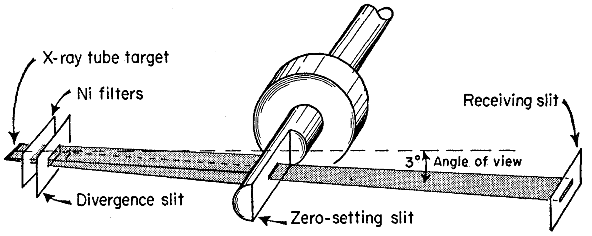

FIG. 1. Zero-setting slit for determination of the zero point of the goniometer.

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[*] Sponsored by the U. S. Office of Naval Research, the U. S. Army Signal Corps, the U. S. Air Force, and the U. S. Ordnance Material Research Office.

[1] M. E. Straumanis, J. Appl. Phys. **20**, 726 (1949); see also: M. Straumanis and A. Ievins, *Die Präzisionsbestimmung von Gitterkonstanten nach der asymmetrischen Methode* (Springer-Verlag Berlin, 1940).

[2] H. van Bergen, Ann. Physik [5] **33**, 737 (1938).

[3] E. R. Jette and F. Foote, J. Chem. Phys. **3**, 605 (1935).

[4] A. J. C. Wilson, J. Sci. Instr. **27**, 321 (1950).

[5] This instrument is built by North American Philips Company and was called a "spectrometer" until 1952. A similar instrument is made by General Electric Company and recently by some European companies.

[6] W. Parrish and E. A. Hamacher, Transactions of Instruments and Measurements Conference, Stockholm, 1952 (unpublished).

[7] E. A. Hamacher and W. Parrish, Technical Report No. 42, Philips Laboratory, Irvington-on-Hudson, New York, April 26, 1951 (unpublished).

[8] H. P. Klug and L. E. Alexander, *X-Ray Diffraction Procedures* (John Wiley and Sons, Inc., New York, 1954).

[9] M. Tournarie, J. phys. et radium **15**, 11A (1954).

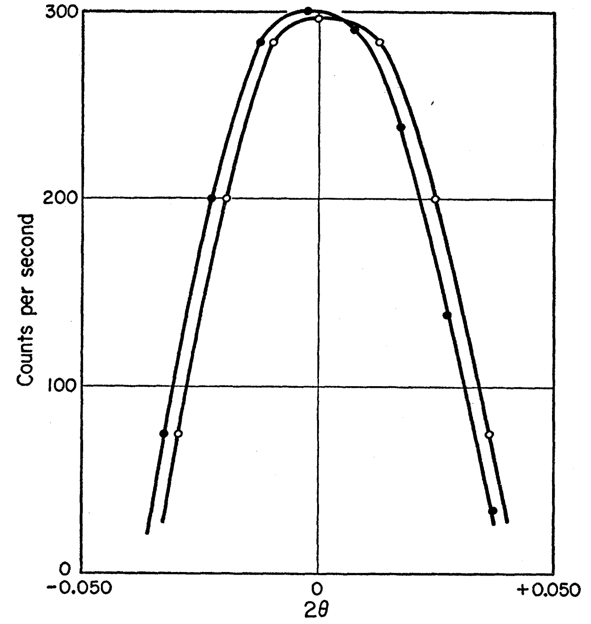

FIG. 2. Determination of the zero point of the goniometer by means of the zero-setting slit for two holder positions differing by 180°.

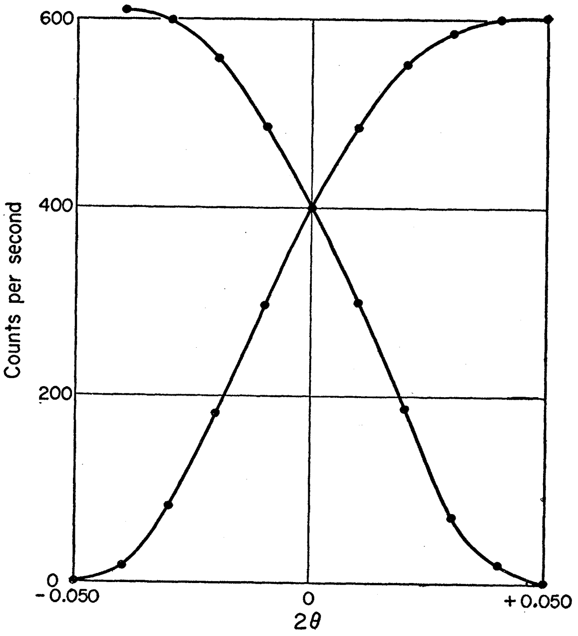

FIG. 4. Determination of the zero point of the goniometer by means of the shielding wedge for two holder positions differing by 180°.

was obtained by a special slit[7] (0.05 mm wide, 50 mm long) placed in the position of the sample holder (Fig. 5). An accuracy better than 0.01° was achieved, which is entirely adequate.

For lattice-constant precision higher than in the third decimal, the temperature of the sample, in general, must be controlled. The instrument as received from the factory does not have a temperature control, and therefore a special temperature chamber was built[10] (Fig. 6). This chamber connected to a Hoeppler ultrathermostat reduced temperature fluctuations to less than 0.05°C. The sample-holder axis was heated electrically to the same temperature when the room temperature was below 25°C in order to prevent a temperature gradient in the sample.

CRYSTALS AND SAMPLE PREPARATION

The materials investigated were: Al, Ge, Si, Ag, TlCl, TlBr, CaF_2, and CsI. This selection was made because Al is considered as the best x-ray standard material; Ge and Si are the standard semiconductors and can be made in very high purity; Ag, TlCl, and TlBr are strong absorbers for x-rays; and CaF_2 and CsI were not yet measured accurately. Only Ag was in microcrystalline form; the other materials were available in large single crystals.

The purity of the materials was: Ag[11] (99.999%) contained Cu, Pb, Fe, each $<0.0001\%$, and Mg $\simeq 0.0001\%$; Al[12] (99.99+%) contained 0.001% Si and a few other elements each $<0.0001\%$; Ge[13] and Si[13] were characterized by the resistivity of 40 and 100 ohm-cm, respectively; TlCl, TlBr, and CsI were prepared in this laboratory; the thallium halides, according to spectroanalysis, showed only traces of a few common elements; a reciprocal contamination of anions was

TABLE I. Accuracy of zero-point determination of goniometer by zero-setting-slit and shielding-wedge method.

	Zero-setting-slit 2θ	Shielding-wedge 2θ
	0.0075°	0.0065°
	0.0080	0.0067
	0.0073	0.0065
Average	0.0076°	0.0066°
Difference	0.001°	

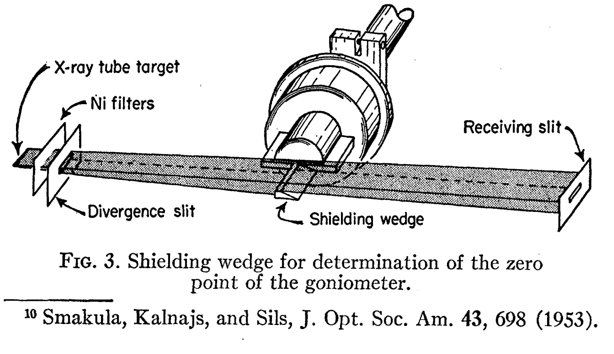

FIG. 3. Shielding wedge for determination of the zero point of the goniometer.

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[10] Smakula, Kalnajs, and Sils, J. Opt. Soc. Am. 43, 698 (1953).

[11] Material and spectroscopic analysis from Handy and Harman, Bridgeport, Connecticut.

[12] Material and spectroanalysis from Aluminum Company of America, New Kensington, Pennsylvania.

[13] Material and resistivity data from Raytheon Manufacturing Company, Waltham, Massachusetts (courtesy of Dr. H. Statz).

prevented by the use of Br-free HCl and Cl-free HBr for the synthesis; CaF_2[14] contained <0.001% of Ag, Fe, Mg, and Si, and somewhat more Sr; CsI was contaminated by Fe, K, Mg, Na, Sr, and V to <0.001% and by Rb about 0.01%.

The powder of the soft crystals (Al, TlCl, TlBr) was prepared by filing with a fine jeweler saw, using light strokes to prevent excessive heating. The harder crystals (Si, Ge, CaF_2, CsI) were ground in an agate mortar. The powder of all crystals except Si and Ge was annealed for 15 to 20 hours (Al, Ag, and CaF_2 at 500°C and CsI, TlCl, and TlBr at 300°C) in sealed Pyrex tubes, then sieved through a 325-mesh screen and packed gently in a Pyrex specimen holder with flat polished surfaces on an optically flat support. A few drops of a dilute binder (Lucite, collodion, or oil) were added when necessary. The powder specimens of TlCl and TlBr, not requiring a binder, were baked over night at *ca* 200°C with the sample top on an optical flat to prevent surface deformation. The packing density of powders was *ca* 65 percent.

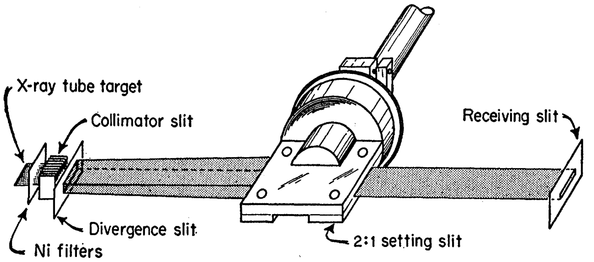

FIG. 5. Determination of the zero positions of the sample by means of (2:1) setting slit.

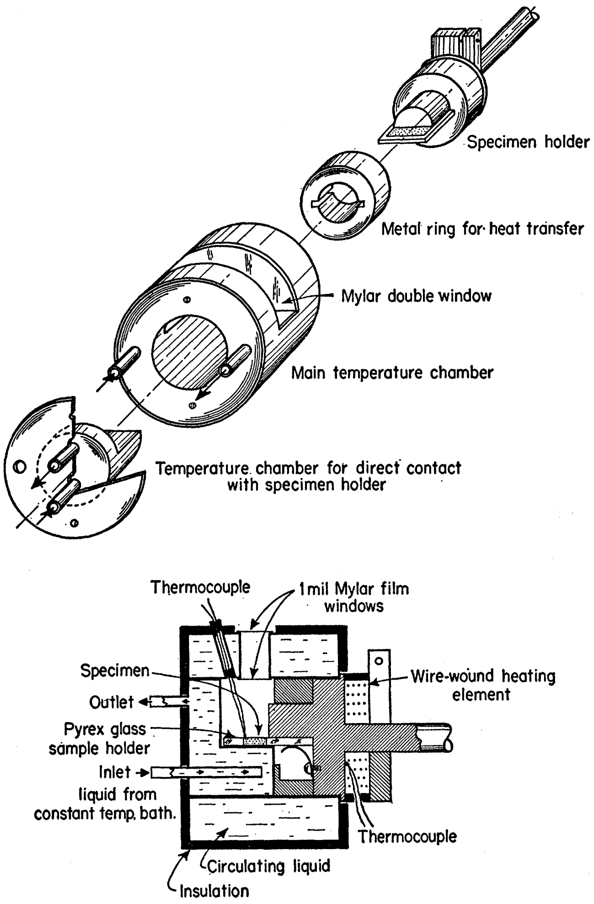

FIG. 6. Constant temperature chamber.

LOCATION OF DIFFRACTION ANGLES

A preliminary recording of the diffraction spectrum for each crystal gave the approximate location and intensity of the lines. After temperature equilibrium had been reached and the equipment stabilized, final measurements were taken by counting, scanning in steps of 0.025° or 0.05° in 2θ. The step setting was done by motor drive ($\frac{1}{8}$° per minute) from high to low angles. This method gave a better reproducibility than setting by hand. The total number of counts was always greater than 10 000 for each point. This corresponds to an average error of about 0.67 percent. The counting rate was not greater than 600 counts per second, that is, in the linear range of the Geiger counter.

The peaks of the diffraction lines, which were used only for lattice-constant determinations, are found graphically by extrapolating the center lines to the maximum of the intensity curves (Fig. 7). The accuracy of 2θ determined this way is $\pm 0.001°$. Only sharp, single, and well-resolved lines in the back reflection region $2\theta > 120°$ were used. The zero point of the goniometer was rechecked frequently and the aperture (4°) and the receiving slit (0.006 in.) not touched during the measurements. The short-anode Cu tube was operated with 35 kvp and 15 ma and nickel filter.

SYSTEMATIC ERRORS

Although the diffraction angles can be determined very accurately, the computed lattice constants contain

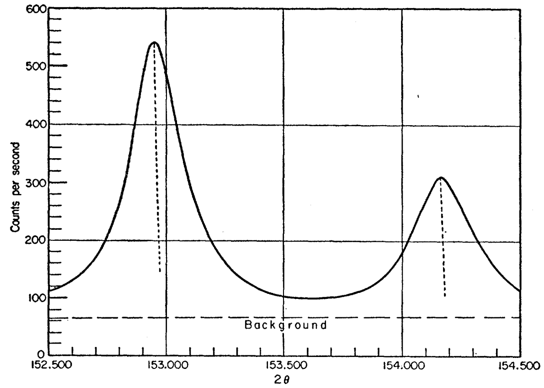

FIG. 7. Graphical determination of the peak of diffraction-line profile (711/551) for Cu $K\alpha_1$ and Cu $K\alpha_2$ for germanium.

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[14] From Optovac, North Brookfield, Massachusetts.

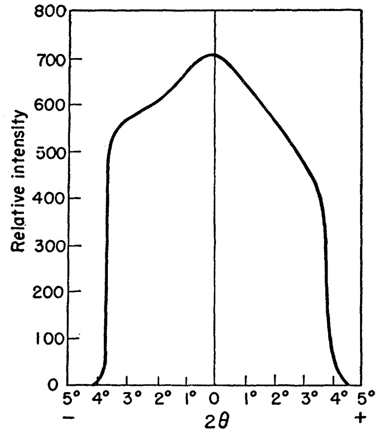

FIG. 8. Intensity distribution of the primary beam from a short anode Cu tube for 4° divergence slit and 0.15 mm receiving slit.

various systematic errors which have to be eliminated. The position and the shape of diffraction lines as given by the diffractometer are determined by the following factors[4,15]: (1) alignment of the instrument; (2) width and the profile of the primary beam; (3) width of the receiving slit; (4) penetration depth of the x-rays; (5) flatness of the sample; (6) sample displacement from the axis of rotation; and (7) vertical divergence of the x-ray beam.

The alignment and adjustment of the instrument was carried out as accurately as possible according to the manufacturer's instructions to obtain maximum intensity, optimum resolution, and correct line position.

The effective width of the primary beam was 0.06 mm. The vertical intensity distribution of the primary beam of our x-ray tube is shown in Fig. 8. It proves to be unsymmetrical; the shoulder is steeper on the low angle side. This might have an influence on the asymmetry of the diffraction lines at high angles.

The receiving slit used was 0.15 mm, that is, 2.5 times the effective width of the x-ray source, but still only about 1/10 of the half-width of the diffraction lines.

While the first three errors cause only a broadening of the lines, the next four cause a displacement and asymmetrical broadening. According to Wilson[4] and Eastabrook,[15] the total displacement of the center of gravity of a diffraction line caused by the last four errors is given approximately by

$$\Delta\theta = -\left\{\frac{\sin 2\theta}{4\mu R} - \frac{t\cos\theta}{R[\exp(2\mu t/\sin\theta)-1]}\right\} - \frac{A^2\sin 2\theta}{6R^2} - \frac{s\cos\theta}{R} - \frac{\delta^2\cot 2\theta}{12},$$

where μ = linear absorption coefficient of the material for x-ray wavelengths used in cm^{-1}; R = radius of the goniometer in cm; t = critical thickness of the sample $= (3.2/\mu)(\rho/\rho')\sin\theta$ in cm; ρ = density of solid material in g/cm^3; ρ' = density of the powder in g/cm^3; A = one-half the effective length of the specimen in cm; δ = spacing of the Soller slits divided by their length; and s = displacement of the front face of the specimen from the axis of rotation in cm.

The bracketed member in the above equation corrects for the penetration depth; the next, for the flatness of the sample; the third, for the sample displacement from the axis of rotation; and the fourth, for the vertical divergence of the x-ray beam. The first three are always negative and cause the diffraction lines to shift toward smaller angles. The shifting decreases with increasing 2θ and becomes zero for $2\theta = 180°$. The behavior of the vertical divergence error is different; it is negative for $2\theta < 90°$, zero for $2\theta = 90°$, and positive for $2\theta > 90°$. In the back-reflection region of interest here, the errors 4, 5, and 6 oppose that of 7; hence a diffraction angle exists at which the sum of the error goes through zero.

COMPARISON OF THEORETICAL AND EXPERIMENTAL SYSTEMATIC ERRORS

The error for penetration depth was computed, using $R = 17$ cm and 65 percent of the linear absorption coefficients (packing density of the samples was 65 percent). The second member in the bracket, containing the exponential function, can be disregarded because it is several orders smaller than any of the other members. Computed errors of penetration depth for Al, Ge, and TlCl are given in Fig. 9. It was possible to

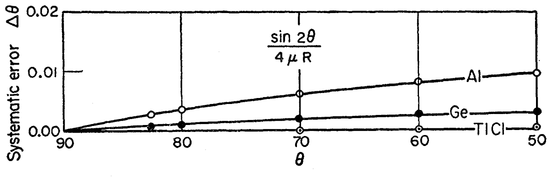

FIG. 9. Computed errors of penetration depth for Al, Ge, and TlCl.

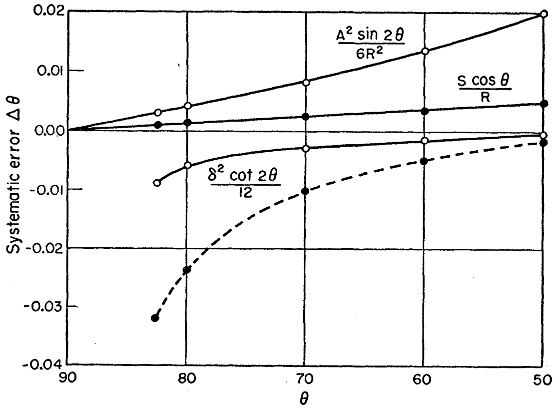

FIG. 10. Computed errors for flatness of the sample $A^2\sin 2\theta/6R^2$, for sample displacement from the axis of rotation $s\cos\theta/R$, and for vertical divergence $\delta^2\cot 2\theta/12$; dashes for $\delta = 0.04$; solid line for $\delta = 0.02$.

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[15] J. N. Eastabrook, Brit. J. Appl. Phys. 3, 349 (1952).

check the error of penetration depth experimentally by comparing the computed and experimental differences for Al (low absorbing) and TlBr (high absorbing). The values agree within 20 percent.

The error caused by specimen flatness is computed for $A_0 = 0.6$ cm, using the change of A with 2θ according to $A = A_0/\cos(90-\theta)$. The effective length A of irradiated area is not constant, but varies with 2θ, having a minimum A_0 at $2\theta = 180°$. An experimental check of this error was made on Ge with 1° and 4° apertures. A discrepancy by a factor three was found. This is, however, not surprising since the theoretical value of the error for 1° aperture cannot be correct. The derivation is valid only as long as the penetration depth is small compared with the effective length of the sample, which is not the case for 1° aperture.

The sample displacement from the center of rotation is somewhat uncertain. It is reasonable to assume $s = 0.01$ to 0.03 mm.[4] Errors for specimen flatness and displacement from the center are given in Fig. 10.

In the vertical divergence error the constant δ was determined by measuring the total width of the collimator slit set (in this case 10 mm), dividing it by 18 spacings, and correcting for the thickness of the spacers, $d = 0.025$ mm. We obtain for the single slit width $w = 0.53$ mm. Since the length of the slit is 12.5 mm, $\delta = 0.53/12.5 = 0.042$. The error computed with this value of δ is for $2\theta > 140°$ higher than the sum of three others and increases rapidly with 2θ (Fig. 10). If this magnitude of the error is true, a strong decrease of the lattice constant *vs* 2θ would take place. This has not been observed (Fig. 11). The lattice constant continuously decreases with 2θ without any indication of an inflection. We assume therefore that the vertical divergence error in the region of $2\theta = 160°$ is approximately equal to the sum of the three other errors or smaller; we set the effective value of δ to one half of the geometrical value.

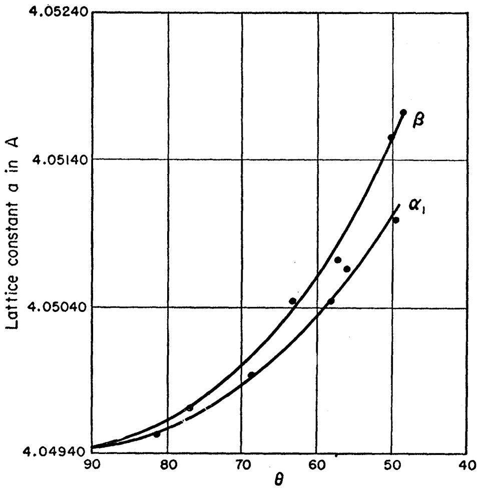

FIG. 11. Lattice constant of aluminum computed from various diffraction angles for Cu $K\alpha_1$ and Cu $K\beta$.

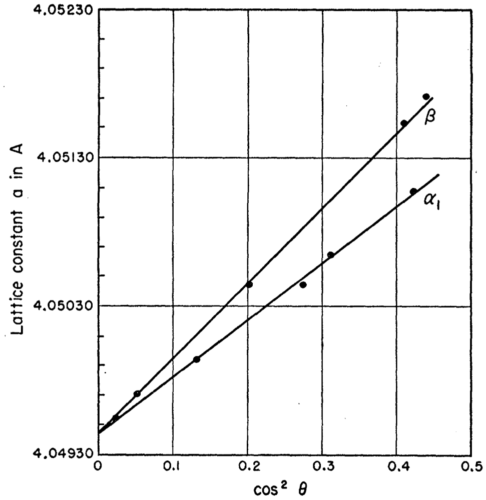

FIG. 12. Extrapolation of lattice constant *vs* $\cos^2\theta$ for aluminum for Cu $K\alpha_1$ and Cu $K\beta$.

TABLE II. Lattice constants determined by the Geiger-counter diffractometer using extrapolation and Cu $K\alpha_1$($\lambda = 1.54051$A) in angstroms at 25.0°C.

		Correction for		Δa error	
	a_{uncorr}	refractive index	a_{corr}	random	λ
Al	4.04957	0.00003	4.04960	0.00002	0.00016
Ag	4.08609	0.00012	4.08621	0.00002	0.00016
Si	5.43068	0.00004	5.43072	0.00002	0.00022
Ge	5.65745	0.00009	5.65754	0.00002	0.00023
TlCl	3.84240	0.00007	3.84247	0.00002	0.00015
TlBr	3.98580	0.00008	3.98588	0.00002	0.00016
CaF_2	5.46337	0.00005	5.46342	0.00002	0.00022
CsI	4.56766	0.00006	4.56772	0.00002	0.00018

The experimental determination of total errors was done in the following way. The lattice constants computed from various diffraction angles were plotted *versus* $\cos^2\theta$ according to Wilson.[4] This plotting gives straight lines as shown in Fig. 12 for Al. The extrapolation to $2\theta = 180°$ gives the lattice constants a, corrected for systematic error. From lattice constants so obtained the diffraction angles were computed for the same Miller indices for which the diffraction lines were found with the diffractometer. The difference gives the total systematic errors. They are of the same order as the errors computed from the Wilson-Eastabrook equation.

FINAL DETERMINATION AND THE ACCURACY OF THE LATTICE CONSTANTS

In using the $\cos^2\theta$ extrapolation one has to keep several points in mind. Diffraction lines obtained with different x-ray wavelengths have to be plotted separately because of absorption effect. The absorption effect is shown in Figs. 11 and 12 for Cu $K\alpha_1$ and β lines. If the packing density from sample to sample is different, the position of the lines will vary. Points obtained with different apertures will not lie on the same line. All

TABLE III. Lattice constants from the literature recomputed for 25.0°C and corrected for new wavelengths according to Lonsdale.[a]

	Purity	Temperature	Original a in kx units	Error	Exp. coefficients	Recomputed $a_{25^\circ\mathrm{C}}$
Al	99.971%	25°C	4.04139	±0.00008	...	4.04958[b]
	99.9986	25°	4.04145	±0.00002	23.29×10^{-6}	4.04963[c]
	99.992	25°	4.04134	...	...	4.04953[d]
	99.99	20°	4.04091	±0.00006	...	4.04958[e]
Ag	99.999	25°	4.07787	±0.00008	18.72×10^{-6}	4.08613[b]
	...	25°	4.07784	±0.00003	...	4.08610[f]
Si	99.84	25°	5.41982	±0.00034	4.15×10^{-6}	5.43078[b]
	...	23°	5.41975	±0.00005	...	5.43078[f]
	99.9	18.5°	5.41964	±0.00005	...	5.43075[g]
	99.97	25°	5.42002	±0.00003	...	5.43100[h]
Ge	99.999	25°	5.646235	±0.000002	5.92×10^{-6}	5.65758[h]
TlCl	spec. pure	25°	3.83459	±0.00004	54.57×10^{-6}	3.84236[f]
	spec. pure	25°	...	±0.00010	$53.5\ \times10^{-6}$	3.84270[i]
TlBr	spec. pure	25°	3.97778	±0.00004	$51.2\ \times10^{-6}$	3.98584[f]
CaF_2	j	25°	...	±0.0002	...	5.4626[k]
CsI	l	...	...	±0.0001	$48.6\ \times10^{-6}$	4.5678[m]

[a] See reference 16.
[b] See reference 3.
[c] See reference 17.
[d] See reference 18.
[e] See reference 19.
[f] See reference 1.
[g] See reference 20.
[h] See reference 21.
[i] See reference 22.
[j] Spectroscopic analysis: As, B, Fe, Mg, Si, and Sr <0.001% each. Ag and Cu <0.0001% each.
[k] See reference 23.
[l] Na and K 0.01% each, Rb 0.03%.
[m] See reference 24.

parameters have to be kept constant for each extrapolation.

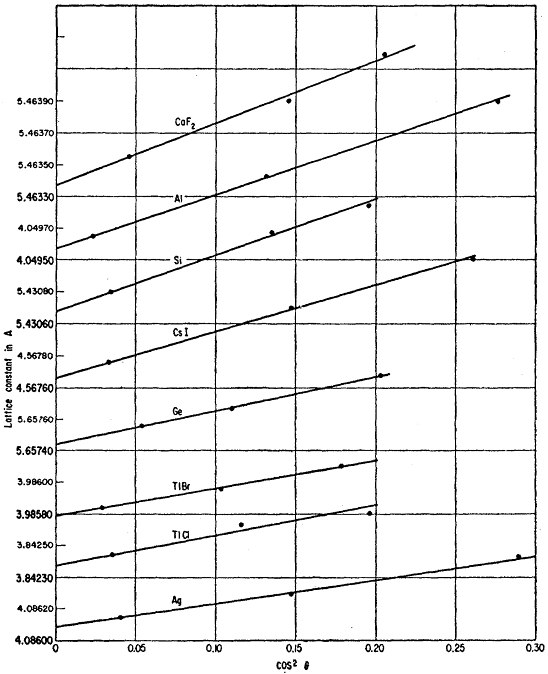

FIG. 13. Determination of lattice constants by extrapolation. A different ordinate is used for each crystal.

The extrapolation for the eight crystals are presented in Fig. 13 and the final lattice constants summarized in Table II. Column 2 shows the extrapolated values, column 3 corrections for refraction, and column 4 the corrected values. Table III gives the corresponding data from the literature.[1,3,16–24]

The accuracy of our lattice constants is determined by random and by systematic errors. The random errors are connected with the determination of diffraction angles. The average error of θ is ±0.001°. Since the error in the zero position is ±0.001° (Table I), the total random error in θ is ±0.002°. Using diffraction lines for $\theta > 50°$ the $\Delta\theta$ error has a very small influence on the lattice constant since $\Delta a = -a \cot\theta\Delta\theta$. For $\theta = 60°$, $\Delta a \simeq 0.002\%$ and for $\theta = 80°$, $\Delta a \simeq 0.0006\%$.

The systematic error consists of wavelength and extrapolation errors. The value of Cu $K\alpha_1$ is 1.54051 A, based on newer measurements[25] and recommended by

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[16] K. Lonsdale, Acta Cryst. **3**, 400 (1950).

[17] A. Ievins and M. Straumanis, Z. physik. Chem. **B34**, 402 (1936).

[18] A. J. C. Wilson, Proc. Phys. Soc. (London) **53**, 235 (1941).

[19] H. van Bergen, Ann. Physik [5] **39**, 553 (1941).

[20] H. Lipson and L. E. R. Rogers, Phil. Mag. [7] **35**, 544 (1944).

[21] M. E. Straumanis and E. Z. Aka, J. Appl. Phys. **23**, 330 (1952).

[22] P. G. Hambling, Acta Cryst. **6**, 98 (1953).

[23] H. E. Swanson and E. Tatge, National Bureau of Standards Report, 1953 (unpublished), Vol. 1, p. 69.

[24] T. B. Rymer and P. G. Hambling, Acta Cryst. **4**, 565 (1951).

[25] Y. Cauchois and H. Hulubei, *Tables des constantes et données numériques* (Hermann et Cie., Paris, 1947).

Lonsdale.[16] The wavelength error, according to Bragg,[26] is $\Delta\lambda = \pm 0.004\%$ ($\pm 0.001\%$ for kx and $\pm 0.003\%$ for the conversion factor λ_g/λ_s). Assuming the correctness of the extrapolation *vs* $\cos^2\theta$, the accuracy of the extrapolated values averages $\pm 0.0005\%$ or less. Thus the main error in the absolute lattice constants is caused by the uncertainty of x-ray wavelengths.

As an indication of wavelength uncertainty we show in Table IV the lattice spacings of Ge ($hkl = 711/511$) and of TlBr ($hkl = 510/431$) determined by Cu $K\alpha_1$ and Cu $K\alpha_2$, using the older λ values given by Bragg[27] and the newer by Lonsdale.[16] The new α_1 value is increased and α_2 decreased by 0.00001 A. Since α_1 and α_2 are very close, all instrumental errors will be practically the same. As one can see from Table IV, this small change in wavelength causes a change in spacing by 8 and 5 units in the fifth place. From the extrapolation lines, however, the difference in spacing should be not more than 2 units.

An objection can be made against the use of the peaks instead of the centers of gravity for determination of 2θ. According to Wilson, his formula is derived for the center of gravity, but at high diffraction angles it is valid for peaks also. In addition, as one can see from Fig. 8, the intensity distribution of the primary beam is unsymmetrical, contrary to the theory. Another influence on asymmetrical broadening is caused by vertical divergence which is also not included in Wilson's computation.

A comparison with published data obtained by Debye-Scherrer technique (see Table III) shows that our lattice constants are close to older data. However, a slight systematic discrepancy seems to exist. Our data are somewhat lower for low-absorbing crystals (Al, Si, Ge) and higher for high-absorbing (TlCl, TlBr, Ag). The deviation for high-absorbing crystals is probably caused by the absorption error in the Debye-Scherrer method. The reason for the opposite deviation in low-absorbing crystals is not known. A possibility of contamination by impurities was checked on Si and Ge. The data obtained for 9 Si and 7 Ge samples are shown in Tables V and VI. For all samples the average error in θ is less than 0.001° and the error in lattice constant ± 0.00001 A. Thus we can exclude a noticeable influence of impurities. The strongest support for the reliability of our lattice constants is given by good agreement of densities of our crystals computed from lattice constants and measured by weighing.[28]

TABLE IV. Lattice constants of Ge (711/511) and TlBr (510/431), determined from Cu $K\alpha_1$ and α_2, using $\lambda_{\alpha_1} = 1.54050$ A and 1.54051 A, and $\lambda_{\alpha_2} = 1.54434$ A and 1.54433 A.

	λ according to Bragg[a]			λ according to Lonsdale[b]		
	$\lambda = 1.54050$	$\lambda = 1.54434$	Δa	$\lambda = 1.54051$	$\lambda = 1.54433$	Δa
Ge	5.65752	5.65751	0.00001	5.65756	5.65747	0.00009
TlBr	3.98583	3.98579	0.00004	3.98585	3.98576	0.00009

[a] See reference 27.
[b] See reference 16.

TABLE V. θ values for three diffraction lines of various Si crystals at 25.0°C, Cu $K\alpha_1$ and corresponding lattice constants.

No.	(444) $\theta°$	(533) $\theta°$	(620) $\theta°$
1	79.303_8	68.432_8	63.757
2	79.306	68.434	63.755
3	79.305	68.432_8	63.759
4	79.305_5	68.432_8	63.760_3
5	79.306_5	...	...
6	79.307	68.434_3	63.757_8
7	79.308_3	68.430_8	63.755_8
8	79.307_5	68.431_5	63.756_3
9	79.308	68.429	63.754_5
Average	79.306_4 ± 0.0005	68.432_3 ± 0.0006	63.757 ± 0.0007
a	5.43080 ± 0.00001	5.43117 ± 0.00002	5.43135 ± 0.00003

$a_{\text{extrap}} = 5.43068$ A

TABLE VI. θ values for three diffraction lines of various Ge crystals at 25.0°C, Cu $K\alpha_1$ and corresponding lattice constants.

No.	(711/551)	(444)	(533)
1	76.477_5	70.600	63.216
2	76.478	70.597_5	63.214
3	76.478	70.601	63.219_5
4	76.479	70.601_5	63.217_5
5	76.477	70.602_5	63.217
6	76.479	...	...
7	76.477_5	70.601_5	63.217_5
Average	76.478_0 ± 0.0003	70.600_7 ± 0.0007	63.216_9 ± 0.0007
a	5.65755 ± 0.00001	5.65769 ± 0.00003	5.65788 ± 0.00003

$a_{\text{extrap}} = 5.65745$ A

We conclude that with the Geiger-counter diffractometer a precision in lattice constant measurement of ± 0.00002 A can be obtained, but that the absolute value is limited to 0.004 percent by the uncertainty of the x-ray wavelengths.

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[27] W. L. Bragg, Acta Cryst. **1**, 46 (1948).
[28] Smakula, Kalnajs, and Sils, Phys. Rev. **99**, 1747 (1955), this issue.