The case of $p=4$ stands for a generation of $60R$ from $4H$ and that of $p=5$ stands for a generation of $72R$ from $4H$. In these cases the large block $[4H]_p$ obeys the s.s.p. and, furthermore, incidental slips of the small blocks (AB) and (CB) occur.

Agrawal & Trigunayat (1968) reported that $12R$ was coalesced with $4H$. In the present study $18R$ was coalesced with $6H$ (Fig. 1). The layer sequences characteristic of R polytypes shown above are easily understood if, on the basis of these facts of coalescence, a hypothesis is built up that the s.s.p. with a slip unit of integral multiples of the period of an H polytype occurs in the H polytype, $4H$, $6H$, $8H$, $10H$ or $14H$, and an R polytype is formed.

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References

AGRAWAL, V. K. & TRIGUNAYAT, G. C. (1968). *Acta Cryst.* B**24**, 971–972.
CHADHA, G. K. & TRIGUNAYAT, G. C. (1967). *Acta Cryst.* **22**, 573–579.
JAIN, R. K., CHADHA, G. K. & TRIGUNAYAT, G. C. (1970). *Acta Cryst.* B**26**, 1785–1791.
LAL, G., CHADHA, G. K. & TRIGUNAYAT, G. C. (1971). *Acta Cryst.* B**27**, 2293–2298.
MARDIX, S., KALMAN, Z. H. & STEINBERGER, I. T. (1968). *Acta Cryst.* A**24**, 464–469.
MITCHELL, R. S. (1956). *Z. Kristallogr.* **108**, 296–315.
PRASAD, R. & SRIVASTAVA, O. N. (1971). *Acta Cryst.* A**27**, 259–264.

Acta Cryst. (1976). A**32**, 375

Coherent Nuclear Scattering Amplitudes of Germanium, Copper and Oxygen for Thermal Neutrons

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The forward nuclear scattering amplitudes of Ge, Cu and O for thermal neutrons have been determined from the refractive bending by pure single-crystal right prisms of germanium, copper, and quartz. The results are $b_{Ge}=8{\cdot}1929$ (17) fm and $b_{Cu}=7{\cdot}689$ (6) fm which are in good agreement with previous less accurate determinations by other methods and $b_O=5{\cdot}830$ (2) fm which disagrees with an independent accurate determination by four standard deviations.

Introduction

Both Pendellösung and prism refraction offer effects from which neutron structure factors or scattering amplitudes can be determined with a precision of one part in 5000 (Shull & Shaw, 1973; Schneider, 1973). Small-angle scattering (Koester & Knopf, 1971) from powders immersed in liquids of differing refractive indexes and mirror reflection (Donaldson, Passel, Bartolini & Graves, 1965) can lead to scattering amplitudes with precisions of one part in 1000. Since the small neutron–electron Foldy (1951) interaction disappears in the forward scattering direction, prism refraction, mirror reflection and small-angle scattering experiments on non-magnetic materials yield direct values for the nuclear-force scattering amplitude. Comparison of the prism and Pendellösung results can yield information on the Foldy scattering amplitude, $b_{Atom}=b_{Nucleus}+b_{Foldy}$ where $b_{Foldy}=+0{\cdot}00131\,(1-f)Z$ fm where Z is the number of nuclear protons and f is the electron form factor, if the Debye–Waller temperature factor is known with sufficient precision. The excellent work of Batterman & Chipman (1962) provides the Debye temperature $\theta_{Ge}=290$ (5) K with just less than sufficient precision to draw significant conclusions here. The discrepancy between the theoretical (Foldy, 1951) and experimental (Krohn & Ringo, 1966, 1973) values for the neutron–electron interaction must then be resolved by other means.

The purpose of this work is rather to improve the basic knowledge of thermal neutron scattering amplitudes for several common elements: Ge, Cu, and O. The need for scattering amplitudes is not only to extend our knowledge of the neutron interactions with atoms, but also to allow more detailed studies of materials' structures where diffracted intensities depend upon structure and scattering amplitudes.

The prism technique used here extracts the scattering

amplitude from the refractive index (Goldberger & Seitz, 1947)

$$n=1-N\lambda^2 b/2\pi=1-\delta\,, \qquad (1)$$

where N is the atomic density, λ is the neutron wavelength and b is the forward atomic scattering amplitude which for our three samples is simply that due to the nuclear potential. The imaginary component of the scattering amplitude can be ignored since the refractive index is affected by less than a part per million. The above expression for δ is exact in first order or within a part in 10^5 which is here neglected. Finally, the refractive bending suffered by a neutron ray in passing through a right prism as shown in Fig. 1 is found from Snell's Law applied at the entrance and exit surfaces:

$$n_{\text{air}}\cos\varphi=n_{\text{prism}}\cos(\varphi-\Delta_1) \qquad (2a)$$

$$n_{\text{prism}}\sin(\varphi-\Delta_1)=n_{\text{air}}\sin(\varphi-\Delta_T)\,, \qquad (2b)$$

where φ is the angle between the neutron ray and first prism surface, Δ_1 is the refraction suffered at the first surface and Δ_T is the total refraction due to both surfaces. From these two equations Δ_1 can be eliminated yielding:

$$n^2_{\text{prism}}=n^2_{\text{air}}[\cos^2\varphi+\sin^2(\varphi-\Delta_T)]\,, \qquad (3)$$

which is exact. Since we seek δ which is already first-order approximate, a working equation may be used,

$$\delta_{\text{prism}}=\tfrac{1}{2}[\sin^2\varphi-\sin^2(\varphi-\Delta_T)]+\delta_{\text{air}} \qquad (4a)$$

or

$$\delta_{\text{prism}}=\tfrac{1}{2}\sin\Delta_T\sin(2\varphi-\Delta_T)+\delta_{\text{air}}\,, \qquad (4b)$$

which is accurate within a part in 10^5.

The finite collimation of the refracted beam (FWHM $=9{\cdot}6'$) caused in first order a refractive profile broadening and required a second-order correction to the refractive shift:

$$\Delta_T(\varphi_0)=\Delta_T(\text{measured})\left\{1-\frac{\beta_H^2}{12}\left[4\cot^2(2\varphi_0)+1\right]\right\} \qquad (5)$$

where β_H is the horizontal collimation FWHM and φ_0 is the central angle of the incident beam from the first prism surface. This collimation correction is always less than a part per thousand.

A correction was also applied for the change in effective wavelength passing through the prism due to its preferential attenuation away from the apex and the wavelength dispersion across the width of the prism as defined by the slits following the prism or by the prism edge itself for the more absorbing Cu prism.

The slits for Ge and SiO_2 varied in width as the prism angle was changed, but the slit center was held constant. The prism, which rotated about a vertical axis through the center of its first surface, passed a beam of fixed center (see Fig. 1). The originally uniform intensity across the slits became canted owing to prism absorption and the effective squared wavelength became:

$$\langle\lambda^2\rangle=\int_{-w/2}^{w/2}\lambda^2 I(x)\mathrm{d}x=\int_{-w/2}^{w/2}(\lambda_0^2+2\alpha x\lambda_0)\frac{\mu\exp(-\mu x)\mathrm{d}x}{2\sinh z_1}$$

$$\langle\lambda^2\rangle=\lambda_0^2[1+(2\alpha/\lambda_0\mu)(1-z_1\coth z_1)]\,, \qquad (6)$$

where $z_1=\mu w/2$, $I(x)$ is normalized, $\alpha=\mathrm{d}\lambda/\mathrm{d}x=\lambda_0\cot\theta\mathrm{d}\theta/\mathrm{d}x$, where x is the coordinate of translation across the slit and θ is the monochromator Bragg angle, $\mu=N\sigma\csc\varphi$ where N is the atomic density and σ is the

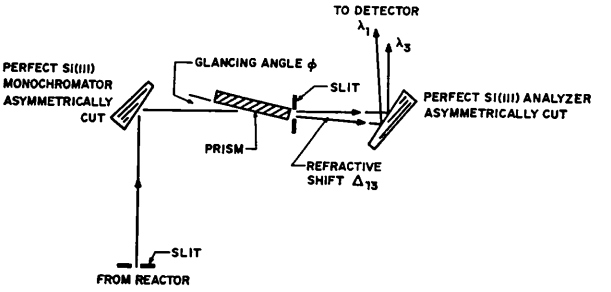

Fig. 1. Schematic diagram of double-crystal spectrometer used in measuring prism refractive bending of thermal neutrons. The pair of slits and monochromator determine the neutron wavelength–angle dispersion.

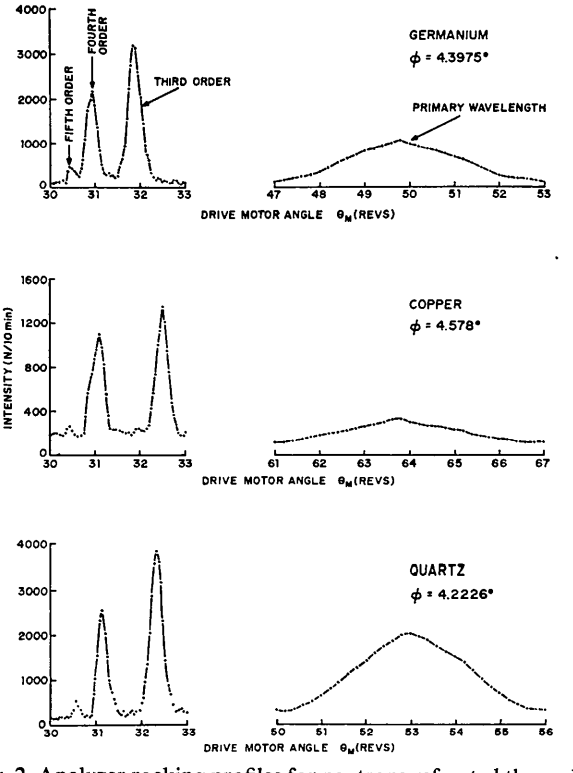

Fig. 2. Analyzer rocking profiles for neutrons refracted through right prisms with first surfaces at roughly 4° glancing angle from the incident beam. The higher-order wavelengths passed by the silicon monochromator are useful in measuring the deflections (1 rev = 6·540 μrad).

atomic cross section for beam attenuation, and w is the slit width for the prism orientation φ. This correction was always less than a part per thousand and can be applied to Δ_T which is closely proportional to λ^2.

$$\Delta_T(\lambda_0)=\Delta_T(\text{meas})\,[1-(2\alpha/\lambda_0\mu)\,.\,(1-z_1\coth z_1)]\,. \quad (7)$$

The effective squared wavelength for the various Cu prism settings was determined by the position of the leading prism edge in the beam whose wavelength varied as before according to the dispersion established by the Si monochromator crystal. The beam width was defined by the projection of the prism length upon the beam cross section for each prism setting φ. Thus, the effective squared wavelength became:

$$\langle\lambda^2\rangle=\lambda_0^2[1+(2\alpha\,\sin\varphi/\lambda_0)\,(l-l_0)] \quad (8)$$

where

$$l_0=[1-\exp(-z_2)\,(1+z_2)]/N\sigma\,,$$

and

$$z_2=N\sigma L\,\sec\varphi;$$

N, σ, and α have been defined and $l=3{\cdot}40$ (12) cm is the length of the leading prism edge from its axis of rotation and $L=5{\cdot}987$ (3) cm is the total prism length. Since the sec φ function is slowly varying for small φ used here, the entire square bracket can be taken as nearly constant: $l_{\text{eff}}=2{\cdot}27$ (13) cm for all φ. Again, this correction can be applied inversely to Δ_T.

Experiment

The technique for measuring the very small angles of neutron refraction has been well established (Schneider, 1973; Schneider & Shull, 1971; Just, Schneider, Ciszewski & Shull, 1973); these angles, from 10 to 100″ in this experiment, are resolved on a double perfect crystal refractometer whose rocking width is a few seconds of arc and from which the centers of peaks may be determined within a few milliseconds of arc. In order to automate the data collection, the first and third orders of wavelengths reflected from the Si monochromator crystal were observed in refraction so that the measured angular shift (see Fig. 1) between these refracted components would require neither removal of the precisely oriented prism nor corrections for analyzer crystal curvature due to spatial separation of adjacent refracted and unrefracted beams.

The accumulated data consisted of intensity profiles due to scanning by the analyzer crystal for each of several orientations of the prism. These rectangular prisms, Ge: $17\times26\times116$ mm, Cu: $12\times23\times60$ mm, and SiO_2: $16\times26\times108$ mm were placed between the monochromator and analyzer crystals upon a precise indexing head capable of fixing the orientation within $\frac{1}{4}''$ (nominal) at angles varying in 1° steps. It is necessary to measure the refractive separations for several prism settings in order to determine the relative angle φ between the beam center line and the first prism surface, by fitting the separations to equation (4).

Typical intensity profiles are shown in Fig. 2 where there appear fourth- and fifth-order wavelength peaks as well as the primary and third-order peaks used to evaluate the refractive shift. The intensity points of these latter peaks were fed into a computer program which fit the profile to a profile calculated by convoluting the Darwin reflectivity function with itself. These centers were compared with some 100 centers evaluated with the centroid routine of Thomson & Yap (1968), and were found to agree within one standard deviation. Best fit of the nearly triangular data peaks, after automatic correction for the fourth order contaminant tail, gave the peak center with an uncertainty slightly larger than that due to counting statistics. This increased uncertainty arose because of relative temperature changes of the monochromator and analyzer crystals, despite heavy thermal insulation of the massive system, resulting in drifting of the crystal interplanar spacing. Since the effective rate of change in the Bragg angle due to planar spacing changes was less than 10 msec arc h^{-1} and cycled daily with a range of less than 100 msec arc, the scanning control was programmed to repeat in four hour cycles to optimize data significance. The resulting peak separations were then averaged with the uncertainty quoted due to the distribution of values. These results are shown in Tables 1, 2, and 3 for Ge, Cu, and SiO_2 prisms, respectively.

Table 1. *Summary of experimental measurements and corrections for the germanium prism deflection of a neutron beam*

Prism angle φ(°)	Measured separation (rev) $\Delta_T(\lambda)-\Delta_T(\lambda/3)$	Measured separation (μrad)	Attenuation corrected equation (7)	Collimation corrected equation (5)	Primary deflection (μrad) ($\Delta_T(\lambda_0)$	Fitted results $N\lambda^2b/2\pi$ (μrad)
2·3975	33·012 (9)	215·91 (6)	215·88 (6)	215·80 (6)	242·78 (7)	10·1177 (29)
3·3975	23·287 (8)	152·40 (5)	152·36 (5)	152·33 (5)	171·37 (6)	10·1234 (35)
4·3975	18·003 (3)	117·85 (2)	117·81 (2)	117·80 (2)	132·52 (2)	10·1224 (15)
5·3975	14·694 (6)	96·20 (4)	96·16 (4)	96·15 (4)	108·17 (5)	10·1241 (46)
6·3975	12·430 (4)	81·38 (3)	81·34 (3)	81·34 (3)	91·51 (3)	10·1290 (34)
7·3975	10·776 (5)	70·55 (3)	70·51 (3)	70·51 (3)	79·32 (4)	10·1246 (50)
8·3579	9·511 (4)	62·28 (3)	62·25 (3)	62·25 (3)	70·03 (3)	10·1152 (43)

Thus $\langle Nb\lambda_0^2/2\pi\rangle=10{\cdot}1222$ (16) μrad. Since $\lambda_0=0{\cdot}41934$ (2) nm then $Nb=3{\cdot}6168$ (6) $\times10^{14}$ m^{-2}. Since $(Nb)_{\text{air}}=0{\cdot}0043$ (1) $\times10^{14}$ m^{-2} then $(Nb)_{\text{Ge}}=3{\cdot}6211$ (6) $\times10^{14}$ m^{-2}. Since $\varrho_{\text{Ge}}=5327{\cdot}2$ (2) kg m^{-3} then $N_{\text{Ge}}=4{\cdot}4198$ (6) $\times10^{28}$ m^{-3} and $b_{\text{Ge}}=8{\cdot}1929$ (17) fm.

Calibration of the torsion goniometer used to control the analyzer crystal was performed before and after each period of data collection with excellent agreement found when the torsion assembly was allowed to relax after initial tightening of the clamping plate on the torsion column. This calibration, effected using a He Ne laser-corner cube interferometer mounted above the torsion column, is remarkably linear owing to cancelling effects of the ball contact and torsion arm bending over the instrumental range. Results are shown in Fig. 3.

Wavelength calibration over the range of slit settings was also performed before and after data collection, again showing excellent agreement. The wavelength was determined without the prism inserted for intensity reasons, by measuring the Bragg angle of reflection in the parallel and nonparallel modes from the (111) planes of a Si crystal whose faces had been roughened to increase the reflected intensity by a factor of ten in the non-parallel or defocusing mode. This method, described before (Schneider, 1973), yields the wavelength–slit center variation shown in Fig. 4.

Analysis of results

The separations of first- and third-order refracted wavelengths were converted to radians with the calibration of Fig. 3, the corner cube separation and the laser wavelength. They were corrected for prism attenuation according to equations (6) or (8) with an insignificant error due to the assumption that the primary and third-order wavelengths suffer equal attenuations, corrected for finite collimation according to equation (5) and transformed to primary deflections $\Delta_T(\lambda_0)=\frac{9}{8}[\Delta_T(\lambda_0)-\Delta_T(\lambda_0/3)]$. From the fit of these primary deflections to equation (4) for the refractive index deviation from unity, the product Nb was extracted using the known mean wavelength $\lambda_0=$ 0·41934 (2)nm. After correcting Nb for the presence of air outside the refracting surfaces the nuclear scattering amplitudes were extracted using hydrostatically measured densities: $N=\varrho N_a/M$ where ϱ is the mass density, N_a is Avagodro's number and M is the molecular weight. These steps are summarized in Tables 1, 2, and 3.

The resulting scattering amplitude $b_{Ge}=8{\cdot}1929$ (17) fm can be compared with Shull's Pendellösung value of $b_{Ge}=8{\cdot}1858$ (36) fm which depends upon assumed values of crystal planar spacing, Foldy interaction and Debye temperature. Using a more recent determination (Deslattes, 1968; Deslattes & Henins, 1973) of the Ge lattice constant, $a_0=0{\cdot}56582$ nm, Shull's result becomes $b_{Ge}=8{\cdot}1878$ (36) fm so that our standard deviations now overlap.

Our determination of the Cu scattering amplitude $b_{Cu}=7{\cdot}689$ (6) fm, compares well with the Bragg dif-

Table 2. *Summary of experimental measurements and corrections for the copper prism deflection of a neutron beam*

Prism angle φ (°)	Measured separation (rev)	Measured separation (μrad)	Attenuation corrected equation (8)	Collimation corrected equation (5)	Primary deflection $\Delta_T(\lambda_0)$ (μrad)	Fitted results $Nb\,\lambda^2/2\pi$ (μrad)
2·578	55·288 (50)	361·74 (33)	360·98 (33)	360·87 (33)	405·98 (38)	18·160 (17)
3·578	39·912 (20)	261·38 (13)	260·63 (13)	260·59 (13)	293·16 (14)	18·217 (9)
4·578	31·271 (20)	204·85 (13)	204·10 (14)	204·08 (14)	229·59 (15)	18·241 (12)
6·578	21·868 (10)	143·32 (7)	142·55 (9)	142·56 (9)	160·37 (10)	18·238 (11)
8·578	16·872 (4)	110·39 (3)	109·63 (5)	109·63 (5)	123·33 (5)	18·182 (7)

Thus $\langle Nb\lambda_0^2/2\pi\rangle=18{\cdot}208$ (15) μrad. Since $\lambda_0=0{\cdot}41934$ (2) nm then $Nb=6{\cdot}506$ (5) $\times 10^{14}$ m^{-2}. Since $(Nb)_{air}=0{\cdot}0043$ (1) $\times 10^{14}$ m^{-2} then $(Nb)_{Cu}=6{\cdot}510$ (5) $\times 10^{14}$ m^{-2}. Since $\varrho_{Cu}=8933{\cdot}1$ (5) kg m^{-3} then $N_{Cu}=8{\cdot}4663$ (5) $\times 10^{28}$ m^{-3} and $b_{Cu}=7{\cdot}689$ (6) fm.

Table 3. *Summary of experimental measurements and corrections for the quartz prism deflection of a neutron beam*

Prism angle φ(°)	Measured separation (rev) $\Delta_T(\lambda)-\Delta_T(\lambda/3)$	Measured separation (μrad)	Attenuation corrected equation (7)	Collimation corrected equation (5)	Primary deflection $\Delta_T(\lambda_0)$ (μrad)	Fitted results $Nb\,\lambda^2/2\pi$ (μrad)
2·2226	41·338 (5)	270·40 (3)	270·35 (3)	270·23 (3)	304·01 (4)	11·735 (2)
4·2226	21·725 (4)	142·23 (3)	142·19 (3)	142·17 (3)	159·94 (3)	11·732 (2)
6·2226	14·800 (3)	96·94 (2)	96·91 (2)	96·90 (2)	109·01 (2)	11·740 (2)
8·2226	11·250 (5)	73·70 (3)	73·68 (3)	73·68 (3)	82·89 (4)	11·730 (6)

Thus $\langle Nb\lambda^2/2\pi\rangle=11{\cdot}736$ (2) μrad. Since $\lambda_0=0{\cdot}41934$ (2) nm then $Nb=4{\cdot}1934$ (11) $\times 10^{14}$ m^{-2}. Since $(Nb)_{air}=0{\cdot}0043$ (1) $\times 10^{14}$ m^{-2} then $(Nb)_{SiO2}=4{\cdot}1977$ (11) $\times 10^{14}$ m^{-2}. Since $\varrho_{SiO_2}=2648{\cdot}9$ (1) kg m^{-3} then $N_{SiO_2}=2{\cdot}6551$ (1) $\times 10^{28}$ m^{-3} and $2b_O+b_{Si}=15{\cdot}810$ (4) fm. Since $b_{Si}=4{\cdot}1485$ (8) fm then $b_O=5{\cdot}830$ (2) fm.

fracted intensity values of Shull & Wollan (1951) where $b_{Cu}=7{\cdot}6$ (3) fm and of Keating, Neidhardt & Goland (1958) where $b_{Cu}=7{\cdot}90$ (23) fm as well as our own initial unpublished refraction results at MIT using $\lambda_0=0{\cdot}2393$ nm: $b_{Cu}=7{\cdot}63$ (3) fm.

The present determination for O, $b_O=5{\cdot}830$ (2) fm agrees with the older less accurate determinations: Shull & Wollan's (1951) powder diffraction result of $b_O=5{\cdot}8$ (2) fm and the Donaldson *et al.* (1965) mirror reflection result of $b_O=5{\cdot}80$ (5) fm. The more recent mirror determination by Koester (European American Nuclear Data Committee, 1969) with $b_O=5{\cdot}75$ (4) fm and the 130eV transmission cross-section determination by Dilg, Koester & Nistler (1971), where $b_O=5{\cdot}804$ (7) fm stand lower than our result. Owing to the 4N5 purity of our SiO_2 prism, the corresponding agreement of the measured density with that calculated from the perfect crystal lattice, and the negligible corrections due to attenuation and collimation, we find no explanation for the discrepancy. Improved mirror measurements as well as Pendellösung and Christiansen filter determinations will no doubt resolve the disagreement.

The author takes this opportunity to apply the exact right prism analysis of this work to the results of the Si prism refraction reported earlier (Schneider, 1973). The refraction data have an improved fit, yielding for the Si nuclear scattering amplitude, $b_{Si}=4{\cdot}1478$ (16) fm, in even better agreement with Shull's Pendellösung value of 4·1491 (10) fm. The signs of all four scattering amplitudes have been observed to be positive, as defined by equation (1), from the direction of the prism refractive bending.

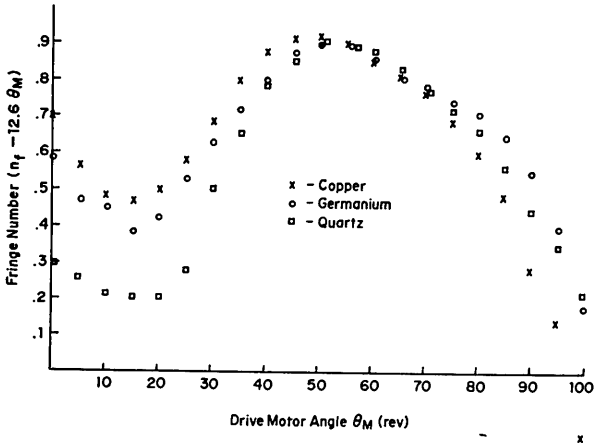

Fig. 3. Calibrations of the analyzer torsion goniometer using a laser interferometer (one fringe = 0·51904 μrad) expressed as the deviation from a linear calibration. All refraction data was taken in the motor angle range from 30 to 80 revolutions.

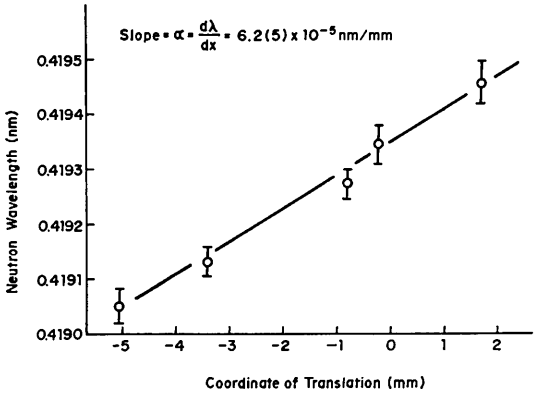

Fig. 4. Variation of the neutron beam wavelength with position across the slit following the prisms, as determined from the angle between parallel and non-parallel Bragg reflections from a silicon analyzer crystal.

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References

BATTERMAN, B. W. & CHIPMAN, D. R. (1962). *Phys. Rev.* **127**, 690–693.

DESLATTES, R. D. (1968). *Appl. Phys. Lett.* **12**, 133–135.

DESLATTES, R. D. & HENINS, A. (1973). *Phys. Rev. Lett.* **31**, 972–975.

DILG, W., KOESTER, L. & NISTLER, W. (1971). *Phys. Lett.* **36B**, 208–210.

DONALDSON, R. E., PASSEL, L., BARTOLINI, W. & GRAVES, D. (1965). *Phys. Rev.* **138B**, 1116–1119.

EUROPEAN AMERICAN NUCLEAR DATA COMMITTEE (1969). *Progress Report on Nuclear Data Research in the Euratom Community for* 1968, pp. 40–41.

FOLDY, L. L. (1951). *Phys. Rev.* **83**, 688.

GOLDBERGER, M. L. & SEITZ, F. (1947). *Phys. Rev.* **71**, 294–310.

JUST, W., SCHNEIDER, C. S., CISZEWSKI, R. & SHULL, C. G. (1973). *Phys. Rev.* B**7**, 4142–4145.

KEATING, D. T., NEIDHARDT, W. J. & GOLAND, A. N. (1958). *Phys. Rev.* **111**, 261–263.

KOESTER, L. & KNOPF, K. (1971). *Z. Naturforsch.* **26a**, 391–399.

KROHN, V. E. & RINGO, G. R. (1966). *Phys. Rev.* **148**, 1303–1311.

KROHN, V. E. & RINGO, G. R. (1973). *Phys. Rev.* D**8**, 1305–1307.

SCHNEIDER, C. S. (1973). *Rev. Sci. Instrum.* **44**, 1594–1598.

SCHNEIDER, C. S. & SHULL, C. G. (1971). *Phys. Rev.* B**3**, 830–835.

SHULL, C. G. & SHAW, W. M. (1973). *Z. Naturforsch.* **28**, 657–661.

SHULL, C. G. & WOLLAN, E. O. (1951). *Phys. Rev.* **81**, 527–535.

THOMSEN, J. S. & YAP, F. Y. (1968). *Acta Cryst.* A**23**, 702–703.