Il ressort de ces exemples que la TFP constitue un outil particulièrement adapté aux calculs cristallographiques. Le fait que, contrairement aux méthodes classiques, elle travaille sur des nombres complexes et qu'elle exige l'expansion des ensembles introduits jusqu'à une puissance entière de 2 altère à peine ses performances. Si nous considérons, par exemple, un ensemble de 3000 points traités d'une part classiquement et d'autre part par TFP aprés complémentation à 4096 points, le rapport des temps reste supérieur à 40. De telles performances rendent également précieuse l'emploi de la TFP dans la convolution de 2 fonctions pour peu que leurs supports soient assez larges.

Références

Cooley, J. W., Lewis, P. A. W. & Welch, P. D. (1969). *IEEE Trans.* E-12, 27.

Cooley, J. W. & Tukey, J. W. (1965). *Math. Comput.* **19**, 297.

IEEE Trans. (1967). AU-15, October.

Acta Cryst. (1971). A27, 494

A redetermination of the coherent neutron scattering amplitude of Cs*.

By D. E. Cox and V. J. Minkiewicz,
Physics Department, Brookhaven National Laboratory, Upton, New York 11973, *U.S.A.*

(*Received* 25 *February* 1971)

The coherent neutron scattering amplitude of Cs has been determined by a least-squares refinement of powder neutron diffraction data from CsCl at 4·2°K. The value obtained was $0{\cdot}55 \pm 0{\cdot}02 \times 10^{-12}$ cm, which differs considerably from the value currently tabulated in the literature.

There is a great deal of current interest in double halides of the type $CsNiCl_3$, which exhibit pronounced one-dimensional magnetic behavior. In the course of neutron diffraction investigations of several of these compounds, it became evident that the recently reported value of $0{\cdot}75 \times 10^{-12}$ cm for the coherent neutron scattering amplitude of cesium (Zivadinovic & Prelesnik, 1966; Neutron Diffraction Commission, 1969) was seriously in error. Preliminary calculations showed, in fact, that the intensity data in each case could be accounted for much more satisfactorily with a figure closer to the original value of $0{\cdot}49 \times 10^{-12}$ cm determined by Shull & Wollan (1951).

This is strikingly similar to the history of rubidium, for which an accurate scattering amplitude value has very recently been determined (Wang & Cox, 1970; Copley, 1970; Meriel, 1970), and a similar determination of $b_{\rm Cs}$ was therefore undertaken. Since the double halides are rather difficult to handle and characterize, CsCl was chosen as the most suitable standard. High-purity material is readily available, and also $b_{\rm Cl}$, relative to which $b_{\rm Cs}$ is determined, is known quite accurately. A polycrystalline sample of Johnson–Matthey 'Specpure' material was used (Ca, Mg, Rb, Na < 1 ppm, other cations below limits of detection by spectrographic analysis). This was heated at 550°C in an atmosphere of hydrogen chloride to convert any oxide or hydroxide to chloride, transferred to a glove bag to avoid pick-up of moisture, and loaded into an aluminum sample holder $\frac{3}{8}$ inch in diameter and 3 inches long. Powder data were collected with neutrons of wavelength 1·031 Å as described previously in the case of RbCl (Wang & Cox, 1970), except that the sample was maintained at a temperature of 4·2°K rather than at room temperature in order to minimize thermal diffuse scattering. The diffraction pattern showed a single body-centered cubic phase with a lattice parameter of $4{\cdot}08_8$ Å. A total of thirteen peaks were used in a least-squares refinement based upon intensities, the scattering amplitude of Cl being taken as $0{\cdot}963 \times 10^{-12}$ cm (Koester, 1967). Final parameter values are listed in Table 1, and the corresponding observed and calculated intensities in Table

Table 1. *Parameter values from least-squares refinement of neutron powder data from* CsCl *at* 4·2°K

Standard errors are given in parentheses.

$b_{\rm Cs}$ ($\times 10^{12}$ cm)	0·549 (7)
$B_{\rm Cs}$ (Å^2)	0·23 (13)
$B_{\rm Cl}$ (Å^2)	0·36 (8)
R value	0·025
Weighted R value	0·036

Table 2. *Comparison of observed and calculated intensities for* CsCl *at* 4·2°K

Space group *Pm*3*m*; Cs in 1(*a*) at 0, 0, 0; Cl in 1(*b*) at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. Parameters as in Table 1.

hkl	$I_{\rm calc}$	$I_{\rm obs}$
100	68	67
110	918	906
111	overlap with Al	
200	overlap with Al	
210	53	54
211	610	604
220	overlap with Al	
300 221	36	37
310	366	372
311	23	26
222	102	113
320	19	18
321	523	515
400	overlap with Al	
410 322	overlap with Al	
411 330	307	283
331	13	13
420	overlap with Al	
421	overlap with Al	
332	169	172

---

* Work performed under the auspices of the U.S. Atomic Energy Commission.

2. It should be noted that a number of peaks overlapped with Al peaks from the sample holder and could not be used in the refinement. In view of possible correlation effects, significance tests on b_{Cs} were carried out as described by Hamilton (1965). The confidence intervals at the 95 and 99% levels were found to be $\pm 0{\cdot}015$ and $\pm 0{\cdot}022 \times 10^{-12}$ cm respectively, and the final value of b_{Cs} has therefore been chosen as $0{\cdot}55 \pm 0{\cdot}02 \times 10^{-12}$ cm.

At 4·2°K, the observed temperature factors may be equated directly to the zero-point energy parameter $6h^2/4km\theta$ in the Debye–Waller expression, which yields θ values of 94 and 225°K for Cs and Cl, respectively. The Debye temperature of CsCl has been reported as 159°K from elastic measurements (Fedorov, 1968) or 166°K from specific heat measurements (*International Tables for X-ray Crystallography*, 1962).

The value of $0{\cdot}55 \times 10^{-12}$ cm for b_{Cs} is radically different from the value of $0{\cdot}75 \pm 0{\cdot}02 \times 10^{-12}$ cm recently reported by Zivadinovic & Prelesnik (1966), who also worked with CsCl. It is, however, in excellent agreement with values obtained by refinement of intensity data from some CsMX_3 compounds (Table 3), and within experimental error agrees with the value of $0{\cdot}49 \times 10^{-12}$ cm found by Shull & Wollan (1951). It is perhaps significant that Zivadinovic & Prelesnik's figure was derived in a different way, namely from measurements of the intensities of strong reflections of the type $h+k+l=2n$ only, which were then placed on an absolute basis by means of a nickel standard. This procedure requires fairly substantial absorption corrections, which must be determined experimentally. It is to be noted that an amplitude of $0{\cdot}75 \times 10^{-12}$ cm would result in an intensity ratio of about 2% for the first two peaks, while the observed ratio in their published pattern is clearly about 10%. No details of the amount of half-wavelength contamination were given, but for neutrons of 1·03 Å wavelength it is unlikely that this can account for more than a small fraction of this difference. Moreover, rough intensity calculations based on several peaks of this pattern do in fact yield a value of about $0{\cdot}5 \times 10^{-12}$ cm. It is not clear why the use of an external standard should give such a different result.

Table 3. *Values of* b_{Cs} *obtained from least-squares refinement of neutron powder data from various* Cs *compounds*

Standard errors are given in parentheses. Other scattering amplitudes taken from the Neutron Diffraction Commission (1969) compilation.

Compound	b_{Cs} ($\times 10^{12}$ cm)
CsCl	0·549 (7)
CsNiCl_3	0·551 (15)
CsCoBr_3	0·569 (23)
CsMnBr_3	0·546 (8)

References

Copley, J. R. D. (1970). *Acta Cryst.* **A26**, 376.

Fedorov, F. I. (1968). *Theory of Elastic Waves in Crystals*, p. 356. New York: Plenum Press.

Hamilton, W. C. (1965). *Acta Cryst.* **18**, 502.

International Tables for X-ray Crystallography (1962). Vol. III, p. 240. Birmingham: Kynoch Press.

Koester, L. (1967). *Z. Phys.* **198**, 187.

Meriel, P. (1970). *C. R. Acad. Sci. Paris*, **B270**, 560.

Neutron Diffraction Commission (1969). *Acta Cryst.* **A25**, 391.

Shull, C. G. & Wollan, E. O. (1951). *Phys. Rev.* **81**, 527.

Wang, F. F. Y. & Cox, D. E. (1970). *Acta Cryst.* **A26**, 377.

Zivadinovic, M. S. & Prelesnik, B. V. (1966). *Acta Cryst.* **20**, 315.

Acta Cryst. (1971). **A27**, 495

Supplement to the powder intensity project of the IUCr.

By M. Linkoaho, E. Rantavuori and U. Korhonen,
Laboratory of Physics, Helsinki University of Technology, Otaniemi, Finland

(*Received* 16 *March* 1971)

It is concluded that an accuracy of ± 1 % can be attained, at least relatively, in powder intensity measurements.

The results of the *Powder Intensity Project* of the International Union of Crystallography (Jennings, 1969) show that 'integrated intensities may not be relied on to better than 5%, even on a relative basis' and 'it does appear that the absolute measurements do not show markedly more range than the relative measurements'. In order to obtain some explanation of the discrepancies in these results the respondents were asked to answer a detailed questionnaire considering the method and the treatment of the results of their measurements. Only three laboratories were, however, able to give a thorough clarification. For that reason these participants obviously form a definite group. In this report we shall consider further results obtained in these laboratories.

Paakkari, Suortti & Inkinen (1970) have published a thorough report, where they have described the research work performed in connexion with the project. The details of their experimental arrangement have been studied very carefully. Table 3 of their report shows new absolute values of $10^4 f^2/\mu$ for the nickel sample SC. These results are shown in Table 1 column 3, normalized (the original values are in parenthesis) so that the sum of the reflexions is the same as Jennings' results for sample SL, which are in column 2.

We have, in our laboratory, devoted considerable effort to the measurements of the polarization factor (k) of our quartz monochromator. We have repeatedly measured integrated intensities from both powder specimens and from a perfect Ge crystal with and without monochromator. Our final result was $k = 0{\cdot}90 \pm 0{\cdot}02$. This value is near to that expected for a perfect crystal, $k = \cos 2\theta_M = 0{\cdot}89$, but differs from our previous result used in the project. Table 1 shows our corrected results (for sample SJ) compared with other