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An Investigation of the Crystal Structure of Mn_5Ge_3 Using Single-Crystal Neutron Time-of-Flight Techniques

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The Harwell electron linear accelerator has been used to provide a pulsed source of thermal neutrons. The intensities of many diffraction peaks from a single crystal of Mn_5Ge_3 were measured simultaneously by a time of flight technique. The various corrections to this data are described and the set of structure factors derived is compared with a calculated set. The time of flight technique using a pulsed white beam of thermal neutrons provides structure factors at several wavelengths thus giving an immediate indication of the magnitude of the extinction effect. For the majority of reflexions measured in this experiment extinction effects cannot be neglected for measurements made at wavelengths greater than 0·5 Å. After approximate correction for these effects optimum values for the two positional parameters in the $D8_8$ structure are found.

Introduction

When determining the structure of a complex molecule by diffraction techniques it is difficult to resolve sufficient lines from a powder sample so one is normally forced to use a single crystal. X-ray diffraction is the quickest and cheapest method of structure factor determination but in certain cases the distinct advantages of neutron diffraction (Bacon, 1955) are indispensable. However, intense neutron sources (reactors and others) are expensive and it is important to make maximum use of the available time. For this reason it is desirable to improve the rates of data collection using existing neutron sources and to be ready to use the next generation of intense sources which is likely to consist mainly of pulsed reactors (Brugger, 1968).

What we will call the conventional technique is shown in Fig. 1(*a*). A monochromatic (mono-energetic) beam of neutrons is selected from a well collimated beam of thermal neutrons by Bragg reflexion from a large single crystal and the small specimen crystal is set so that each plane in turn reflects this beam on to a detector. The measurement of the intensity of each peak involves an (ω, 2θ) scan over some few degrees.

In the time-of-flight technique (Buras, Mikke, Lebech & Leciejewicz, 1965; Lowde, 1956) a pulsed white beam of neutrons falls on the specimen and the diffracted neutrons are analysed as a function of direction and time of flight and hence wavelength [Fig. 1(*b*)]. Thus with a large enough array of detectors the intensities of reflexions above some minimum wavelength (and hence below some maximum Miller indices) can be determined.

Comparisons are difficult to make but we have shown (Day & Sinclair, 1968) that a one kilowatt pulsed neutron source based on a rather low-powered electron linear accelerator (linac) can give comparable results to the conventional technique on a fairly high flux (15 MW) reactor, and that the use of a chopped beam on that reactor would show a considerable gain over the conventional technique. When intense pulsed neutron sources become available time of flight techniques seem to be the only logical approach to achieving the potential gain in rates of data acquisition.

However, there are obviously many questions to be answered and many difficulties to be overcome before the crystallographer can be expected to welcome these techniques. This paper aims to show that many of these

problems are insubstantial and the rest capable of solution, also that the technique promises several incidental advantages.

Briefly, the electron pulses from the Harwell linac strike a gold target producing X-rays (Bremsstrahlung) and these produce fast neutrons by (γ,n) and $(\gamma,$fission) reactions in natural uranium. The fast neutrons are moderated in a polyethylene slab to give a burst of thermal neutrons. Diffraction of these pulsed thermal neutrons by elastic Bragg scattering from a single crystal into an array of counters, and analysis of the resultant intensity peaks in terms of neutron time of flight and Bragg angle, enables one to derive structure factors for many reflexions simultaneously.

Ideally one requires a large area coordinate detector, *i.e.* one that detects not only the arrival of a neutron but also its position. A high detection efficiency is obviously needed if one is to take full advantage of this potential increase in the rate of data collection. Lacking such a detector we have assembled a 128° arc of 32 scintillation detectors and the diffracting crystal is mounted so that a zone of reflexions coincides with the detector bank. This means that with a horizontal layer of detectors a prominent axis of the crystal is vertical.

We would like to derive $|F|_{hkl}$, the structure factor for a particular Bragg reflexion *hkl*, from our measurement of $I(\lambda;m)_{hkl}$, the number of counts or intensity in a peak detected by counter m. If the wavelength dependence of the detection efficiency is the same for all the counters, including that used to measure the incident neutron spectrum, then (Buras *et al.*, 1965)

$$F^2_{hkl} \propto I(\lambda,m) \sin^2\theta/\varphi(\lambda,0)E''(m)\lambda^4 \qquad (1)$$

where θ is the Bragg angle, $\varphi(\lambda,0)$ is the measured spectrum of the neutron flux incident on the crystal, m is 0 for a counter in the direct beam, $E''(m)$ is the relative sensitivity of the detector.

Experimental details

The experiments were performed using the Harwell linac to provide 35 MeV electron pulses one microsecond in duration at a rate of 96 per second. The mean current into the target was 20 to 25 microamperes, giving a target power of about 0·9 kW. The linac is now giving 5 kW at 192 p.p.s. into a new target; some newer accelerators give much higher outputs and projects such as the super-booster (Constantine, Crocker, Jones, Poole, Robinson & Ruffle, 1967) and the repetitively

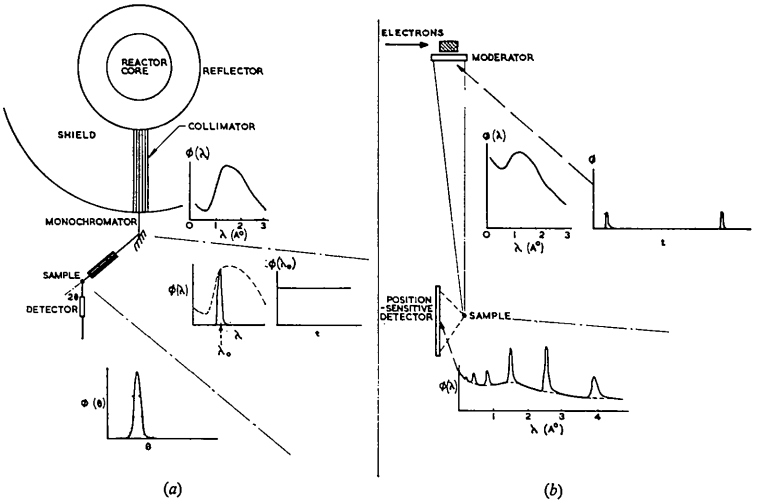

Fig. 1. (*a*) Conventional technique, (*b*) time of flight technique.

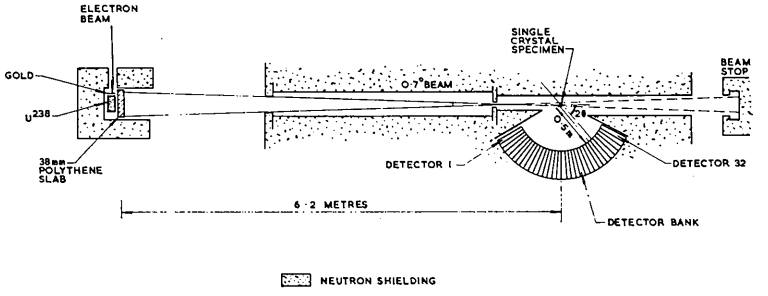

Fig. 2. Plan of the experiment.

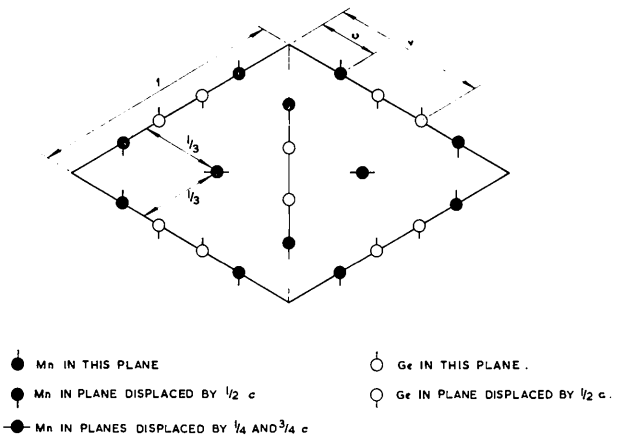

Fig. 3. Projection of the Mn_5Ge_3 structure along the [001] axis.

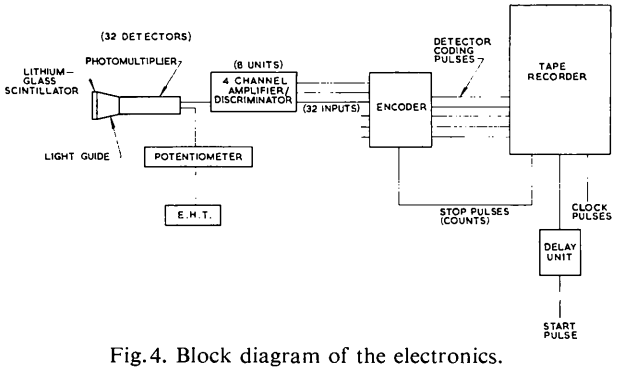

Fig. 4. Block diagram of the electronics.

pulsed test facility (Fluharty, Simpson, Russell & Morris 1967) would give gains of over 1000.

The air-cooled target consists of two three-millimetre layers of gold and a cylinder of natural uranium six centimetres in diameter and ten centimetres long. This target yields fast neutrons and these are thermalized in a slab of moderator which is put as close as possible to the target.

We have described elsewhere (Day & Sinclair, 1969*a*) the optimization of moderators for pulsed neutron sources. In this work we required the maximum intensity of thermal neutrons, so we used a 44 mm slab of polyethylene moderator. The measured peak widths at half the maximum intensity varied from 12 microseconds at 0·5 Å and 45 microseconds at 1 Å to 85 microseconds at 2 Å, the corresponding times of flight were 850, 1700 and 3400 microseconds so that the resolution was around 2%. Since there were normally only three or four reflexions on each detector and usually these were several orders of a single reflexion and hence well spaced in time, this was adequate resolution. The diffraction peaks were symmetrical in time at small wavelengths but asymmetric and dominated by the exponential decay of the thermal neutron pulse at longer wavelengths. If this exponential decay is a problem it can be reduced in the wavelength region of interest by cooling the moderator but with some loss of intensity.

The geometry of the experiment is shown in Fig. 2, the beam onto the sample has angular spreads of $\frac{2}{3}°$ horizontally and $1\frac{1}{3}°$ vertically. The flight path is lined with borated wax and boron carbide in resin collimators.

The specimen was a pillar of Mn_5Ge_3 0·83 cm high and approximately 0·38 by 0·27 cm in section. The crystal is of the $D8_8$ type [space group $P6/mcm$ (D^3_{6h})] with lattice parameters $a=7{\cdot}170$, $c=5{\cdot}043$ Å (Castelliz, 1953). The projection along the [001] axis (Amark, Boren & Westgren, 1936) is shown in Fig. 3; the parameters u and v are given by Castelliz as 0·25 and 0·61.

The detector bank consists of 32 square lithium glass scintillators (35 × 35 × 3 mm) each coupled to a 25 mm diameter (EMI9524 B) photomultiplier. The scintillators (Thorn GS20 containing 7·8% lithium enriched to 96% in the ^{6}Li isotope) are each joined to a Perspex light guide and the light guide to a photomultiplier using a silicon grease. Each scintillator/light guide unit is surrounded by a crumpled aluminum foil reflector and light-sealed to the photomultiplier housing with a rubber 'O' ring; each detector is therefore optically separate from its neighbours. The detectors are mounted in a light-tight box with a thin aluminum front window with the scintillator glasses arranged in an arc on a 0·5 m radius. Each scintillator subtends 4° horizontally and vertically at the crystal mounting position and since the dead spaces between the glasses are very small (less than 0·2 mm) the whole arc subtends 128° at the crystal.

A block diagram of the electronics is given as Fig. 4. The counts from a 24 hour run are recorded on one inch magnetic tape by writing the time of flight and detector number for each event. This tape is analysed in ten minutes by sorting the counts into a computer store and writing the 32 resultant spectra on to half inch magnetic tape for further analysis.

The total flight path is 6·73 metres, including 0·5 metres from the specimen to the detector. The neutron pulses from the source are 10,400 microseconds apart so that overlap occurs only for neutrons of wavelengths greater than 6·1 Å. We initially record the spectra in 2048 four microsecond channels after a 300 microsecond initial delay: on analysis this is usually reduced to 512 16 microsecond channels. Thus the observed wavelength range is from 0·2 to 5 Å.

Measurements

Counter sensitivities $E''(m)$ can be determined either by putting a small neutron source at the specimen position or by scattering the incident beam from a vanadium specimen (Day & Sinclair, 1969*b*). The neutron scattering from vanadium is almost entirely incoherent and hence nearly isotropic. The counter sensitivities for this bank of detectors were stable and could be measured to 2%. Future work with large area

detectors should reduce the errors due to this factor.

The obvious way to measure the incident spectrum $\varphi(\lambda,0)$ is to put one of the detectors in the beam. Unfortunately these detectors are γ sensitive and the 'γ flash' from the target saturates the scintillator at short times and it appears to take some time to recover (Day, Johnson & Sinclair, 1968). More recent work has shown that a spectrum measured with a BF_3 proportional counter and corrected with the calculated counter efficiencies for the BF_3 detector and the lithium glass scintillator agrees with the spectrum derived by applying an absorption correction to the spectrum of the neutrons scattered by a thin vanadium sample. This latter spectrum has therefore been used to correct the Mn_5Ge_3 data (Fig.5).

The dead space between detectors is less than 0·2 mm whilst the diffracted spot is about 1 cm wide. An experiment in which several diffraction spots were observed on single counters and then on the joins between these counters and the adjacent ones showed the count loss to be not more than 1 % and it has been neglected.

If we had a large area position-sensitive detector it would be possible to mount the crystal in any position and then to index the measured diffraction peaks. In this pilot experiment using a relatively small array of detectors the crystal had to be accurately positioned so that a zone of reflexions lay fully on the detectors. The pillar shaped sample was therefore mounted approximately vertically and the intensities of several strong reflexions were measured at several settings of the two goniometer arcs. The resultant plots of intensity *versus* angle were flat-topped since the vertical spread of the diffraction spots was about half the detector height. The arcs were then set so that reflexions all along the detector bank were centred. This setting up involved perhaps 12 runs each lasting between 4 and 12 hours but we amphaize that this wuuld be unnecessary with a larger area detector.

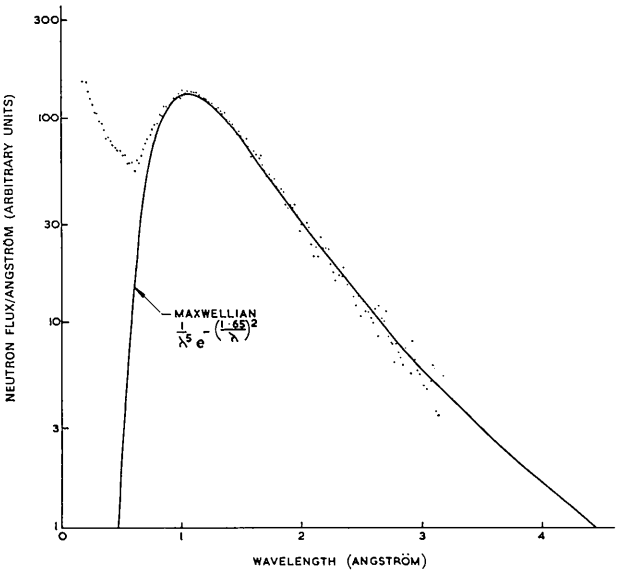

Fig. 5. Incident neutron spectrum.

The crystal was positioned and five 18 houı runs carried out followed by vanadium scattering and neutron source runs to intercalibrate the detectors. Slx weeks later the crystal was returned to the same position for a further fine 18 houı runs. Later still the crystal was remounted and fresh positioning runs performed. A set of nine 15 hour runs was then made but with rather lower linac currents than before.

In each of these three sets of data the retults of each run were finally recorded on $\frac{1}{2}$ inch magnetic tape and summed. For each detector we then had the counts in 512 sixteen microsecond time channels and a typical

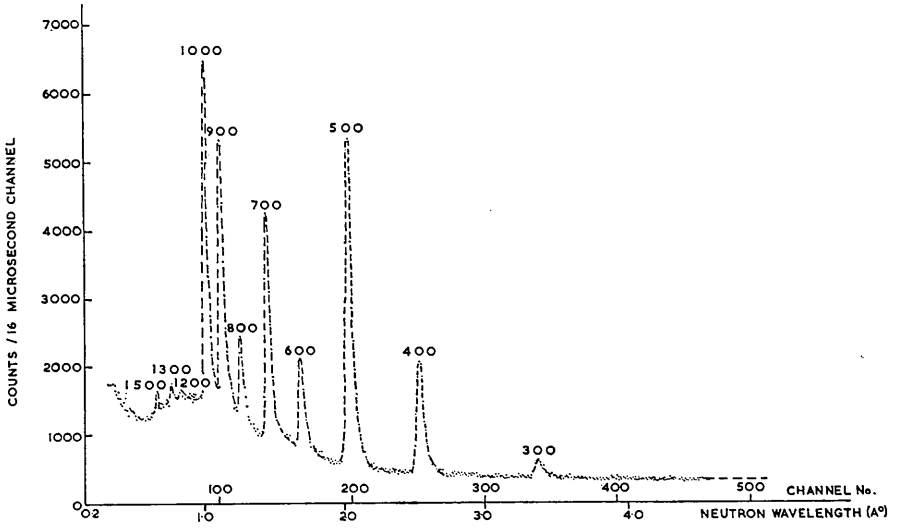

Fig. 6. A typical diffracted neutron spectrum.

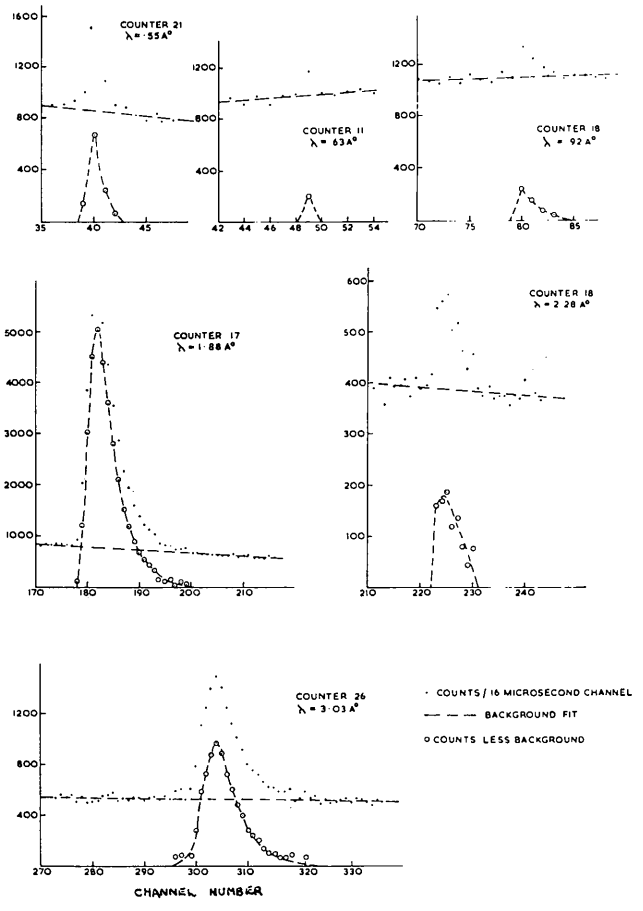

Fig. 7. Typical peaks and background fits.

spectrum from a single detector is shown in Fig. 6. The limits of the wavelength bands including significant peaks were punched on cards together with counter efficiencies and the incident spectrum shape. A computer program was written to fit a linear background under each peak (Fig. 7). This background was subtracted from the total count to give $I(\lambda,m)$ the number of counts in each peak. The five highest channels in the peak were then fitted to a parabola to give the wavelength at the maximum and the structure factor was calculated using the expression given as equation (1).

θ, the Bragg angle, is only known to within two degrees because of the relatively coarse angular resolution of the detector bank. The output from the program includes a plot of the reflexions in reciprocal space; the known reciprocal lattice (Fig. 8) can then be superimposed to index the reflexions. Combining the lattice parameters of the specimen, which are known from X-ray data, with the Miller indices it is easy to calculate the plane spacing d and since λ is known to within 1 %, θ can then be derived with an error of less than $\frac{1}{2}°$. Thus we can recalculate F^2 with an error determined mainly by the statistical error on the measured number of counts and the 2 % error in the counter efficiency.

A sample of the results is given in Table 1. The first two sets of runs included 160 peaks, 96 of which were partial peaks which occurred on two counters. Thus there were 112 reflexions observed, 48 crossed the join between two counters and 64 were on one only. For the third set of runs the comparable figures are 100 peaks, 70 reflexions, 30 on joins and 40 on one detector only.

Comparison of the first two sets of results (Table 2) shows good agreement. The differences are of the same order as the statistical errors, despite some changes in the measured counter efficiencies between the two occasions. The third set of runs was normalized to the mean of the first two sets, on the basis of the assumption that there was little extinction on the weaker reflexions. When the mean of the first two sets is compared with the final set of results the same reflexions were measured at very different wavelengths and there are significant differences between the results.

Part of this difference is due to the strong neutron absorption of the manganese which becomes large for long wavelength neutrons. If one can reasonably make a correction assuming that the path length in the crys-

Table 1. *Some typical results, set* 2

Reflexion	λ	Counts	Background subtracted	θ	% error
$\bar{1}$ 3 0	4·500	198	1332	73·4	27
$\bar{3}$ 20 0	0·641	311	1806	68·4	20
$\bar{2}$ 10 0	1·243	691	4902	66·5	15
$\bar{2}$ 12 0	0·998	1404	12753	63·5	12
4 5 0	0·744	1345	7894	27·9	10
4 7 0	0·706	1580	4705	33·3	7
2 6 0	1·1225	3200	7099	40·7	4
$\bar{1}$ 5 0	2·462	7377	9992	65·3	2
3 2 0	0·873	52699	22663	17·8	0·6
5 10 0	0·546	1164	3421	35·6	8
0 16 0	0·631	190	979	54·4	24
2 8 0	0·923	450	3321	42·9	19
1 4 0	1·884	33164	16220	44·0	0·8
1 3 0	2·279	960	3096	41·4	9
1 1 0	3·031	8036	12004	25·0	2

tal is equal to the diameter of a circle of the same cross sectional area the correction is 15% at 1 Å, 31% at 2 Å and 50% at 3 Å. After this correction has been applied to the results there is still a difference between the two sets which is correlated with scattered intensity and wavelength thus indicating that significant extinction corrections are needed.

Because of the known symmetry of the crystal the data can be condensed:

$$F^2_{hk0}=F^2_{kh0}$$

$$F^2_{\bar{h}k0}=F^2_{k\text{-}h,h,0.}$$

Thus we have measured F^2 for each reflexion at up to five different wavelengths.

A set of structure factors was calculated using the structure given by Castelliz (1953) and Amark, Boren & Westgren (1936) with various values of the parameters u and v.

$$F^2_{hkl}=\{\sum b\cos 2\pi(hx+ky)\}^2 .$$

The values of the coherent neutron scattering amplitude used were $-0{\cdot}37\times10^{-12}$ cm for manganese and $0{\cdot}84\times10^{-12}$ cm for germanium (Bacon, 1955). The calculated values of F^2 were corrected for the temperature effect assuming a Debye temperature of 340° K.

The measured values were then normalized to the calculated values of F^2 assuming that there was no extinction on the weaker reflexions. The best fit with the experimental results was achieved with $u=0{\cdot}23$ $v=0{\cdot}60$ and Fig. 9 shows some typical experimental results (corrected for absorption) measured at various wavelengths compared with the calculated values (corrected for temperature effects) which are plotted at $\lambda=0$. The dotted lines link the results for each reflexion, they do not imply that the extinction effect depends exponentially on wavelength. These results show the expected result that the extinction correction is greatest at long wavelengths and on intense peaks. It is also clear that extinction can only be neglected on the very weakest peaks for which the statistical errors are rather large. There are many more reflexions with extrapolated values of F^2 between 1·0 and 10 (on the graph scale) which have been omitted for the sake of clarity.

Conclusions

The results show that the structure factors for a single crystal can be measured using the time of flight method. The measured structure factors are in fair agreement with a calculated set. However, because of the uncertainties in the rather large extinction corrections required we have not yet performed a full refinement of the structure.

Table 2. *Comparison of sets* 1 *and* 2

Reflexion	Set 1	Set 2	Reflexion	Set 1	Set 2	Reflexion	Set 1	Set 2
$\bar{7}$ 22 0	12·9±8·5	13·6±8·1	0 15 0	15·0±2·4	13·3±2·9	3 12 0	4·7±2·7	3·9±2·1
			0 13 0	10·0±1·7	13·6±3·4	3 10 0	26·2±2·1	26·1±2·3
$\bar{5}$ 20 0	25·3±5·3	22·1±4·4	0 12 0	1·6±0·7	3·0±0·8	3 9 0	13·8±1·2	12·2±1·2
$\bar{5}$ 18 0	5·8±2·8	6·1±2·5	0 10 0	71·1±1·5	71·5±1·1	3 8 0	1·0±0·4	0·8±0·4
$\bar{5}$ 15 0	2·6±1·3	3·4±1·7	0 9 0	38·8±1·3	40·5±0·7	3 7 0	61·0±1·2	59·8±1·4
			0 8 0	8·6±0·3	8·6±0·4	3 6 0	37·6±1·0	35·9±1·0
$\bar{4}$ 16 0	7·1±1·6	5·2±1·3	0 7 0	20·6±0·3	20·8±0·3	3 5 0	34·6±0·7	33·1±0·7
$\bar{4}$ 14 0	10·1±1·0	9·5±1·1	0 6 0	8·0±0·2	8·8±0·2	3 2 0	72·8±0·5	71·4±0·5
$\bar{4}$ 13 0	15·7±1·4	16·2±1·5	0 5 0	32·2±0·3	33·4±0·3			
$\bar{4}$ 11 0	11·0±0·6	10·8±0·6	0 4 0	13·5±0·2	13·3±0·2	4 13 0	13·0±3·9	26·9±6·8
			0 3 0	3·2±0·2	3·1±0·3	4 12 0	16·0±4·6	27·4±4·6
$\bar{3}$ 13 0	27·1±1·4	27·5±1·7				4 10 0	13·2±2·3	12·5±2·8
$\bar{3}$ 12 0	12·4±0·8	10·5±0·7	1 13 0	25·1±2·7	19·6±2·5	4 9 0	18·0±2·0	17·4±2·1
$\bar{3}$ 10 0	34·6±0·6	36·0±0·7	1 11 0	1·9±0·7	2·4±0·8	4 7 0	30·7±1·7	35·4±2·1
			1 10 0	2·9±0·6	2·5±0·6	4 5 0	10·3±0·8	9·9±1·0
$\bar{2}$ 15 0	23·0±3·0	34·5±3·5	1 8 0	15·0±0·4	16·5±0·5	4 4 0	111·2±1·0	105·2±1·5
$\bar{2}$ 12 0	30·4±1·3	26·5±1·4	1 7 0	11·0±0·3	11·9±0·4			
$\bar{2}$ 10 0	6·9±0·5	7·2±1·1	1 6 0	1·1±0·1	0·8±0·1	5 15 0	12·2±5·5	23·5±6·6
$\bar{2}$ 9 0	0·9±0·2	1·4±0·2	1 4 0	30·3±0·3	30·7±0·3	5 13 0	9·5±3·7	23·7±15·0
$\bar{2}$ 8 0	4·2±0·2	4·0±0·3	1 3 0	11·0±0·1	12·0±0·2	5 10 0	77·5±5·7	65·8±5·3
$\bar{2}$ 7 0	7·7±0·2	7·9±0·3	1 2 0	7·0±0·2	6·7±0·2	5 7 0	16·7±1·8	17·0±2·5
$\bar{2}$ 6 0	1·8±0·2	1·4±0·3	1 1 0	14·9±0·2	15·1±0·1	5 5 0	25·9±4·8	26·8±2·1
						5 4 0	10·1±1·5	7·6±1·7
$\bar{1}$ 17 0	16·3±3·5	6·3±2·7	2 15 0	12·3±2·8	7·3±3·3	5 3 0	29·6±2·0	
$\bar{1}$ 14 0	17·1±1·5	21·3±2·1	2 13 0	27·3±3·5	23·7±3·3			
$\bar{1}$ 12 0	3·8±0·9	6·0±1·0	2 10 0	40·5±1·9	34·3±1·9	6 6 0	21·7±5·8	25·5±3·9
$\bar{1}$ 11 0	2·1±0·4	3·4±0·5	2 8 0	10·0±0·7	8·1±0·8	6 3 0	20·1±3·4	29·4±4·4
$\bar{1}$ 9 0	11·8±0·4	12·1±0·5	2 7 0	1·3±0·3	1·6±0·25			
$\bar{1}$ 8 0	7·2±0·3	8·4±0·4	2 6 0	7·3±0·2	7·4±0·3	7 8 0	9·6±3·4	7·1±3·9
$\bar{1}$ 7 0	0·3±0·1	0·5±0·1	2 5 0	11·8±0·3	11·8±0·2	7 6 0	6·4±3·2	9·9±3·2
$\bar{1}$ 5 0	19·2±0·3	20·9±0·4	2 4 0	2·1±0·2	1·4±0·2			
$\bar{1}$ 4 0	7·6±0·2	7·6±0·3	2 3 0	50·4±0·4	49·4±0·3			
$\bar{1}$ 3 0	3·7±0·6	3·9±0·7	2 2 0	3·5±0·2	4·2±0·1			
			2 1 0	11·4±0·1	11·2±0·1			

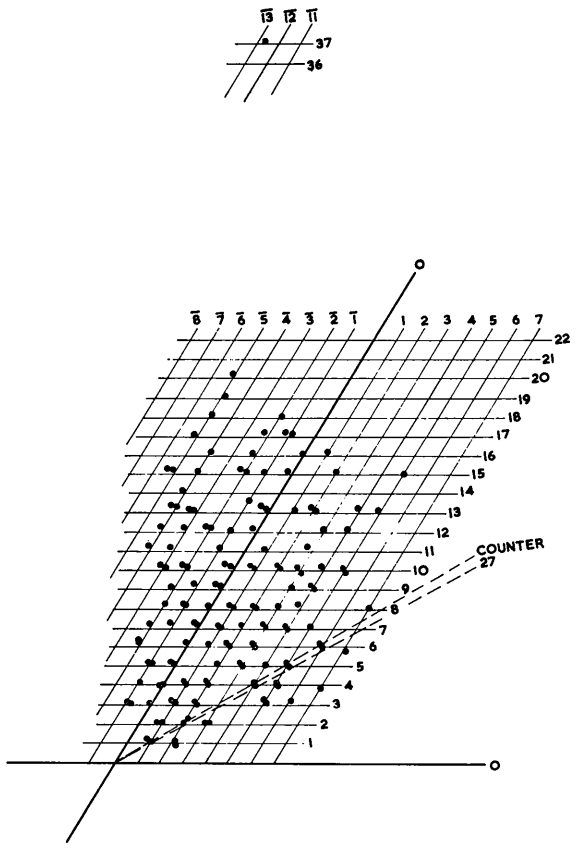

Fig. 8. Set of results (1) superimposed on the (001) plane of the reciprocal lattice for a hexagonal crystal.

The results show that the time of flight technique not only gives valid results but additional information on the variation of measured structure factors with wavelength, which enables one to estimate the size of the extinction correction. We intend to study the limit of accuracy for the technique using a simple ionic crystal of low absorption and known extinction.

The authors would like to thank Dr J. B. Forsyth for providing the crystal, D. A. G. Johnson for assistance with the experiment, Dr M. J. Poole for his continuing interest, and the operating staff of the Harwell Linac.

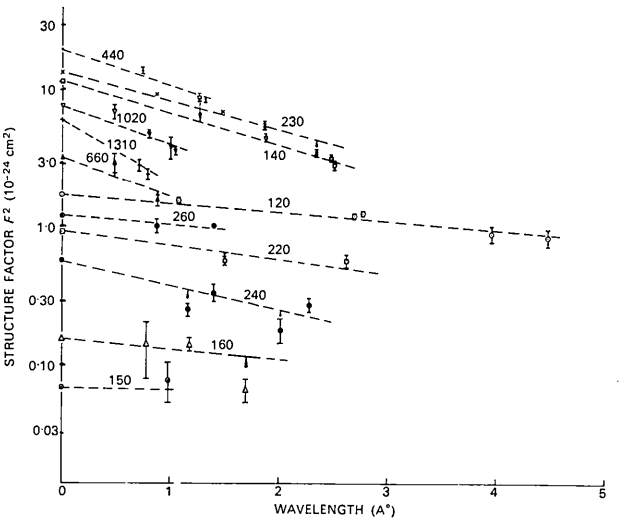

Fig. 9. Comparison of measured and calculated structure factors.

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