

D1A, A HIGH RESOLUTION NEUTRON POWDER DIFFRACTOMETER WITH A BANK OF MYLAR COLLIMATORS

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This paper describes a first attempt at following the design criteria set out earlier for a high resolution conventional powder diffractometer. An existing machine, D1A, has been modified using a bank of ten high pressure ^3He counters and almost perfect 10' mylar foil collimators. The system is more successful than earlier multicollimator arrangements because each of the collimator/counters is virtually identical; this permits automatic addition of the intensities so that a single high resolution profile, up to X40 times as intense as on the original diffractometer, is obtained just as easily as on a single counter machine. A comparison is made with the other powder diffractometers, D1B and D2 at the ILL.

1. Introduction

In an earlier paper¹⁾ the design of a conventional neutron powder diffractometer was discussed. It was shown that the resolution could be improved to the limits imposed by the powder particle size, while at the same time the effective intensity could be increased by the two orders of magnitude needed for the more complex organic and inorganic structures which could then be examined using Rietveld's profile refinement technique^{2,3)}.

Unfortunately it was not possible to obtain a beam position on the high flux ILL reactor for this machine, and a guide tube position had to be used instead. The design resolution then had to be lowered by a factor of two to compensate for the fact that the effective intensity from the guide tube is only about 10% of that from the reactor itself. (This loss factor of ten is due to loss factors of five in vertical divergence and two in the guide.) The guide tube position is then, for this type of machine, approximately equivalent to a beam tube position on a medium flux reactor, e.g. of the DIDO type. It follows that the high resolution diffractometer described in this paper could be duplicated on most of the reactors used throughout the world for neutron beam research.

2. The D1A geometry

The guide tube transmits about 50% of thermal neutrons within a small solid angle ($11' \times 11'$ for 1.4 Å neutrons). The collimation in the horizontal plane is then already better than normally used, so no additional Söller collimator α_1 is required. In the vertical plane it would of course be desirable to increase the divergence to several degrees, as is possible

on a reactor beam tube, to obtain an intensity gain of an order of magnitude or more. This is more difficult to do on a guide tube, but we will describe a solution to this problem in our next paper.

A squashed germanium [551] monochromator is

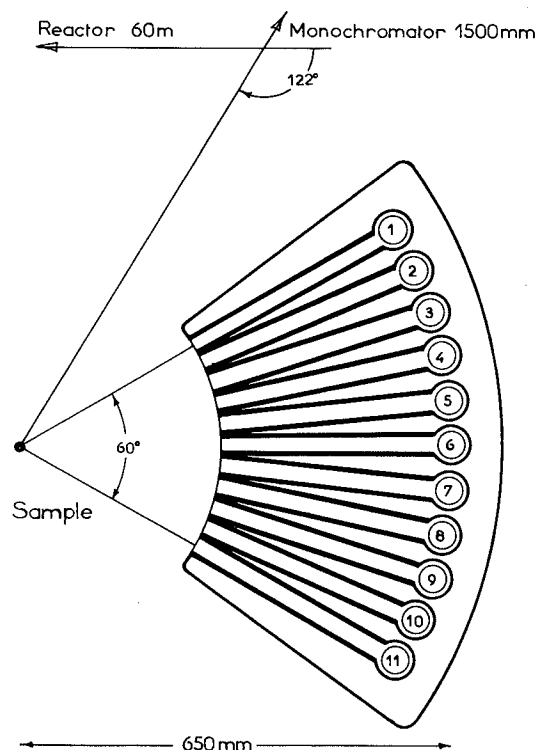


Fig. 1. Schematic diagram of the D1A multicollimator diffractometer. The large monochromator take-off angle means that the diffraction pattern is focussed for the parallel geometry shown ($2\theta = 122^\circ$). The counter bank can be swept through 0° to $2\theta = 160^\circ$ for the highest angle counter, usually in steps of 0.05° .

used in reflexion at a take-off angle at $2\theta_M = 122^\circ$ to give $\lambda = 1.384 \text{ \AA}$ (fig. 1): the idea is to extend the region of focussing to large values of $\sin \theta/\lambda$ in reciprocal space, where high resolution is most needed. The $[551]$ plane was chosen instead of the earlier proposed $[533]$ for three reasons. Firstly, the best results are obtained when germanium is squashed along $[110]$, and a $[110]$ crystal will give $[551]$ in reflexion if it is rotated by only 8.05° toward $[001]$: secondly the planes $[331]$ giving $\lambda = 2.268 \text{ \AA}$ and $[771]$ giving 0.994 \AA can be obtained with additional small rotations of 5.21° and -2.28° : finally, it was felt that the extra range in $\sin \theta/\lambda$ available with 1.384 \AA instead of 1.509 \AA from $[551]$ would make the shorter wavelength a little more attractive for most applications.

Since it is necessary to choose a counter divergence α_3 approximately equal to α_1 , a fixed value of $10'$ was chosen. It would be expensive to duplicate a bank of Söllers with a different collimation, and in any case doubling the divergence only increases the peak intensity by a factor of $\sqrt{2}$. Again following the earlier reasoning, no α_2 Söller is used since the effective α_2 collimation is already about $4\alpha_1$ for our usual sample of 16 mm diameter 1500 mm from the monochromator.

This monochromator-sample distance is needed with the backscattering type of geometry to allow the counter bank to swing up to angles of $2\theta = 160^\circ$, where the resolution is still quite good. The diffractometer dimensions (fig. 1) are determined by the length of the collimators, their angular separation and the shielding required. A collimator length of 320 mm with 1 mm blade separation was chosen for reasons of ease and economy of construction, and because these were the first collimators made using the mylar foil technique. It has since been shown⁴) that for $10'$ divergence the collimator dimensions can be halved with a sacrifice of only about 10% in transmission. (Alternatively, high transmission $5'$ collimators could be made the same length as the $10'$ collimators now on DIA.)

The angular separation of the collimators was fixed at 6° because we wanted to use standard 5 cm diameter ^3He counters, but it has since been shown⁵) that rectangular counters can be made at much the same cost. Rectangular counters would permit a much smaller angular separation, and therefore a larger, number of counters could be used. With 6° separation, only ten counters were chosen to fill an angle of 60° , but our experience now suggests that the original specification of 32 counters could be met and even doubled with the latest collimator and counter designs,

and that a larger angle of coverage could be used. A bank of 50 collimator/counters can be compared with the 400 counting elements used on the position sensitive detector DIA, except that with the multicollimator design the sample could have $\times 10$ times the volume for the same resolution, and container background is eliminated. Such a large collimator/counter bank would also be comparable in cost to the position sensitive detector now that collimator construction techniques have been perfected.

3. Collimator and counter performance

The Söller collimators were developed at the Rutherford Laboratory in England, and those made for DIA are described in the original report⁶). Their useful cross-section is 100 mm high by 20 mm wide, with a divergence of $10'$ horizontal and 6.6° vertical for 100 mm high counters 650 mm from the 50 mm sample. The blades are $25 \mu\text{m}$ aluminized mylar foil coated on each side with $25 \mu\text{m}$ of gadolinium oxide paint. The mylar is prestretched on frames which later become the spacers when the ends are cut off. Unlike similar designs using plastic blades⁷) it is not necessary to curve the ends of the blades to prevent curling of the foil edges. The transmission of these collimators was measured to be 96.6% with an almost perfect triangular transmission function⁶). The measured profile of one collimator rocked against another is shown in fig. 2, together with the theoretical curve for the convolution of two triangles. It is clear that even very thin gadolinium oxide coatings stop practically all neutrons at such low

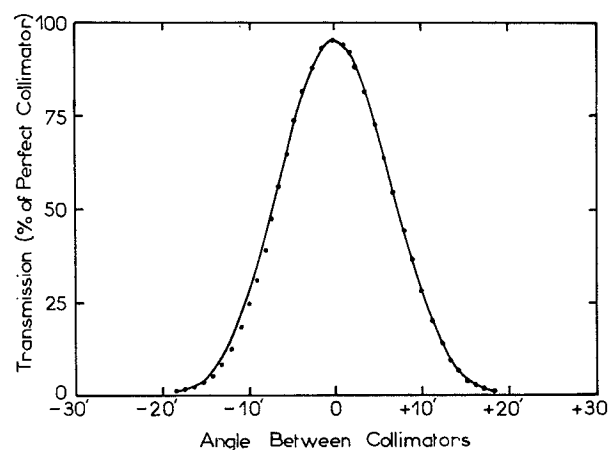


Fig. 2. Observed and calculated profiles for two $10'$ mylar collimators rocked against each other (from ref. 6). The horizontal axis gives the angle between the collimators and the vertical axis shows the transmission of the second collimator as a percentage of that expected for an "ideal" collimator.

glancing angles, and there are no apparent effects of total internal reflexion from the surfaces⁸). Collimators of this type are now available commercially through the Rutherford Laboratory, and have been ordered for several other diffractometers.

The counters are 50 mm diameter \times 100 mm long ³He at 5 atm. Since they are used side-on, the walls have been reduced to 0.1 mm, but otherwise they are of standard design. At 1.4 Å their efficiency is more than 90%.

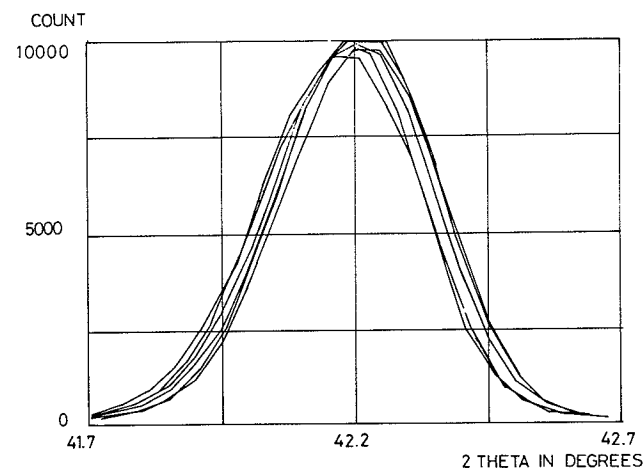
The collimators are located by pegs in holes on an aluminium plate, with no adjustment to their alignment possible. This works very well, since the standard deviation in alignment was measured to be only $\pm 0.02^\circ$. Even though this is almost half of the normal step size of 0.05° , it causes no problem with profile refinement because these small displacements of the separate profiles are corrected by interpolation before addition.

The original shielding consisted of 15 cm of paraffin wax in aluminium containers, followed by 1 mm of cadmium, but because of gaps between the welded aluminium parts, this shielding has been replaced by 5 cm of B₄C loaded araldite. The B₄C/araldite is moulded in two pieces around the counters and collimators, a procedure which eliminates all gaps. The counters are again sheathed with cadmium, except for apertures in close contact with the collimators. The protection against thermal neutrons is increased with the new shielding and that against epithermal neutrons reduced, since the latter are a less serious problem near the guide tube position, which is 60 m from the reactor itself. The weight of the shielding has been kept to a minimum so that no special modifications have been necessary to support it on the standard ILL diffractometer base.

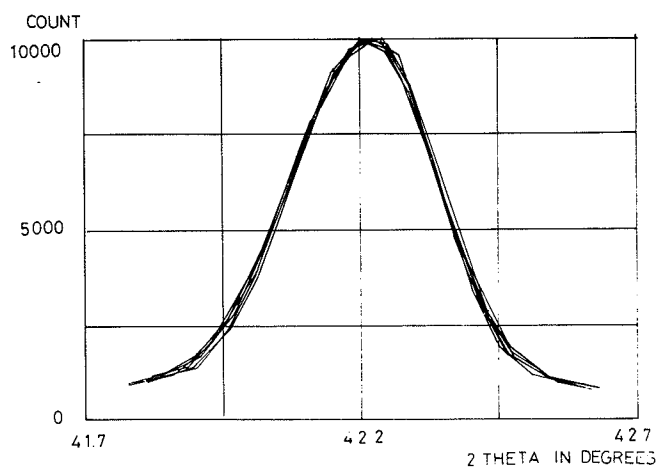
Fig. 3a shows peak data observed with the first six counters and collimators before any corrections for efficiency and displacement. (The remaining four counters had not arrived when this data was collected.) Fig. 3b shows the same peaks after correction. The fact that the differences in efficiency and alignment between counters are very small and can be simply corrected is the key to the success of the addition process. Earlier attempts to add the results of a bank of counters, for example on D2, have been less successful because variations between collimators have resulted in large differences in the profiles. This problem has led to suggestions that the profile refinement program be modified to accept data from all of the counters separately, but this would be very wasteful of computer time, and is now unnecessary. Any variations in effi-

ciency with time are no more serious with a multicounter arrangement than with a single counter, where it is assumed that the efficiency does not change during a scan. In fact, with the multicounter, efficiency variations and time dependent noise can be detected by routine comparison of the separate profiles in the course of their addition on the computer.

Fig. 3 also shows that each of the new collimator/



(a)



(b)

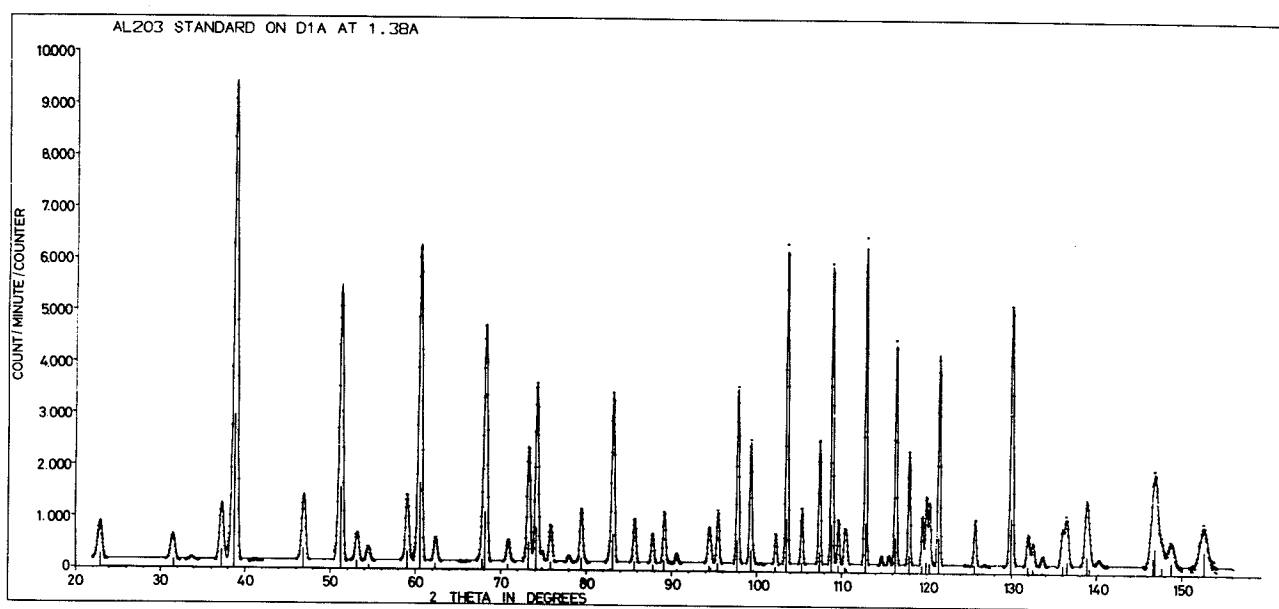
Fig. 3. (a) The $|311| \text{Al}_2\text{O}_3$ peak as seen by the first six collimator/counters. The errors shown here in collimator alignment ($\pm 0.02^\circ$) and counter efficiency ($\pm 4\%$) are corrected by interpolation and scaling. (b) As a result of these corrections, the separate profiles are now effectively identical, and can be added to produce a single composite profile for refinement. The whole process is of course automatic, being part of the data retrieval program. This program also compares each of the profiles to pick up any systematic errors (e.g. time dependent noise).

counters is about $\times 4$ as efficient as the old collimator/counter. This factor consists of $\times 2.5$ for the increased vertical divergence between sample and counter, and the remainder for the increased transmission of the collimator itself. With ten counters, the new D1A is therefore up to $\times 40$ times as efficient as the old single counter machine for experiments in which scans of at least 60° are required. For experiments in which only a single peak is to be studied or for which an exact $\theta-2\theta$ scan is required, the gain factor is reduced to $\times 4$ since only one counter can be used. In either case

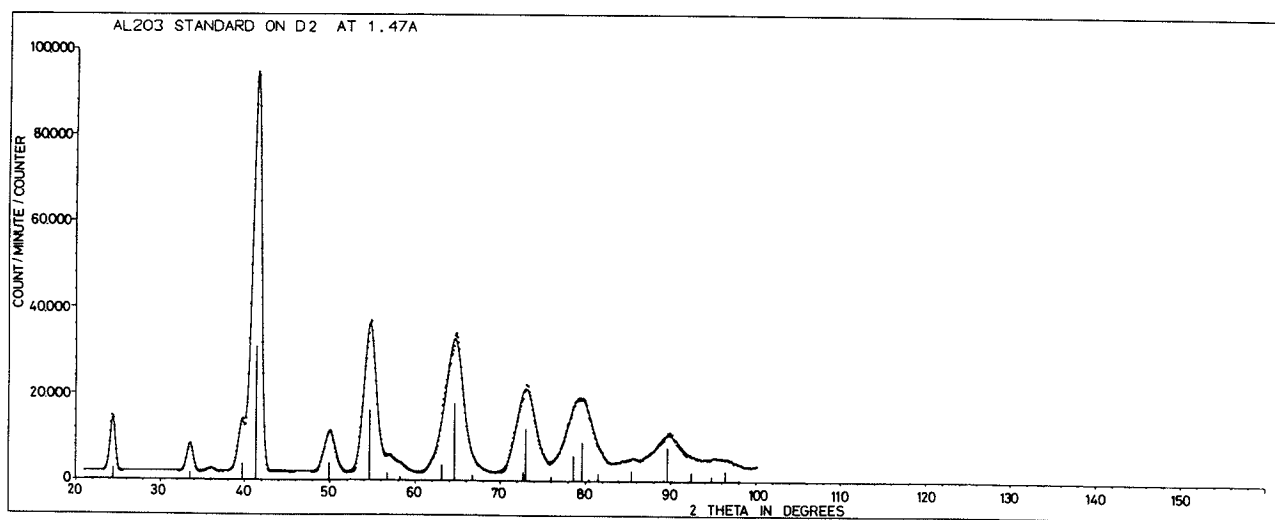
the use of mylar collimators and side-on counters is obviously advantageous.

4. Control and data collection

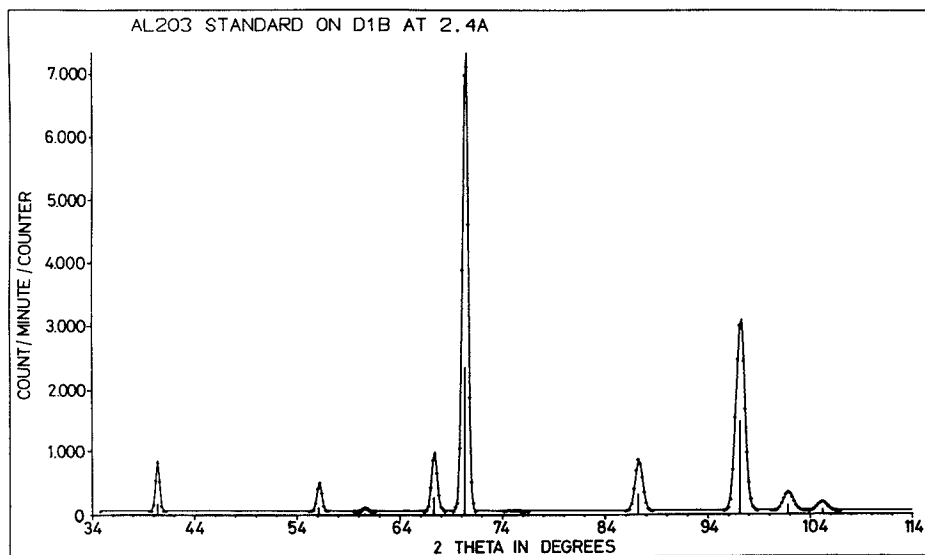
D1A shares a remote Télémécanique computer system (Carine II) with five other diffractometers. Scan data is entered on a local terminal and the results are saved on magnetic tape for automatic daily transfer to a disk on the central PDP10. Data can also be printed and plotted on line if required, and the system is arranged so that this is done while the diffractometer



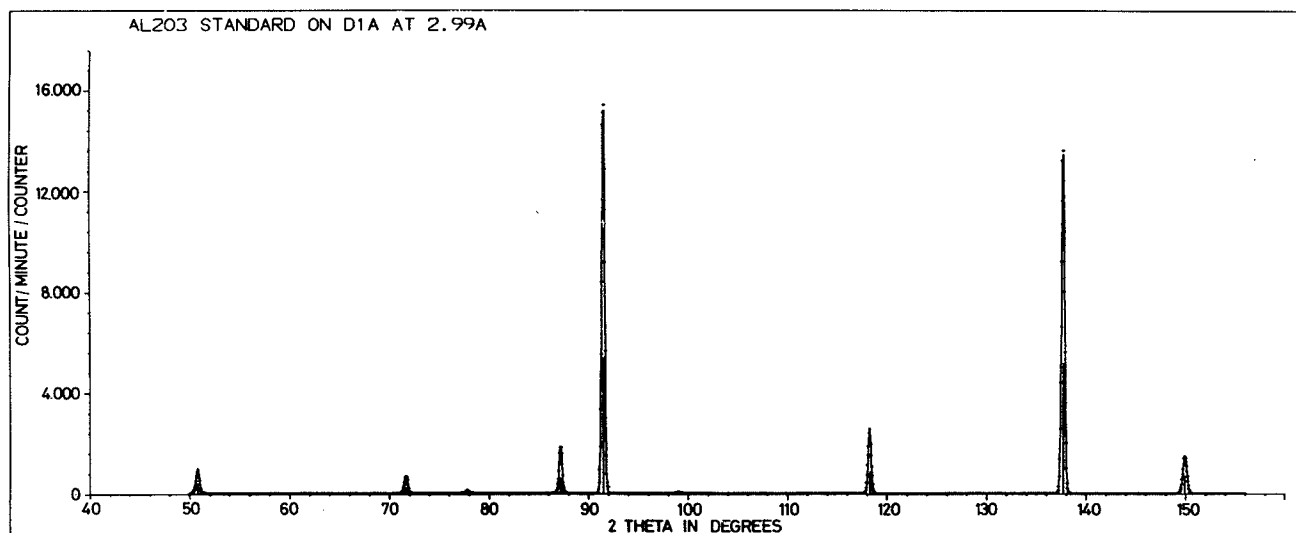
(a)



(b)



(c)



(d)

Fig. 4. (a) Observed and calculated composite profile for the Al_2O_3 standard sample on D1A at $\lambda = 1.384 \text{ \AA}$. The six separate profiles have been averaged in the regions where they overlap (most of the range), so the statistics are equivalent to a single profile six times as intense. (b) Observed and calculated profiles for the same sample on D2 at $\lambda = 1.48 \text{ \AA}$. The intensity per counter is very high, but only one of the four counters could be used at this time because of differences between the collimators. This problem will be overcome when new mylar collimators are fitted as on D1A. (c) Observed and calculated profiles for the standard sample on D1B at $\lambda = 2.4 \text{ \AA}$. All 400 channels are collected simultaneously, so the intensity is effectively 400 times

that shown for the individual counters. (d) Observed and calculated profiles for the same sample on D1A at 2.99 \AA . The resolution at low $\sin \theta/\lambda$ is much improved, and this is useful for magnetic structures, for deciding the symmetry of "pseudo-symmetric" structures, and for help in finding a suitable starting model for profile refinement. The individual peak intensity is almost doubled again in going to $\lambda = 5.7 \text{ \AA}$: the diffraction pattern is not shown for this wavelength, since the dispersion is so great that only the first peak of Al_2O_3 can be seen out to $2\theta = 160^\circ$. Such a long wavelength, with a beryllium filter, could be used to resolve structures with cell dimensions of up to 50 \AA .

is counting at the next position. All ten counters can be plotted so that after a scan of only 6° a 60° profile is obtained. In this way the multicollimator diffractometer D1A can simulate the operation of the position sensitive detector D1B, and quickly produce a picture of a large part of the diffraction pattern.

Once on the DEC-10 disk, the data can be manipulated on-line using a self-teaching system of conversational programs. Profiles can quickly be added (or subtracted in the case of background runs), checked against each other, plotted, and set up for profile refinement. Using this system it is just as easy to work with a large bank of counters as with the usual single counter. This is important because otherwise the experimentalist would be overloaded with so much data that it would be impossible to use it effectively.

5. Profile refinement with a bank of counters

The Al_2O_3 sample, provided by Andresen and Sabine⁹⁾ for the Neutron Diffraction Commission Intercomparison project, is a sintered pressed cylinder of 13.5 mm diameter \times 16 mm height. With this sample it is possible to compare the intensity and resolution of D1A with the more than thirty other diffractometers throughout the world which have been involved in the intercomparison project, as well as with the other powder diffractometers, D1B and D2 at the ILL.

A profile refinement on D1A using the old single counter with a standard 10' cadmium collimator has already been reported¹⁰⁾ and a high precision result was obtained. It is interesting to compare this result with that from refinement of the composite data obtained by adding the counters in the way described above.

In both cases, the quantity minimized is the statistical sum

$$\chi^2 = \sum \frac{(y_{\text{obs}} - y_{\text{calc}})^2}{y_{\text{calc}}}$$

summed over all points in the profile. This is of course a least-mean-squares refinement with weights

$$y_{\text{calc}}^{-1} \approx y_{\text{obs}}^{-1}$$

For convenience, so that all parts of the summed profile are on the same scale, independent of the number of counters contributing, the sum y_{obs} can be replaced by the counter average $\bar{y}_{\text{obs}} = y_{\text{obs}}/n$. Then the equation for chi-squared becomes

$$\chi^2 = \sum \frac{n(\bar{y}_{\text{obs}} - \bar{y}_{\text{calc}})^2}{\bar{y}_{\text{calc}}}$$

In practice, we simply replace the weight $1/y_{\text{calc}}$ by n/\bar{y}_{calc} for each point, and then Rietveld's profile refinement program³⁾ can be used without further modification.

With an effective intensity $\times 4 \times 6$ as large with the six available counters, we counted for only 15 s at each point instead of the 1 min needed before. The composite profile, averaged over the six counters, is shown in fig. 4a, together with that computed.

Apart from the intensity gain factor, there are some small differences between this profile and that recorded earlier. The wavelength is now 1.384 Å, so that about one third more reflexions can be recorded out to $2\theta = 156^\circ$. Since a larger number of reflexions are crowded into the same angular range, and the vertical divergence is larger, some peaks are not quite so well resolved as before. This occurs mainly at low angles where there are in any case fewer peaks, and according to the criteria set out earlier¹⁾ this is a desirable compromise: using profile refinement, more information can be extracted than with the longer wavelength.

The *R*-factor for structure factors is now only 0.9% compared with 1.35% with the single counter data, and χ^2 is 5.68 compared with 8.45 obtained earlier. Since the whole pattern was collected in only one quarter of the time to compensate for the $\times 4$ gain of each collimator/counter, the improvement in the fit corresponds to the improvement in statistics obtained by averaging the six counters. A further improvement is expected with the increase to ten counters, but already the remaining small discrepancies between calculated and observed profiles are probably mainly due to model limitations and systematic errors, rather than to the statistics.

The improvement in the statistics is reflected in the lowering of the standard deviations calculated for the parameters. Except for the temperature factors, we obtain agreement within one standard deviation for all structural parameters (table 1).

These calculated standard deviations, now less than ± 0.0005 Å for the atom positions, are very small out the results appear to be quite reproducible, even on quite different diffractometers.

6. Comparison with D1B, D2 and other diffractometers

For the diffractometer intercomparison project, Andresen and Sabine⁹⁾ chose the strongest Al_2O_3 reflexion, (113). Since this is a low order reflexion, it can be reached by all machines, even those operating with long wavelengths, and it is well resolved on machines operating with small monochromator angles.

TABLE 1

Structural parameters for Al_2O_3 determined on the new and old D1A, and on D1B and D2. The standard deviation calculated for the cell edge a_0 assumes that the wavelength is known precisely, and does not include any systematic errors, due to sample misplacement for example. The rhombohedral angle, scattering length b_{A1} and structural parameters $u(A1)$ and $u'(0)$ are in surprisingly good agreement for three such very different diffractometers. The Debye-Waller factors $B_{ij} = 8\pi^2 \langle U^2 \rangle_{ij}$ contain systematic errors due to uncertainty in the background subtraction for D2, and because only low order reflexions are available on D1B. The R -factor for D1B is small because there are so few reflexions to fit, and on D2 because the low resolution again means a small amount of data for the number of parameters.

	New D1A	Old D1A	D1B	D2	Other work (X-ray)
a_0	5.13448 (5)	(5)	(60)	(80)	
α	55.270 (1)	55.270 (1)	55.269 (6)	55.268 (6)	55.266 ¹²⁾ 55.277 ¹²⁾ 55.289 ¹³⁾
b_{A1}	0.349 (2)	0.349 (4)	0.345 (16)	0.345 (3)	
b_0	0.580				
$U(A1)$	0.35222 (7)	0.35219(12)	0.35225(36)	0.35261(14)	0.352 ¹⁴⁾
$U'(0)$	0.55635 (8)	0.55621(17)	0.55645(51)	0.55596(23)	0.556 ¹⁴⁾
$B_{11}(A_1)$	0.22 (2)	0.19 (5)	0.00 (50)	0.08 (7)	
$B_{12}(A_1)$	-0.07 (1)	-0.05 (2)		-0.02 (4)	
$B_{11}(0)$	0.22 (1)	0.34 (3)	-0.10 (45)	0.08 (4)	
$B_{33}(0)$	0.15 (2)	0.22 (3)		0.05 (8)	
$B_{12}(0)$	-0.06 (1)	-0.14 (3)		-0.09 (5)	
$B_{13}(0)$	-0.06 (1)	-0.08 (1)		0.03 (3)	
R_F	0.96	1.35	0.50	0.71	
χ^2	5.68	8.45	5.70	4.48	

However, it occurs far below the focussing angle on D1A, and although it is still well resolved, it does not indicate our best resolution, which occurs for angles near $2\theta = 120^\circ$. We could obtain a much better result for this one reflexion by choosing a wavelength much larger than our 1.384 Å, since the reflexion intensity would then be increased, and a larger would also move (311) closer to our focussing angle.

The advantages of using long wavelengths (~ 2.6 Å) for magnetic structures have been described by Loopstra¹¹⁾, and a number of machines have been designed on these principals, including D1B. Essentially, for a given reflexion (d -spacing) both the resolution and the sample scattering power increase with $\tan \theta$, and of course $\sin \theta$ increases with λ : the resolution is measured by $\Delta d/d = \Delta \theta / \tan \theta$, and the reflexion intensity goes as $\lambda^3 / \cos \theta \sin 2\theta = d^3 \tan \theta$.

On D1A we have sacrificed these advantages in working with a relatively short wavelength, because for crystal structure refinement it is important to collect data at large values of $\sin \theta / \lambda$ in reciprocal space. We compensate for the intensity lost by colli-

mation by using a bank of high efficiency collimator/counters. For intercomparison then, we propose to retain the reflexion (311), but to normalize the sample scattering power on the different diffractometers by dividing the (311) intensity by $\tan \theta_{311}$. The resolution will be quoted as the average $\Delta d/d$ over the one third of the diffraction pattern for which it is smallest. Then we find the values given in table 2 for the intensities and resolutions of several diffractometers.

If we compare $I/\tan \theta$ we see that the ILL machine D2 has more than twice the intensity of the second best machine, that at the Brookhaven National Laboratory. However, if one remembers that the multidetector D1B has 400 counting elements, if then has effectively more than twenty times the intensity of a single counter on D2! This advantage will be reduced when D2 is equipped, like D1A, with a bank of collimators for which the separate profiles can be added; but for equal sample sizes, D1B will still be the most intense diffractometer for experiments in which the whole counter bank can be used. Of course, for optimum resolution, a sample diameter of 5 mm rather than

TABLE 2

Al_2O_3 standard sample (113) peak intensities per counter, I_{peak} , corrected for the "Lorentz factor" $1/\tan \theta$, together with $\Delta d/d$, the smallest resolvable difference in lattice spacing d . D1A has good resolution $\Delta d/d = \Delta \theta \cot \theta$ because the focussing angle $\theta = 61^\circ$ is large and the collimation $\Delta \theta \lesssim 10'$ is good. With guide tube losses, the intensity per counter is much the same as for an instrument such as PANDA on the medium flux reactor at Harwell, but the effective intensity of D1A is much higher because there are ten counters which can be added. D1B, with 400 counting elements is the most efficient machine, but the best resolution can only be obtained with a sample 1/8th as large. D2 has the highest intensity for single counter applications (e.g., θ - 2θ scans).

	λ (Å)	I_{peak} (c/min)	$I/\tan \theta$	$10^3 \Delta d/d$
D1A	1.38	$9\,500 \times 10$	$26\,700 \times 10$	2
D1B	2.40	$7\,200 \times 400$	$11\,600 \times 400$	8
D2	1.48	95 000	250 000	18
PANDA I	1.48	7 500	19 700	8
Petten	2.57	15 900	24 000	7
BNL	2.41	71 000	114 000	10
Oak Ridge	1.07	11 700	42 400	18

13.5 mm must be used on D1B, and this would reduce the intensity by a factor of more than seven. As well, D2 is superior for experiments in which only a small part of the counter bank is useful, for example when a θ - 2θ scan is required for experiments with a magnetic field in the scattering plane.

It is interesting to note that, with its bank of ten counters and very much higher resolution, D1A already has the same effective intensity as a single counter on D2. The focussing monochromator, soon to be installed, will give a further factor of $\times 10$ in intensity, whereas on D2 a much smaller gain could be expected. D1A should then have much the same effective intensity as D1B, even for the same sample size.

From the actual diffraction patterns (fig. 4) we can readily appreciate the qualitative differences between D1A, operating with a large take-off angle and a short wavelength, and a machine such as D1B, operating with a long wavelength, or D2, with a small take-off angle. In either case, much more structural information is contained in the diffraction pattern obtained on D1A. We might expect then that D1A will give a much more precise structure for the Al_2O_3 standard. It is therefore a little surprising that the atom positions found from the D1B and D2 data are in such excellent agreement with those found on D1A (table 1). Certainly the standard deviations are larger, and the vibrational amplitudes are less well defined with the

D1B and D2 data, but the overall agreement is better than might at first be expected. On D2 this is a tribute to the efficiency of the profile refinement technique, and we can conclude that the resolution of D2 is probably sufficient for such a simple structure.

The high resolution of D1A becomes necessary only for more complex structures, where the average peak intensity falls with the multiplicity and with the cell volume. This means that high resolution is attractive only if high intensity can be obtained as well, and this is why on D1A more emphasis has been given to increasing the intensity than to further improving the resolution. Exceptionally, when it is difficult to find a good starting model for the profile refinement, high resolution again becomes important to enable individual peaks to be identified.

In fact, for these purposes, and for magnetic structures, the resolution of D1A at low $\sin \theta/\lambda$ can be further improved by selecting a much longer wavelength, as recommended by Loopstra¹¹). Since the monochromator take-off angle is so large, a wavelength of 3 Å is obtained from the (311) germanium planes (fig. 4d). Even more interestingly, a wavelength of 5.7 Å has been obtained from the (111) planes¹⁵); this gives a resolution sufficient to just resolve all the lines in an hypothetical 50 Å cell! Furthermore, even though we do not use a cold source, the peak intensity for a given reflexion almost doubles in going from 1.38 Å to 3 Å, and again in going from 3 Å to 5.7 Å. This is because of the transmission characteristics of the guide tube (the solid angle of transmission goes as the square of the wavelength), and because of the $\tan \theta$ factor mentioned earlier.

Fortunately, order contamination is not a problem at these long wavelengths, since we are still able to use the (odd, odd, odd) germanium reflexions. Then $n\lambda$ and $\frac{1}{2}\lambda$ are forbidden, while $\frac{1}{3}\lambda$ is only 0.5% at $\lambda = 3$ Å, again because of the guide tubes properties. For $\lambda = 5.7$ Å, $\frac{1}{3}\lambda$ and $\frac{1}{4}\lambda$ are eliminated by a beryllium filter. Such long wavelengths will become more important for large structures when a molecular constraints program for profile refinement becomes available.

For normal profile refinement, the best compromise between intensity and resolution has already been discussed¹); it was emphasized that with high resolution it is necessary to obtain intensity gain factors of two orders of magnitude over the usual single counter diffractometer. On D1A this has been achieved, using a bank of high efficiency collimators, and eventually a focussing monochromator.

As it is now, D1A does not reflect the advantages of

the high flux reactor, and could be duplicated on the many medium flux reactors normally used for neutron diffraction. However, it proves all of the techniques needed to build the machine described in our earlier paper¹⁾ with even higher resolution and intensity. As well, it extends considerably the range of problems which can be attempted, to include quite complex structures, hydrogenous materials, and samples as small as 100 mg at pressures of up to 30 kbar or temperatures up to 2500°C. It gives an indication of the wider horizons for crystal structure work using neutron powder profile refinement, compared to the traditional single crystal studies at standard temperature and pressure.

Many people have contributed to the earlier DIA diffractometer, especially P. Burlet, R. Chagnon, P. Convert and M. Steiner. P. Hay and the NBRU, Rutherford Laboratory have been responsible for the new collimator development, G. Gobert and the ILL workshops for the mechanics, J.-C. Falaise and J. Jacoby for the electronics and counters, and A. Bar-

thelemy and W. Kaiser for the control and data acquisition system.

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