X-ray powder diffraction data

X-ray diffraction patterns were obtained for single-phase, −200-mesh material in the range 5 to 140° 2θ at a rate of 0.01° s⁻¹ using a 173 mm diffractometer with a graphite single-crystal monochromator and a 13.0 mm θ compensating slit. The data were analyzed by Ito's method (Azaroff & Berger, 1958) and found to be consistent with a hexagonal unit cell. The resulting diffractograms were subjected to the interactive computer program INDEX (Hom, Jenkins, & Ladell, 1983) to obtain the lattice parameters. No systematic extinctions were observed in this pattern.*

With one molecule per unit cell, the calculated density is compatible with the density (approximately 3 g cm⁻³) of the other compound in this system.

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References


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A comment on The peak in neutron powder diffraction by B. van Laar and W. B. Yelon. By C. J. Howard,† Department of Physics, University of Edinburgh, Mayfield Road, Edinburgh EH9 3JZ, Scotland

(Received 11 June 1984; accepted 26 July 1984)

Abstract

It is shown that the neutron powder diffraction peak as computed by van Laar & Yelon [J. Appl. Cryst. (1984), 17, 47-54] can be rather well approximated using a normalized asymmetric peak profile as proposed by Howard [J. Appl. Cryst. (1982), 15, 615-620] or Prince [J. Appl. Cryst. (1983), 16, 508-511] together with an intensity correction factor from Cooper & Glasspool [J. Appl. Cryst. (1976), 9, 63-67]. Since the differences are material only at the lowest scattering angles (2θ < 10°), in most cases a description of the peak by one of the simpler analytic profile functions should be adequate.

In a recent paper, van Laar & Yelon (1984) computed neutron powder diffraction peaks for the case when a detector of finite height 2H rotates around a sample of finite height 2S at a distance L. van Laar & Yelon (hereafter referred to as vLY) restricted the calculation to H > S. For computation of total diffraction profiles, the diffractometer was assumed to have a resolution function R, which was a symmetric Gaussian. In this communication, we employ the vLY notation throughout.

It is of interest to compare the vLY work with other recent attempts (Howard, 1982; Prince, 1983) to describe the peak profiles associated with the use of detectors of finite height. Howard (1982) has suggested that the peak profile might be approximated by a sum of Gaussians, while Prince (1983) has suggested an Edgeworth expansion be used. Prince (1983) has shown that the sum of (five) Gaussians and the Edgeworth expansion are very closely equivalent, so it suffices to compare the vLY work with that of Howard (1982).

Howard (1982) assumed the deviation from Bragg angle 2θ = 2φ, and in particular to allow the approximation h ≈ L[2(2θ − 2φ) tan 2θ]₁/₂ in vLY equations (9). He also assumed negligible specimen height, that is 2S = 0 in vLY equations (9) and (10). Finally Howard approximated the convolution of vLY equation (16) using Simpson's rule. The result was a sum of Gaussians involving a single asymmetry parameter P = [2H/L]². Compared with the single Gaussian, this sum of Gaussians was shifted [the centroid being shifted by −(P/3 cot 2θ)] broadened and asymmetric. These are qualitatively the effects reported by vLY in their computed peaks. This being so, it seems worthwhile to compare the magnitudes of the peak shifts reported by vLY with the shifts of the sum of Gaussians, for the extreme case 2H = 100 mm, 2S = 40 mm, L = 685 mm (whence P = 0.153) discussed by vLY. The shifts reported by vLY [and in parentheses the shifts calculated from −(P/3 cot 2θ)] are: for 2θ = 10°, 0.24° (0.29°); for 2θ = 20°, 0.15° (0.14°); and for 2θ = 40°, 0.05° (0.06°). The agreement between the peaks computed by vLY and the sum of Gaussians in respect of peak shift is thus quantitatively good, the discrepancy at 2θ = 10° being accounted for by the fact that for a highly asymmetric peak such as shown...
in vLY Fig 7 the centroid shifts rather more than the maximum. It is concluded that the peak shapes obtained by vLY are quite well approximated using the analytic profile functions proposed by Howard (1982) or Prince (1983), and the latter have the advantages that they contain a single refinable asymmetry parameter and are easier to compute. It should be noted that none of the work reviewed allows for an asymmetric diffractometer resolution function $R(A,2\theta)$ as results (for example) from vertical divergence of the primary beam.

Whereas Howard (1982) and Prince (1983) have produced normalized functions to describe peak shapes, vLY have obtained an intensity correction factor from their computations. This factor is shown in Table 1 of vLY. This may be compared with the correction factor $c_1$ obtained by Cooper & Glasspool (1976), derived on the basis that the detector of height $2H$ intercepts the Debye–Scherrer cone in an arc of length greater than $2H$, and which in vLY notation is $\sin^{-1}(H/L \tan 2\theta)/(H/L \tan 2\theta)$. The comparison is again made for the case $2H = 100$ mm, $2S = 40$ mm, $L = 685$ mm.

$^*$See note added in proof.

The correction factors reported by vLY (and the correction factors calculated from the formula above) are: for $2\theta = 6^\circ$, $1.1465$ ($1.105$); for $2\theta = 10^\circ$, $1.0350$ ($1.031$); for $2\theta = 20^\circ$, $1.0059$ ($1.007$); for $2\theta = 40^\circ$, $0.9994$ ($1.001$). While the vLY correction factors are undoubtedly more accurate (and in particular the values for $2\theta = 90^\circ$ can be confirmed without recourse to numerical integration), it is clear that the Cooper & Glasspool (1976) formula provides a good practical approximation to them.

Note added in proof: van Laar & Yelon (private communication) have pointed out that the Cooper & Glasspool (1976) correction gives a less accurate approximation in the case $2H = 50$ mm, $2S = 50$ mm, $L = 500$ mm.

References


Crystal Data


Revised crystal data for magnesium dichloride, MgCl$_2$$^*$ By J. DORREPAAL, Koninklijke/Shell-Laboratorium, Amsterdam (Shell Research BV), PO Box 3003, 1003 AD Amsterdam, The Netherlands

(Received 1 March 1984; accepted 1 May 1984)

Abstract

A refinement of the cell parameters of MgCl$_2$ has been achieved by powder X-ray diffraction. The diffraction pattern was obtained using a standard PW-1050 Philips diffractometer operating in the step-scan mode and equipped with a graphite monochromator and a Xe-filled proportional counter. Cu radiation with a wavelength of 1.54056 Å was used. The specimen was placed in an air-tight sample holder in an N$_2$ atmosphere. An emission spectrographic analysis revealed a purity of >99%, while the XRD pattern showed that only a small amount of Mg(OH)Cl was present. Unit-cell dimensions of $a = 3.640(4)$ and $c = 17.673(15)$ Å have been found with $V = 234.2(7)$, $Z = 3$, $D_x = 2.339$ Mg m$^{-3}$. These values are different from those found by Ferrari, Braibanti & Bigliardi [Acta Cryst. (1963), 16, 846] using a photographic method. The difference is probably due to the higher accuracy obtained for the present data with the diffractometer.

$^*$The full text has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39430 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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