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Absolute Structure Factors for D(+)-Tartaric Acid

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A method of determining absolute structure factors from large, parallel-sided crystals is applied to a sample of D(+)-tartaric acid and is shown to give a random error in the structure factors of less than 1%.

Introduction

When structure factors are measured on a relative scale, the scaling factor is usually determined with respect to a crystal model during a least-squares process. This procedure has two main defects. (1) Different atomic models will give rise to different scattering factors which will result in differing scale factors. (2) Interactions can occur during the least-squares process between the scale factor and the scattering factors and between the scale factor and the temperature factors.

A systematic error in the scale factor can have serious consequences if accurate and detailed information is to be deduced from a difference Fourier synthesis. This error has the effect of superimposing on the difference map a positive or negative electron density whose magnitude is proportional to the true electron density and to the magnitude of the error (Stevens & Coppens, 1975) and this can mask or distort the information which is being sought.

Measurements of absolute structure factors using small, single crystals can be made (Burbank, 1965) but problems in determining the volume of the crystal and in providing a strictly monochromatic beam of uniform incident energy limit their accuracy. These problems can be overcome by using a large, parallel-sided crystal whose cross-sectional area is greater than that of the main beam, a technique investigated by De Marco (1967) in determining the atomic scattering factor of aluminium. A similar method is used in this study.

For such a crystal, the integrated intensity ϱ and the reflectivity of the plane Q, is given by

$$\varrho = QA_c .$$

A_c, the transmission factor, takes into account the geometrical aspects of the scattering as well as the absorption. Since the crystal is uniform in shape, this factor can be determined exactly, (*International Tables for X-ray Crystallography*, 1967), apart from any uncertainties in the thickness of the crystal or in the absorption coefficient. The whole of the main beam is now striking the crystal surface for all reflexions and I_0 now becomes the total energy per unit time striking the crystal and thus the energy per unit area in the main beam need not be constant over the crystal surface.

The main differences between the technique used here and that of De Marco (1967) are the following. (1) The absorption coefficient has been measured with a method described by Lawrence & Mathieson (1976). (2) The thickness of the crystal was measured with a linear differential transducer. (This value of t was the average thickness over those parts of the crystal intersected by the beam during the scattering measurements. For crystals of the order of 0·1 cm thick, an accuracy of 0·1% in each measurement should be achieved. The overall accuracy of the thickness measurement will usually depend on the uniformity of the crystal thickness.) (3) Harmonics in the main beam were eliminated with the (111) planes of a Si crystal as a monochromator. (4) Four measurements of each reflexion at φ, $\varphi+180°$, $\varphi-2\theta$ and $\varphi-2\theta+180°$ were made. The measurements did not have the same integrated intensities since they had different A_c values but they were assumed to give the same structure factor. These structure factors were averaged.

The use of large crystals increases the possibility of the scattering being non-kinematic and this limits the magnitude of the structure factors which can be measured. The identification and elimination of those reflexions suffering from extinction is best done by measuring the structure factors at two wavelengths.

Experimental

D(+)-Tartaric acid was chosen by the International Union of Crystallography as the subject of a single-crystal intensity measurement project (Abrahams, Hamilton & Mathieson, 1970). Its structure had been refined by Okaya, Stemple & Kay (1966) and again by Hamilton & Abrahams (1970) using data from the intensity project. A thin crystal of cross section 0·7 × 0·9 cm and of uniform thickness was grown from an aqueous solution at 0 °C, with the ($h00$) planes parallel to the crystal face. Its average thickness was 0·0429 (3) cm and the μt values for both Cu and Mo radiation were $\mu t(\mathrm{Cu}\ K\alpha) = 0{\cdot}635$ (5), $\mu t(\mathrm{Mo}\ K\alpha) = 0{\cdot}0680$ (5). The absorption factors were, therefore, $\mu(\mathrm{Cu}\ K\alpha) = 14{\cdot}80$ (15) cm^{-1}, $\mu(\mathrm{Mo}\ K\alpha) = 1{\cdot}584$ (15) cm^{-1}.

Integrated intensities of the $h0l$ reflexions which diffracted with Laue-type geometry were measured with a scintillation counter for both Cu and Mo radiations. Appropriate transmission factors were applied to the intensities, the structure factors were deduced and averaged over four measurements. These average structure factors are listed in Table 1.

Table 1. *Absolute structure factors measured with copper radiation*, F(Cu), *and molybdenum radiation*, F(Mo)

h	l	F (Cu)	F (Mo)
−1	1	10·3	10·6
0	1	1·0	1·1
1	1	13·4	17·5
2	1	14·1	15·5
−3	2	11·4	11·6
−2	2	7·8	7·8
−1	2	9·2	9·4
0	2	16·9	24·4
1	2	15·8	19·7
2	2	6·8	6·6
−5	3	9·0	8·7
−4	3	6·5	6·3
−3	3	11·5	11·8
−2	3	15·3	17·1
−1	3	13·8	18·0
0	3	3·3	3·3
1	3	8·9	9·0
2	3	4·3	4·0
4	3	6·9	6·7
−4	4	10·1	10·4
−3	4	6·0	5·9
−2	4	3·2	3·3
−1	4	14·2	17·6
0	4	15·4	18·0
1	4	6·3	6·2
2	4	11·6	11·7
3	4	11·6	11·9
−4	5	15·6	16·8
−3	5	10·9	11·2
−2	5	8·4	8·4
−1	5	6·0	5·8
0	5	3·3	3·1
−4	6	5·9	5·7
−3	6	3·8	3·6
0	6	6·5	6·6

Comparison of the data from the two wavelengths shows that the structure factors measured with Cu radiation are less than those measured with Mo radiation for those structure factors greater than ten. This suggests that these reflexions are suffering from extinction and they can therefore yield no information as to the value of the absolute kinematic structure factors. Of the remaining structure factors, those measured with Cu radiation appear to be about 1 % greater than those measured with Mo radiation, possibly due to a systematic error in I_0 or μ. It must also be remembered that no corrections have been applied for either Compton scattering or thermal diffuse scattering, effects which will tend to reduce the magnitudes of the structure factors.

The variation in the crystal thickness (0·7 %) and uncertainties in the μt values (0·8 %) give a maximum random error in A_c of 1·3 %. Thickness variations must also cause errors in the ϱ values and measurements of ϱ taken at φ and $\varphi + 180$, which should have the same integrated intensities, suggest errors of about 2 % in each ϱ value. Since each structure factor is the average of four ϱ measurements, the total random error in each structure factor will be less than 1 %.

This method could be applicable to any material provided the necessary crystal can be grown. Particularly with organic materials, there should be a sufficiently large number of small 'kinematic' reflexions which can be measured to reasonable accuracy. These structure factors can then be used to place a data set, measured conventionally with a small crystal, on an absolute scale.

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