Short Communications

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Crystallographic and proton magnetic resonance data for L-cysteic acid monohydrate.

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Crystals obtained from the aqueous solution of L-cysteic acid monohydrate belong to space group $P2_12_12_1$ and have cell dimensions $a,b,c = 6{\cdot}955$, 18·968, 5·322 Å. Proton magnetic resonance measurements demonstrate that the proton–proton vector of the water of hydration in these crystals is oriented at 36° to [010] and 87° to [001] and has a magnitude of 1·59 Å.

Crystals of L-cysteic acid monohydrate, $HOOC{-}CHN(H_2){-}CH_2SO_3H.H_2O$, were grown from the aqueous solution of the commercial-grade compound at 22°C. Optical goniometric examination shows the crystals to be orthorhombic prisms elongated along [001] having well developed (010), (110) and (031) faces and a cleavage plane parallel to (010).

Precession photographs of the $h0l$, $h1l$, and $0kl$ levels taken with Cu $K\alpha$ X-radiation confirm the goniometric results and give the cell dimensions $a,b,c = 6{\cdot}955 \pm 0{\cdot}006$, $18{\cdot}968 \pm 0{\cdot}019$, $5{\cdot}322 \pm 0{\cdot}004$ Å. Systematic absences of $h00$ when h is odd, $0k0$ when k is odd and $00l$ when l is odd indicate that the crystallographic space group is $P2_12_12_1$. The density of the crystals measured by flotation in a mixture of bromobenzene and methylene iodide is found to be 1·75 g.cm^{-3}. This is in satisfactory agreement with the value 1·770 g.cm^{-3} obtained from the above cell dimensions, the molecular weight (187·17) and the assumption that there are four formulas per unit cell.

The proton resonance measurements were made in order to establish the magnitude and orientation of the p–p (proton–proton) vectors belonging to the water of hydration. These measurements were made at room temperature on a single crystal of L-cysteic acid monohydrate weighing 0·63 g. The resonance absorption spectra were observed with a standard 'wide-line' Varian spectrometer operated at 16 MHz. Because the crystal has more than one p–p direction, the method employed to determine these directions is the one which has been prescribed by Murty & El Saffar (1962) for complex hydrates. This utilizes the fact that a distinct maximum line-pair separation is observed in the resonance diagram when the external field is less than 30° from a p–p direction. Two such maxima appear in the resonance diagram of Fig. 1 where the rotation plane is (001). The problem of locating the direction of a vector becomes a matter of locating the positions of the maxima produced by a proton pair in two resonance diagrams. In this study, the second resonance diagram is obtained from a rotation plane containing [001] and making an angle of 34° with [010]. Having found the p–p direction from these two planes of rotation, the p–p distance, r, may be calculated from the following equation due to Pake (1948):

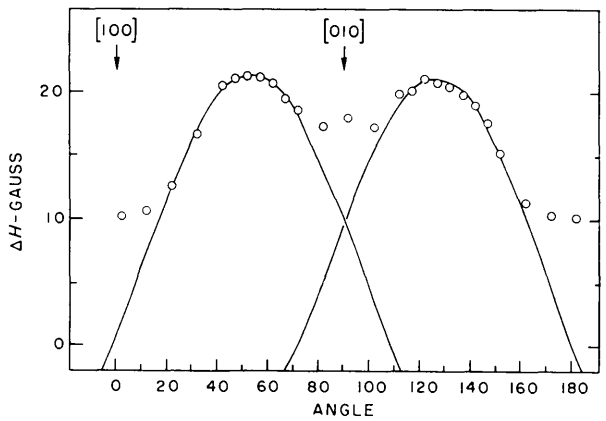

Fig. 1. Resonance diagram obtained for L-cysteic acid monohydrate when rotated in the (001) plane. The circles are experimental points. The smooth curve is a plot of the angular dependence of ΔH calculated from equation (1) and the experimentally determined p–p vectors.

$$\Delta H = 3\mu r^{-3}(3\cos^2\theta - 1)\,. \qquad (1)$$

where ΔH is the line-pair separation in a given orientation, θ is the angle the vector makes with the direction of the external field, and μ is the magnetic moment of the proton. The results indicate that there are four p–p directions related by point group symmetry *mmm* so that they may be represented by only the p–p vector taken in the positive quadrant. This vector makes angles of $36° \pm 1°$ and $87° \pm 1°$ respectively with [010] and [001]. The p–p distance is found to be $1{\cdot}59 \pm 0{\cdot}01$ Å. These above results should be helpful in determining the X-ray structure of L-cysteic acid monohydrate and the positions of the H atoms belonging to the water of hydration.

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