

Cinchonamine zinc chloride complex,
ZnCl₂ · 2(C₁₉H₂₄N₂O · HCl)

This compound was prepared by the method described by Boutroux & Genvresse (1897). It crystallized from aqueous solution in rectangular tablets slightly elongated along [100] and bounded by the forms {010}, {001}, {100}, {011} and {201}. The crystals extinguish sharply when viewed on the three principal faces in plane polarized light. The Laue symmetry is *mmm* and the crystal system is therefore orthorhombic (pseudo tetragonal). The space group is uniquely determined as *P*2₁2₁2₁ by the systematic absences. It is proposed to carry out further work on this compound.

Cinchonamine, C₁₉H₂₄N₂O

The long needle-like crystals of this compound are bounded by the forms {1120} and {1120}, and terminated by the forms {1011} and {1011}. Friedel (1887) found that sections of cinchonamine crystals, cut perpendicular to the needle axis, exhibited sectoral extinction when examined in plane polarized light. Friedel's observations have not been reproducible on the crystals of cinchonamine examined in this laboratory. Sections, perpendicular to the needle axis, of all the crystals examined in plane polarized light show no extinction directions whatsoever, indicating that the crystals are uniaxial. This conclusion is confirmed by the X-ray investigation. X-ray photographs showed that the Laue symmetry is $\bar{3}$ and hence the crystal system is trigonal. Oscillation photographs

and a Weissenberg photograph of the zero layer, obtained with the needle axis as axis of rotation, show only those reflexions for which $h-k+l = 3n$, when indexed on a hexagonal lattice. Hence the space group is either *R*3 or *R*3̄. Since cinchonamine is optically active (Henry, 1949, p. 465) the space group is uniquely determined as *R*3. The dimensions of the hexagonal unit cell are $a = 15.9$, $c = 17.2$ Å. The dimensions of the rhombohedral unit cell calculated from the hexagonal dimensions are $a = 10.8$ Å, $\alpha = 94.5^\circ$. This value of α agrees, within the limits of its accuracy, with that quoted by Groth (1906-19, vol. 5, p. 941), namely, $\alpha = 94^\circ 51'$. The observed density is 1.17 g.cm.⁻³, and the number of molecules per unit cell is calculated to be 8.95. This indicates that the unit cell contains nine molecules.

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Some remarks on J. Gillis's paper on phase determination by the Harker-Kasper method.

By Y. OKAYA and I. NITTA. *Department of Chemistry, Faculty of Science, Osaka University, Nakanoshima, Osaka, Japan*

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Gillis (1948) has determined the signs of U_{h0l} 's, the unitary structure factors, of monoclinic oxalic acid dihydrate by means of the Harker-Kasper method. By the linear inequalities which we have found recently (to be published soon) we have obtained the same results as those of Table 4 in Gillis's paper, except an apparent misprint in the sign of (503̄). At the final stage of the sign determination, there are still involved two parameters, a and c , which must necessarily remain undetermined. As shown by Gillis, the parameter c can be assigned arbitrarily depending upon the choice of origin. However, the other parameter, a , which is concerned with the signs of U_{h0l} 's with $h+l = 2 \times \text{odd}$, and which, according to Gillis, would be determined by

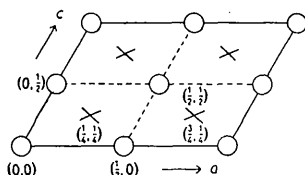


Fig. 1. Centres of symmetry in *b*-axis projection. Circles: $a = -1$ system; crosses: $a = +1$ system (only in *b*-axis projection).

Table 1. Choices of parameters and related centres of symmetry

	Choice	Related centres of symmetry
Put	$a = -1, c = +1$	for (0, 0) or ($\frac{1}{2}, \frac{1}{2}$),
then	$a = -1, c = -1$	for (0, $\frac{1}{2}$) or ($\frac{1}{2}, 0$),
	$a = +1, c = +1$	for ($\frac{1}{2}, \frac{1}{2}$) or ($\frac{3}{2}, \frac{3}{2}$),
and	$a = +1, c = -1$	for ($\frac{1}{2}, \frac{3}{2}$) or ($\frac{3}{2}, \frac{1}{2}$).

chemical or other considerations, is in fact also arbitrarily assignable, because, in the case of the *b*-axis projection, there are other possibilities in choosing the origin, as will be seen from Fig. 1 and Table 1. This is the reason why we have stated that two parameters must remain, and thus, in applying any inequality method, one must consider at the outset how many arbitrary parameters must remain in the final results.

If we take $a = -1, c = -1$, we obtain perfect coincidence with the results obtained by Robertson & Woodward (1936).

References

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