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The structure of benzamide.

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The structure of benzamide has been determined using both two- and three-dimensional photographic intensity data. A note submitted earlier, giving details of the results of the two-dimensional analyses about the [*b*] and [*c*] axes, was withdrawn at the suggestion of the Editor pending the publication of the results of the analysis of the three-dimensional data. The results reported were in general agreement with those published later by Penfold & White (1959) which probably represent the practical limit of refinement using two-dimensional photographic data. The atomic co-ordinates from the three-dimensional photographic data Blake (1959) show some interesting differences compared with the two-dimensional work; these results together with others derived from three-dimensional proportional counter intensity measurements will be published later.

References

Penfold, B. R. & White, J. C. B. (1959). *Acta Cryst.* **12**, 130.

Blake, C. C. F. (1959). Ph.D. Thesis. University of Birmingham.

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New use for old X-ray tubes.

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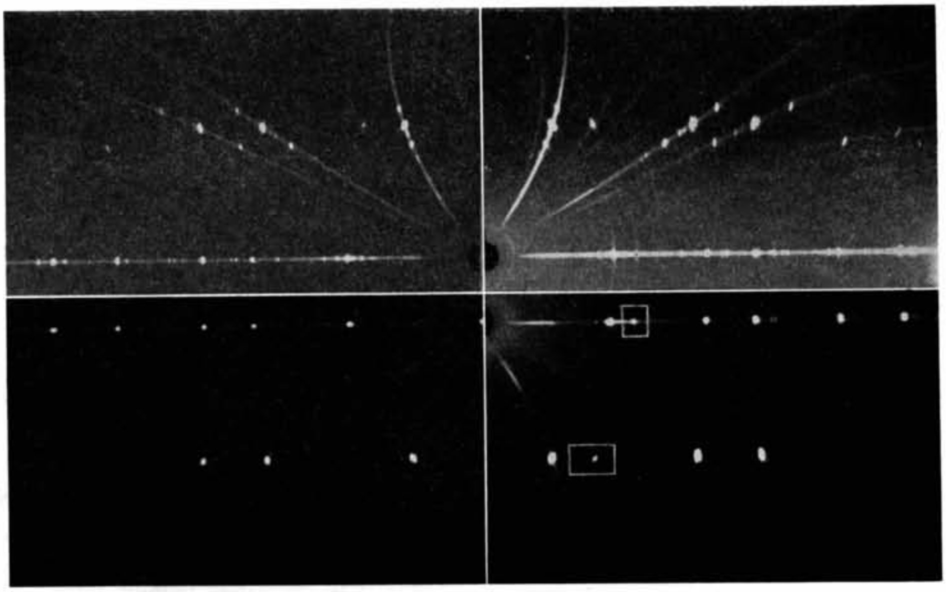
We have recently found a new use for a heavily contaminated sealed-off X-ray tube which was giving, in addition to Cu $K\alpha$ and β, considerable amounts of NiK and WL radiations.



A. *Top right*

Diamond matrix containing Ni-rich inclusions with parallel f.c.c. structure. Rotation about [011] with radiation from Cu target contaminated with Ni and W. Weak powder pattern from unknown impurity with larger spacings also visible. Attention is particularly drawn to the Ni $K\alpha$ and Cu $K\beta$ spots on opposite sides of Cu $K\alpha$. The differential absorption of Ni $K\alpha$ and Cu $K\beta$ by diamond and the inclusion respectively proves that the inclusion contains Ni.

B. *Top left*

A similar pattern with the same crystal and tube but with less exposure. The powder lines are not seen.

C. *Bottom right*

The same crystal used with a clean tube and filtered Cu $K\alpha$ radiation. The 200, (020, 002) spots from the inclusion are marked. Faint powder lines.

D. *Bottom left*

The same with less exposure. No powder lines; and inclusion spots very weak. Almost all the information given by A would be missed in D.

In the course of examining random-rotation photographs of synthetic diamonds made by the General Electric Company of New York, taken with a clean tube, we observed the presence of a satellite face-centred cubic structure, of lattice constant slightly less than that of diamond (3·539 Å cf. 3·567 Å). This structure gives reflexions very close to the positions where the diamond 200 and 420 would have been if not 'forbidden'; and its other reflexions are all just on the larger-angle side of observed diamond spots. The satellite and diamond structures are closely parallel in orientation (Grenville-Wells & Lonsdale, 1958).

A full account of this investigation will be published in the Mineralogical Magazine, but the point to which we wish now to draw attention is that by taking rotation and Weissenberg photographs with the contaminated X-ray tube, and then studying relative intensities of the Ni $K\alpha$, Cu $K\alpha$ and Cu $K\beta$ reflexions for diamond and for the satellite spots respectively it was possible

(1) to prove that the satellite was rich in nickel, since it differentially absorbed the Cu $K\beta$ radiation. This was shown much more convincingly by comparison (for diamond and the satellite respectively) of the Ni $K\alpha$ and Cu $K\beta$ spot intensities, which were of the same order, than by attempting to compare Cu $K\beta$ with Cu $K\alpha$:

(2) to estimate the approximate amount of nickel present, and the size of the grains in which it was concentrated.

The use of an X-ray tube giving many different radiations simultaneously ought, in principle, to provide the means, by a study of differential absorption, both of determining the kinds of inclusion atoms present in a known matrix, and their relative amounts, even when no absorption edge is available and when the structure of the inclusions is unknown.

Reference

Grenville-Wells, H. J. & Lonsdale, K. (1958). *Nature, Lond.* **181**, 758.

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Crystallographic data for certain alkaloids. VI. Some cinchona alkaloids.

By P. J. F. Griffiths,*
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The crystallographic data for the cinchona alkaloids given in Table 1 were obtained from oscillation and Weissenberg photographs (Cu $K\alpha$ radiation). The densities were determined by flotation. Brief notes on the appearance of the crystals and the space-group determinations are given in the text. The maximum error in the cell dimensions and observed densities is of the order of 1%, except where otherwise indicated, and interaxial angles are accurate to within 1°. Identification data for most of the compounds have been included in the X-ray Powder Data File ('A.S.T.M. Index').

Cinchonine sulphate $(C_{19}H_{22}ON_2)_2.H_2SO_4.2H_2O$

This compound was recrystallized from 80% ethanol as transparent, slightly elongated parallelepipeds. The Laue symmetry is $\bar{1}$, so that the space group is either $P1$ or $P\bar{1}$. The compound is optically active (Henry, 1949, p. 427) so that the space group is uniquely determined as $P1$.

Cinchonidine, $C_{19}H_{22}ON_2$

Cinchonidine was recrystallized from ethanol as colourless needles elongated along [001] and bounded by the forms {010}, {110}, and {101}. The Laue symmetry is *mmm* and the space group is determined uniquely as $P2_12_12_1$ by the systematic absences.

Cinchonidine sulphate, $C_{19}H_{22}ON_2.H_2SO_4.8H_2O$

This compound was prepared by dissolving the neutral sulphate in excess dilute sulphuric acid. The crystals obtained were recrystallized from water as long transparent laths. The Laue symmetry is $\bar{1}$, so that the space group is either $P1$ or $P\bar{1}$. The salt is optically active (Henry, 1949, p. 428) so that the space group is determined uniquely as $P1$.

Quinine dihydrobromide, $C_{20}H_{24}O_2N_2.2HBr.3H_2O$

This compound was recrystallized from water as transparent, brown-tinted, tabular crystals elongated along [001] and bounded by the forms {001}, {110}, and {010}, the last being prominent. The Laue symmetry is *mmm* and the space group is determined uniquely as $P2_12_12$ by the systematic absences.

Quinine benzenate, $C_{20}H_{24}O_2N_2.C_6H_6$

This compound was recrystallized from warm benzene as transparent flattened parallelepipeds. The crystals are efflorescent, becoming opaque within a few minutes of removal from the mother liquor. The Laue symmetry is $2/m$ and reflexions $(0k0)$ are systematically absent when k is odd, so that the space group is either $P2_1$ or $P2_1/m$. Since quinine is optically active (Henry, 1949, p. 421) the space group is uniquely determined as $P2_1$. Because of the strongly efflorescent nature of the crystals the observed density is low.

Quinamine hydrochloride, $C_{19}H_{24}O_2N_2.HCl.H_2O$

The crystals used were long transparent needles elongated along [001]. The Laue symmetry is *mmm* and the space group is uniquely determined as $P2_12_12_1$ by the systematic absences.

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