

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,* *Viriamu Jones Laboratory, University College, Cardiff, Wales*

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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 (C₁₉H₂₂ON₂)₂·H₂SO₄·2H₂O

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, C₁₉H₂₂ON₂

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, C₁₉H₂₂ON₂·H₂SO₄·8H₂O

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, C₂₀H₂₄O₂N₂·2HBr·3H₂O

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 $P2_12_12_1$ by the systematic absences.

Quinine benzenate, C₂₀H₂₄O₂N₂·C₆H₆

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 (0 k 0) 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, C₁₉H₂₄O₂N₂·HCl·H₂O

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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Table 1. *Crystallographic data*

Compound	Space group	<i>a</i> (Å)	<i>b</i> (Å)	<i>c</i> (Å)	α	β	γ	Density (g.cm. ⁻³)		<i>Z</i>
								Obs.	Calc.	
Cinchonine sulphate	<i>P</i> ₁	9.4	14.1	7.0	95.5°	90°	97°	1.31	1.31	1
Cinchonidine	<i>P</i> ₂ ₁ <i>2</i> ₁ <i>2</i> ₁	11.0	20.9	7.1	—	—	—	1.22	1.20	4
Cinchonidine sulphate	<i>P</i> ₁	9.5	10.3	6.5	93.5	104	100	1.47	1.48	1
Quinine dihydrobromide	<i>P</i> ₂ ₁ <i>2</i> ₁ <i>2</i>	14.2	23.6	7.0	—	—	—	1.53	1.52	4
Quinine benzenate	<i>P</i> ₂ ₁	9.7	25.3	9.7	—	109	—	1.16*	1.19	4
Quinamine hydrochloride	<i>P</i> ₂ ₁ <i>2</i> ₁ <i>2</i> ₁	13.2	16.6	8.8	—	—	—	1.27	1.26	4
Dihydroquinamine	<i>P</i> ₂ ₁ <i>2</i> ₁ <i>2</i> ₁	10.8	25.5	6.2	—	—	—	1.21	1.21	4
Dihydrocinchonamine hydrochloride	<i>P</i> ₂ ₁	9.5	9.9	9.8	—	98	—	—	1.21	2

* See text.

Dihydroquinamine, C₁₉H₂₆O₂N₂

The crystals used were needles elongated along [001]. The Laue symmetry is *mmm* and the space group is uniquely determined as *P*₂₁*2*₁*2*₁ by the systematic absences.

Dihydrocinchonamine hydrochloride, C₁₉H₂₆ON₂·HCl

The crystals were ill-formed plates. The Laue symmetry is *2/m* and reflexions (*0k0*) are systematically absent when *k* is odd, so that the space group is either *P*₂₁ or *P*₂₁/*m*. Since cinchonamine itself is optically active (Henry, 1949, p. 465) the space group is uniquely deter-

mined as *P*₂₁. The crystals were of such poor quality that the density could not be measured accurately. The density was calculated for the formula unit quoted above.

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Reference

HENRY, T. A. (1949). *The Plant Alkaloids*, 4th ed. London: Churchill.

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Coefficients of thermal expansion for face-centered cubic and body-centered cubic calcium.*

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Pearson (1958) has made a critical survey of the available information on the allotropic behavior of calcium metal and has concluded that above room temperature calcium exists in only two allotropic modifications, f.c.c. and b.c.c. The present work was undertaken in order to determine the coefficients of thermal expansion of these two allotropes. The determination was accomplished by comparing measured values of the *d*-spacings from individual X-ray reflections at different temperatures. The diffraction data were taken on a Geiger-counter diffractometer modified in the manner of Chiotti (1954) for high temperature work. The apparatus allows the use of bar specimens which can be maintained under an inert atmosphere of purified helium. Copper radiation was employed. Values of 2θ were measured with a precision of ± 0.025 degrees. Values of the *d*-spacings computed from the 2θ measurements were fit to the equation

$$d_{hkl} = d_{0,hkl} (1 + \alpha T)$$

by the method of least squares.

Values for the linear coefficient of thermal expansion of the f.c.c. phase are shown in Table 1. Each value is

based upon five to nine experimental points. The greater thermal attenuation of intensity for higher angle reflections accounts for the trend toward narrowing temperature range and reduced precision with increasing Bragg angle. The weak intensity due to the low multiplicity of the 600 reflection made the effect of thermal attenuation particularly severe for that reflection.

Table 1. *Measured coefficients of thermal expansion for f.c.c. calcium*

<i>hkl</i>	$\alpha \times 10^6$ per °C.	Temp. range (°C.)
511	22.4 ± 0.4	26–371
531	22.5 ± 0.4	26–371
600	21.9 ± 1.2	26–164
620	21.9 ± 0.6	26–253
533	22.8 ± 0.8	26–253
Average	22.3 ± 0.2	

In the case of the b.c.c. only six reflections were observed. Of these, only the (211) reflection persisted at elevated temperatures with sufficient intensity and with a significantly large shift in position to be usable to calculate a meaningful coefficient of thermal expansion. The value obtained from the (211) reflection in the range 467–603 °C. was $(33.6 \pm 1.6) \times 10^{-6}$ per °C. A very rough

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