

Table 4. Comparison of endocyclic torsion angles ($^{\circ}$, e.s.d.'s in parentheses) of bicyclo[3.3.1]nonadiene systems

A, B: the present structure; *C*: Radcliffe *et al.* (1984); *D*: Quast *et al.* (1984) (mean values of four closely related structures); *E*: theoretical values for the ideal half-chair.

	<i>A</i>		<i>B</i>	<i>C</i>	<i>D</i>	<i>E</i>
C(4A)–C(12A)	–0.7 (4)	C(6A)–C(10A)	3.6 (4)	0.2 (3)	–1 (1)	0
C(12A)–C(12)	–17.6 (3)	C(10A)–C(11)	–21.3 (4)	–16.1 (4)	–13 (3)	15
C(12)–C(13)	57.4 (4)	C(11)–C(12)	51.5 (3)	50.5 (3)	47 (3)	46
C(13)–C(6)	–76.0 (3)	C(12)–C(13)	–65.5 (4)	–68.7 (3)	–68 (2)	63
C(6)–S	50.8 (3)	C(13)–C(6)	48.1 (3)	51.0 (3)	52 (1)	46
S–C(4A)	–15.0 (3)	C(6)–C(6A)	–17.3 (4)	–16.7 (4)	–18 (2)	–15

accordance with recent NMR data (Šindelář *et al.*, 1987), the molecular skeleton is tetracyclic with two phenyl rings *ortho*-condensed to a central bicyclo[3.3.1]-2-thianonane-3,7-diene system. The bond lengths and angles are all normal, indicating an essentially strain-free conformation. Idealized geometry calculations show that there are two such conformations available which differ in the puckering of the central bicyclononadiene system and, consequently, in the dihedral angle of the aromatic ring planes: (a) twin-sofa central rings with dihedral angle *ca* 140 $^{\circ}$, and (b) twin-half-chair central rings with dihedral angle *ca* 90 $^{\circ}$. In the crystal, the (b) conformation is realized: the dihedral angle has the value of 91.2 (3) $^{\circ}$ and the idealized twofold half-chair axes bisect the C(4A)–C(12A) and C(6A)–C(10A) bonds respectively. This arrangement is closely related (Table 4) to the bicyclo[3.3.1]nonadiene systems whose crystal structures are known (Radcliffe, Gutierrez, Blount & Mislow, 1984; Quast, Gorlach, Stawitz, Peters, Peters & von Schmering, 1984). To our knowledge, no example of such a bicyclic system involving sulfur as a heteroatom has been characterized by crystal structure

determination till now. The asymmetry parameters (Duax, Weeks & Rohrer, 1976) $\Delta C_2(4A-12A) = 5.0^{\circ}$, $\Delta C_2(6A-10A) = 3.5^{\circ}$ indicate, however, that the S atom affects the relative conformation of the rings very little.

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Acta Cryst. (1987). **C43**, 1951–1954

The Structure of the 1:1 Complex of β -1',2',3',4',10,11-Hexahydrocinchonine and β -9-Deoxy-1',2',3',4',10,11-hexahydrocinchonine

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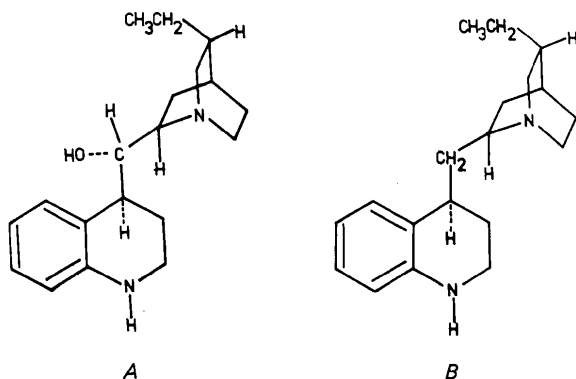
(Received 10 November 1986; accepted 13 April 1987)

Abstract. $C_{19}H_{28}N_2O \cdot C_{19}H_{28}N_2$, $M_r = 584.9$, orthorhombic, $P2_12_12_1$, $a = 12.463$ (2), $b = 13.227$ (3), $c = 20.330$ (4) Å, $V = 3351$ (1) Å 3 , $Z = 4$, $D_m = 1.13$, $D_x = 1.16$ Mg m $^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu(\text{Cu } K\alpha) = 0.46$ mm $^{-1}$, $F(000) = 1280$, room temperature, $R = 0.047$ for 2297 observed reflexions. The complex-

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forming molecules are joined together by a hydrogen bond between the hydroxyl O atom of hexahydrocinchonine and the quinuclidine N atom of 9-deoxyhexahydrocinchonine [O...N = 2.745 (5) Å]. An additional intermolecular hydrogen bond between the tetrahydroquinoline N atom and the quinuclidine N atom of hexahydrocinchonine joins the complex molecules into chains parallel to **b**.

Introduction. During investigations of a mixture of products formed by chemical reduction of dihydrocinchonine with sodium and ethanol a crystalline substance with m.p. 395–396 K has been isolated. It has been identified as a 1:1 complex of β -deoxy-1',2',3',4',10,11-hexahydrocinchonine, *A*, (crystalline compound, m.p. 379.2–379.6 K) and β -1',2',3',4',10,11-hexahydrocinchonine, *B*, (oil) (Suszko & Golankiewicz, 1968). All attempts to separate the constituents of the complex by recrystallization from various organic solvents have failed. An X-ray-diffraction analysis has been undertaken to search for the reasons for its stability.



Experimental. Crystals obtained from ethanol, D_m by flotation, space group from Weissenberg photographs, crystal 0.4 × 0.4 × 0.3 mm, Syntex $P2_1$ diffractometer, cell parameters from least-squares treatment of setting angles of 15 reflexions with $14 \leq 2\theta \leq 20^\circ$. 2523 reflexions measured in range $h: 0 \rightarrow 13$, $k: 0 \rightarrow 14$, $l: 0 \rightarrow 22$. No significant intensity variation for two standard reflexions measured every 1.5 h. Maximum value of $(\sin\theta)/\lambda = 0.587 \text{ \AA}^{-1}$. Peak profile analysis according to Lehmann & Larsen (1974), 2297 reflexions with $I \geq 2\sigma(I)$. Structure solved by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement on F_o , $w^{-1} = \sigma^2(F_o)$, H atoms from $\Delta\rho$ map, included as fixed isotropic contribution to F_o , anisotropic thermal parameters for non-H atoms, empirical isotropic extinction parameter x refined to $1.41(6) \times 10^{-6}$, $R = 0.047$, $wR = 0.052$ for 389 parameters and 2297 reflexions, $S = 4.48$ (Δ/σ)_{max} = 0.1, ($\Delta\rho$)_{max} =

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (\AA^2)

$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
N(1PA)	-0.3832 (3)	0.1360 (3)	-0.4968 (2)	0.078 (2)
C(2PA)	-0.4537 (4)	0.0493 (4)	-0.4998 (2)	0.078 (2)
C(3PA)	-0.3971 (4)	-0.0420 (4)	-0.4720 (2)	0.073 (2)
C(4PA)	-0.3621 (3)	-0.0240 (3)	-0.3998 (2)	0.056 (1)
C(5PA)	-0.2355 (3)	0.0963 (3)	-0.3427 (2)	0.056 (1)
C(6PA)	-0.1779 (3)	0.1860 (4)	-0.3375 (2)	0.065 (2)
C(7PA)	-0.1914 (4)	0.2585 (3)	-0.3862 (3)	0.070 (2)
C(8PA)	-0.2601 (4)	0.2419 (3)	-0.4382 (2)	0.066 (2)
C(9PA)	-0.3175 (3)	0.1510 (3)	-0.4430 (2)	0.058 (2)
C(10PA)	-0.3052 (3)	0.0772 (3)	-0.3948 (2)	0.051 (1)
N(2A)	-0.6000 (2)	-0.1409 (3)	-0.3043 (2)	0.058 (1)
C(2A)	-0.5798 (4)	-0.1247 (4)	-0.2331 (2)	0.072 (2)
C(3A)	-0.5154 (3)	-0.2115 (4)	-0.2016 (2)	0.066 (2)
C(4A)	-0.4715 (3)	-0.2759 (3)	-0.2583 (2)	0.069 (2)
C(5A)	-0.5662 (4)	-0.3258 (3)	-0.2951 (2)	0.077 (2)
C(6A)	-0.6467 (3)	-0.2422 (3)	-0.3128 (2)	0.073 (2)
C(7A)	-0.4143 (3)	-0.2087 (3)	-0.3089 (2)	0.063 (2)
C(8A)	-0.4974 (3)	-0.1387 (3)	-0.3421 (2)	0.052 (1)
C(9A)	-0.4578 (3)	-0.0300 (3)	-0.3504 (2)	0.056 (1)
C(10A)	-0.4319 (4)	-0.1679 (5)	-0.1537 (3)	0.096 (2)
C(11A)	-0.3902 (5)	-0.2376 (5)	-0.1049 (3)	0.105 (3)
N(1PB)	0.0208 (3)	0.2132 (2)	-0.1889 (2)	0.062 (1)
C(2PB)	0.1318 (3)	0.2253 (3)	-0.2095 (2)	0.059 (1)
C(3PB)	0.1496 (3)	0.1609 (3)	-0.2696 (2)	0.057 (1)
C(4PB)	0.1337 (3)	0.0493 (3)	-0.2529 (2)	0.050 (1)
C(5PB)	-0.0199 (3)	-0.0602 (3)	-0.2088 (2)	0.057 (2)
C(6PB)	-0.1170 (4)	-0.0729 (3)	-0.1762 (2)	0.065 (2)
C(7PB)	-0.1675 (3)	0.0097 (3)	-0.1487 (2)	0.060 (2)
C(8PB)	-0.1208 (3)	0.1031 (3)	-0.1533 (2)	0.054 (1)
C(9PB)	-0.0224 (3)	0.1168 (3)	-0.1856 (2)	0.048 (1)
C(10PB)	0.0287 (3)	0.0342 (3)	-0.2156 (2)	0.045 (1)
O(1B)	0.2524 (2)	0.0018 (2)	-0.3417 (1)	0.063 (1)
N(2B)	0.0358 (3)	-0.1065 (2)	-0.3969 (2)	0.056 (1)
C(2B)	0.1217 (3)	-0.1355 (3)	-0.4431 (2)	0.063 (2)
C(3B)	0.1277 (3)	-0.0649 (3)	-0.5031 (2)	0.061 (2)
C(4B)	0.0568 (3)	0.0265 (3)	-0.4886 (2)	0.060 (2)
C(5B)	-0.0602 (3)	-0.0106 (4)	-0.4856 (2)	0.072 (2)
C(6B)	-0.0651 (3)	-0.0973 (4)	-0.4345 (2)	0.070 (2)
C(7B)	0.0850 (3)	0.0724 (3)	-0.4208 (2)	0.058 (1)
C(8B)	0.0603 (3)	-0.0067 (3)	-0.3666 (2)	0.048 (1)
C(9B)	0.1489 (3)	-0.0185 (3)	-0.3137 (2)	0.052 (1)
C(10B)	0.2430 (4)	-0.0378 (4)	-0.5209 (2)	0.077 (2)
C(11B)	0.3109 (5)	-0.1282 (5)	-0.5350 (3)	0.108 (3)

0.25, ($\Delta\rho$)_{min} = -0.14 e \AA^{-3} . Computer programs: *MULTAN80* (Main *et al.*, 1980), *SHELX76* (Sheldrick, 1976) and local programs (Jaskólski, 1982a). Molecular illustrations drawn using *ORTEP* (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates are given in Table 1, bond lengths and angles in Table 2.*

The 9-deoxy-1',2',3',4',10,11-hexahydrocinchonine and 1',2',3',4',10,11-hexahydrocinchonine molecules are labelled *A* and *B*, respectively. *ORTEP* drawings showing the conformations of these molecules and their atom-labelling system are presented in Fig. 1. Bond lengths and angles of quinuclidine and tetrahydroquinoline moieties are similar in both molecules. The

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43964 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and angles (°)

	Molecule A	Molecule B
N(1P)-C(2P)	1.446 (7)	1.455 (5)
N(1P)-C(9P)	1.381 (5)	1.386 (5)
C(2P)-C(3P)	1.508 (7)	1.507 (6)
C(3P)-C(4P)	1.551 (6)	1.527 (5)
C(4P)-C(10P)	1.518 (6)	1.526 (5)
C(10P)-C(5P)	1.394 (6)	1.396 (5)
C(5P)-C(6P)	1.389 (6)	1.389 (6)
C(6P)-C(7P)	1.389 (7)	1.380 (6)
C(7P)-C(8P)	1.378 (7)	1.369 (6)
C(8P)-C(9P)	1.402 (6)	1.402 (5)
C(9P)-C(10P)	1.392 (6)	1.403 (5)
C(4P)-C(9)	1.561 (6)	1.538 (5)
C(9)-O(1)	-	1.435 (5)
C(9)-C(8)	1.530 (6)	1.549 (5)
C(8)-N(2)	1.492 (5)	1.487 (5)
N(2)-C(6)	1.471 (6)	1.477 (5)
N(2)-C(2)	1.485 (6)	1.477 (5)
C(2)-C(3)	1.540 (7)	1.538 (6)
C(3)-C(4)	1.535 (7)	1.526 (6)
C(4)-C(5)	1.547 (7)	1.540 (6)
C(4)-C(7)	1.535 (6)	1.546 (6)
C(5)-C(6)	1.535 (7)	1.549 (7)
C(7)-C(8)	1.545 (6)	1.551 (5)
C(3)-C(10)	1.535 (7)	1.525 (6)
C(10)-C(11)	1.452 (8)	1.492 (8)
N(1P)-C(2P)-C(3P)	109.6 (4)	108.1 (3)
C(2P)-C(3P)-C(4P)	111.3 (3)	110.4 (3)
C(3P)-C(4P)-C(10P)	109.3 (3)	110.4 (3)
C(4P)-C(10P)-C(9P)	121.4 (3)	120.3 (3)
C(10P)-C(9P)-N(1P)	121.5 (3)	121.2 (3)
C(2P)-N(1P)-C(9P)	120.6 (3)	119.0 (3)
C(9P)-C(10P)-C(5P)	118.4 (3)	117.2 (3)
C(10P)-C(5P)-C(6P)	122.3 (3)	122.2 (3)
C(5P)-C(6P)-C(7P)	118.2 (4)	119.7 (3)
C(6P)-C(7P)-C(8P)	120.8 (4)	119.5 (4)
C(7P)-C(8P)-C(9P)	120.4 (4)	121.3 (3)
C(8P)-C(9P)-C(10P)	119.8 (4)	120.0 (3)
C(8P)-C(9P)-N(1P)	118.7 (4)	118.8 (3)
C(5P)-C(10P)-C(4P)	120.2 (3)	122.6 (3)
C(3P)-C(4P)-C(9)	112.7 (3)	111.7 (3)
C(10P)-C(4P)-C(9)	111.0 (3)	115.4 (3)
C(4P)-C(9)-O(1)	-	108.7 (3)
C(4P)-C(9)-C(8)	111.5 (3)	114.3 (3)
O(1)-C(9)-C(8)	-	110.3 (3)
C(9)-C(8)-N(2)	110.7 (3)	110.2 (3)
C(9)-C(8)-C(7)	113.4 (3)	114.8 (3)
C(8)-N(2)-C(2)	110.7 (3)	110.2 (3)
C(8)-N(2)-C(6)	107.3 (3)	108.5 (3)
C(6)-N(2)-C(2)	108.2 (3)	108.0 (3)
N(2)-C(8)-C(7)	109.7 (3)	110.2 (3)
C(8)-C(7)-C(4)	109.2 (3)	108.9 (3)
N(2)-C(2)-C(3)	112.7 (3)	112.5 (3)
N(2)-C(6)-C(5)	111.7 (3)	112.0 (3)
C(3)-C(4)-C(7)	110.3 (3)	110.5 (3)
C(5)-C(4)-C(7)	106.1 (3)	107.8 (3)
C(3)-C(4)-C(5)	109.2 (3)	107.7 (3)
C(4)-C(3)-C(10)	116.3 (4)	114.0 (3)
C(2)-C(3)-C(10)	109.7 (4)	112.1 (3)
C(3)-C(10)-C(11)	116.0 (4)	113.1 (4)

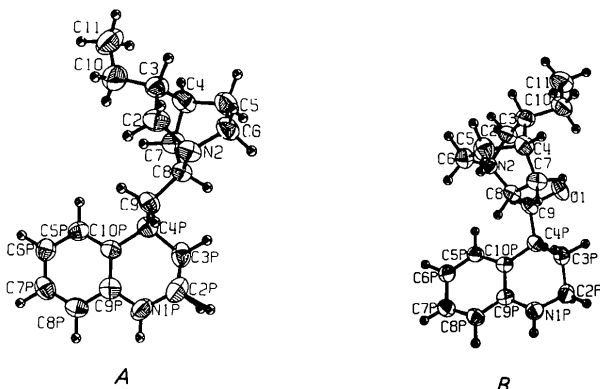


Fig. 1. ORTEP drawings (Johnson, 1976) of molecules A and B.

differences appear only in the vicinity of C(9) which is differently substituted in molecules A and B. Hydrogenation of the pyridine fragment of Cinchona alkaloids gives rise to a new chiral center at C(4') and alters considerably the overall conformation of the molecule, especially, in its quinoline fragment. The configuration at C(4') of partially hydrogenated Cinchona alkaloids has been previously suggested on the basis of consideration of specific rotation (Golankiewicz, Gdaniec, Jaskólski & Kosturkiewicz, 1981). The present results [(4'R) for molecule A, (4'S) for molecule B] corroborate previous assignments under the assumption that the configuration at the chiral center C(8) is R. The 1',2',3',4'-tetrahydropyridine fragment adopts different conformations in molecules A and B. Asymmetry parameters (Duax & Norton, 1975) describe this six-membered ring in molecule A as a distorted half-chair [$\Delta C_2^{2P,3P} = 9.5$ (9), $\Delta C_S^{3P} = 14$ (1), mean torsion angle $|\varphi| = 29$ (1)°] with C(2P) being at the same side of the ring plane as C(9). In molecule B this ring adopts a half-chair conformation [$\Delta C_2^{2P,3P} = 2.6$ (7), $\Delta C_S^{3P} = 20.4$ (9), $|\varphi| = 31.9$ (9)°] with C(9) being *exo* to C(2P). As a consequence of the differences of tetrahydropyridine ring conformation the substituent at C(4P) is placed pseudoaxially in molecule A and pseudoequatorially in molecule B. An analysis of intermolecular distances in the crystal indicates that the molecules are joined together by two types of hydrogen bonds. Molecules A and B are bonded by a rather short hydrogen bond between the hydroxyl O atom of B and the quinuclidine N atom of A [$N(2A) \cdots O(1B) = 2.745$ (5), $N(2A) \cdots H(23B) = 1.78$ Å, $N(2A) \cdots H(23B) - O(1B) = 168^\circ$, (i): $x-1, y, z$]. Such a hydrogen-bond complex of molecules A and B is presented in Fig. 2. An additional intermolecular hydrogen bond between B molecules related by a 2₁ axis parallel to *y* [$N(2B) \cdots N(1PB) = 3.038$ (5), $N(2B) \cdots H(1B) = 2.19$ Å, $N(2B) \cdots H(1B) - N(1PB) = 161^\circ$, (i): $-x, y-0.5, -0.5-z$] joins the complex molecules into infinite chains. According to their ΔH_A parameters (Jaskólski, 1982b) these two hydrogen bonds can be considered as medium strong ($\Delta H_A = 29$) and medium weak ($\Delta H_A = 12$), respectively,

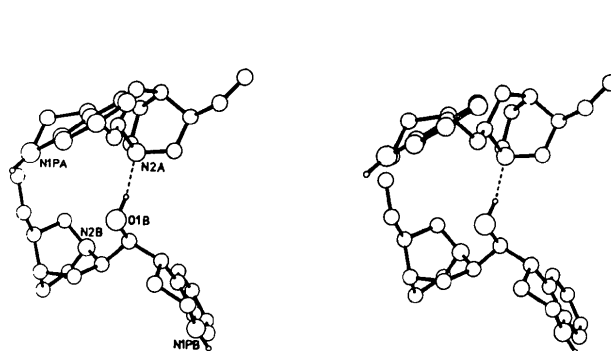


Fig. 2. Stereodrawing of the 1:1 hydrogen-bond complex.

with the first one being responsible for the stability of the complex.

The work was supported by Project RP.II.13.2.13.(MNIŚW).

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Acta Cryst. (1987). **C43**, 1954–1956

Structure of 2,3:4,5-Di-*O*-isopropylidene- β -D-fructopyranose

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(Received 5 March 1987; accepted 8 May 1987)

Abstract. C₁₂H₂₀O₆, $M_r = 260.3$, tetragonal, $P4$, $a = 21.51$ (1), $c = 5.940$ (3) Å, $V = 2748$ Å³, $Z = 8$, $D_m = 1.23$, $D_x = 1.26$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.11$ mm⁻¹, $F(000) = 1120$, $T = 300$ (2) K, final $R = 0.044$ for 2932 observed reflexions. The six-membered β -fructopyranose ring in the two crystallographically independent molecules (*A* and *B*) is in an ²S₀ twist-boat conformation. The hydroxyl groups of four *A* molecules and, independently, four *B* molecules form square donor–acceptor hydrogen-bond systems about the fourfold axes at $00z$ and $\frac{1}{2}z$, respectively. There are no hydrogen bonds between *A* and *B* molecules.

Introduction. It is known that carbohydrates and acetone form isopropylidene acetals in the presence of acids. From the condensation of D-fructose with acetone it is easy to obtain two crystalline diisopropylidene derivatives: 1,2:4,5-di-*O*-isopropylidene- β -D-fructopyranose and 2,3:4,5-di-*O*-isopropylidene- β -D-fructopyranose (Ohle & Koller, 1924). The structure of the first of these has been published (Takagi, Shiono & Rosenstein, 1973). Here we report the crystal structure of the second compound, which occurs in the unusual space group $P4$, in order to compare the molecular structures of both isomers.

Experimental. The title compound was prepared according to the procedure of Ohle & Koller (1924). Colorless, long needle crystals were obtained by slow evaporation at room temperature of the 2-propanol solution. D_m by flotation in an aqueous solution of Ca(NO₃)₂. Preliminary examination by rotation and Weissenberg photographs. A specimen $0.6 \times 0.8 \times 0.08$ mm was cut from a large crystal. Syntex P2₁ diffractometer, Mo $K\alpha$ radiation for lattice parameters (15 reflexions in range $20 < 2\theta < 26^\circ$) and intensity measurements; ω - 2θ scan technique; 4393 reflexions measured in the range $2 \leq \theta \leq 30^\circ$; h 0–30, k 1–30, l 0–8; two standards measured after every 50 reflexions, variation 5%. Absorption ignored. Structure solved by direct methods with *MULTAN80* (Main *et al.*, 1980). 31 atoms located in the *E* map with highest combined figure of merit. The remaining heavy atoms, and finally all the H atoms, were located from subsequent difference Fourier maps. Full-matrix least-squares refinement using *XTL/XTLE* programs (Syntex, 1976) based on *F* values (O, C anisotropic, H isotropic) for 2932 reflexions with $I \geq 2\sigma(I)$, $w = 1/\sigma^2(F_o)$ gave $R = 0.044$ and $wR = 0.036$; max. $\Delta/\sigma = 0.4$. Neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974), real and imaginary components of anomalous dis-