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Structures of Tetrakis(4-benzoylpyridine)bis(isothiocyanato)cobalt(II) (1) and Bis(4-acetylpyridine)diaquabis(isothiocyanato)cobalt(II) (2)

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Abstract. (1) [Co(NCS)$_2$(C$_{12}$H$_9$NO)$_4$]: M_r = 907·9, monoclinic, $P2_1/c$, a = 9·056 (7), b = 20·887 (14), c = 11·265 (10) Å, β = 90·2 (1)°, V = 2130·8 Å^3, Z = 2, D_x = 1·41, D_m = 1·42 g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 5·71 cm^{-1}, F(000) = 938, room temperature, final R = 0·061 for 1605 independent observed reflections. The structure contains discrete centrosymmetric molecules with the octahedral Co atom bonded to two thiocyanates [Co—N 2·058 (7) Å], and four 4-benzoylpyridine ligands [Co—N 2·215 (6), 2·217 (6) Å]. In the ligands, the phenyl rings are twisted out of the planes of the pyridine rings by angles of 54·5 (1), 54·3 (1)°. (2) [Co(NCS)$_2$(H$_2$O)$_2$(C$_7$H$_7$NO)$_2$]: M_r = 453·2, monoclinic, $P2_1/a$, a = 10·313 (8), b = 16·316 (7), c = 6·345 (8) Å, β = 106·5 (1)°, V = 1017·0 Å^3, Z = 2, D_x = 1·48, D_m = 1·46 g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 11·0 cm^{-1}, F(000) = 466, room temperature, final R = 0·058 for 1598 independent observed reflections. The molecule has imposed $\bar{1}$ symmetry. The Co atom is six-coordinate octahedral, bonded to two thiocyanates [Co—N 2·080 (5) Å], two 4-acetylpyridine ligands [Co—N 2·195 (4) Å] and two water molecules [Co—O 2·130 (4) Å]. In the 4-acetylpyridine ligands the acetyl group is twisted from the pyridine ring plane by 14·1 (1)°. There is extensive intermolecular hydrogen bonding between the water groups and the thiocyanates and acetyl groups in adjacent molecules.

Introduction. In our studies of complexes of cobalt(II) with pyridine derivatives we reported (Cabral & Cabral, 1977) the preparation and characterization of a number of halo and pseudohalocomplexes of cobalt(II) with 3- and 4-benzoylpyridine. Of particular interest was the existence of two forms of [Co(NCS)$_2$(4-benzoylpyridine)$_4$] which were coloured buff and orange respectively. We found that the formation of the buff or the orange forms depends on the order of mixing hot ethanolic solutions of cobalt(II) thiocyanate and 4-benzoylpyridine. However, recrystallization of both forms yielded the orange form (as crystals). In order to investigate this phenomenon, we report here the structure (1) of the orange crystals of this sample. We also report the structure of the related compound bis(4-acetylpyridine)diaquabis(isothiocyanato)cobalt(II) (2) which we have prepared.

Experimental. Crystals of (1) were prepared as described previously (Cabral & Cabral, 1977). Crystals of (2) were prepared from the reaction of Co(NCS)$_2$ with 4-acetylpyridine. The resulting precipitate was redissolved in ethanol and kept for two days at 255 K until suitable crystals appeared (Cabral & Cabral, 1985, unpublished results). Densities measured by flotation in CCl$_4$/ligroin. Precession photographs established preliminary cell constants. Suitable crystals of approximate size 0·4 × 0·2 × 0·4 mm (1) and 0·2 × 0·3 × 0·3 mm (2) mounted on a Stoe STADI2 diffractometer to rotate about the a axes. Cell dimensions obtained by measurement of *ca* 20 high-angle axial reflections. Intensity data collected *via* variable-width ω scan, background counts 20 s, step-scan rate of 0·033° s^{-1} applied to a width of (1·5 + sinμ/tanθ)°. Absorption and extinction corrections not applied. Standard reflections h22 measured every 20 measurements for each layer: no significant change in intensity. 3688 ($0 \le h \le 10$, $0 \le k \le 24$, $-13 < l < 13$) data in (1) and 2947 ($0 \le h \le 11$, $-11 < k < 10$, $-12 < l < 11$) data in (2) measured with $2\theta_{\max} < 50°$. 1605, 1598 independent data with $I > 3\sigma(I)$ refined (on F) to R 0·061 (wR 0·068, S 2·35) and R 0·058 (wR 0·065, S 1·75) respectively. Weighting scheme $w = 1/[\sigma^2(F) + 0{\cdot}0003F^2]$ [$\sigma(F)$ from counting statistics] chosen to give similar values of $w\Delta^2$ over ranges of sinθ/λ and F_o.

Structures determined from the Patterson method. H atoms bonded to C included in calculated positions and their thermal parameters refined. H atoms on the water molecule in (2) located in a difference-Fourier map and all their parameters refined. H atoms on the methyl groups in (2) refined as a rigid group with a common thermal parameter. Non-H atoms refined anisotropically *via* full-matrix least squares. Scattering

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factors and dispersion corrections from *International Tables for X-ray Crystallography* (1974). Final difference-Fourier maps showed no important features [max. 1·23, min. −0·54 e Å^{-3} in (1), max. 1·04, min. −0·73 e Å^{-3} in (2)]. In final cycles of refinement no shift $>0{\cdot}1\sigma$. Calculations carried out using *SHELX*76 (Sheldrick, 1976) and our own programs on the Amdahl V7A at the University of Reading.

Discussion. Final coordinates are given in Tables 1 and 2 and molecular dimensions in Tables 3 and 4.* The molecular geometries are shown in Figs. 1 and 2 together with the atomic-numbering schemes. Both molecules have a crystallographically imposed centre of symmetry. In both (1) and (2) the Co atom is six-coordinate octahedral. In (1) the metal atom is bonded to two thiocyanate ligands [Co—N 2·058 (7) Å] and four 4-benzoylpyridine ligands [Co—N 2·215 (6) and 2·217 (6) Å]. In (2) the metal atom is bonded to two thiocyanates [Co—N(1) 2·080 (5) Å], two 4-acetylpyridine ligands [Co—N 2·195 (5) Å], and to two water molecules [Co—O 2·130 (4) Å].

Structures of a large number of octahedral cobalt complexes have been determined. Several structures have features in common with (1) and (2). For example in centrosymmetric *trans*-bis(isothiocyanato)tetrakis(pyridine)cobalt(II) (Hartl & Brudgam, 1980), bond lengths are exactly comparable to those in (1), *i.e.* Co—N(CS) is 2·084 Å and Co—N(py) is 2·191, 2·214 Å. Similar values are also found in bis(isothiocyanato)tetrakis(4-vinylpyridine)cobalt(II); for the orthorhombic form (Foxman & Mazurek, 1982), Co—N(CS) is 2·100, 2·049 Å and Co—N(py) 2·197, 2·169, 2·186, 2·147 Å and in the tetragonal form, where the molecule is centrosymmetric (Andreetti & Sgarabotto, 1972), unique values are 2·086 Å for Co—N(CS) and 2·170, 2·206 Å for Co—N(py). In *cis*-bis(2,2′-bipyridyl)bis(isothiocyanato)cobalt(II) (Veidis, Dockum, Charron, Reiff & Brennan, 1981), dimensions are Co—N(CS) 2·063, 2·075 Å and Co—N(bpy) 2·155, 2·191, 2·157, 2·149 Å. In diaquabis(isothiocyanato)bis(nicotinamide)cobalt (Tsintsadze, Dzhavakhishvili, Amiraslanov & Kvitashvili, 1979) bond lengths are Co—O 2·120, 2·085 Å, Co—N(CS) 2·111, 2·093 Å, Co—N 2·204, 2·164 Å.

All these values suggest that the Co—N bond lengths in (1) and (2) are unexceptional.

In both structures the pyridine rings are planar within experimental error. In (1) the Co atom is 0·137 (4), 0·145 (4) Å from the two pyridine ring planes, in (2) it is 0·290 (3) Å from this plane. In (1) the two benzoyl groups are twisted by 54·6 (1), 54·3 (1)° from the plane of the pyridine rings while in (2) the acetyl group is twisted by only 14·1 (1)° from the plane of the pyridine ring.

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 ×10^3) *for* (1) *with e.s.d.'s in parentheses*

	x	*y*	*z*	$\bar{U}_{eq}$*
Co	0	0	0	47 (2)
S(1)	−4849 (3)	−478 (1)	1510 (3)	88 (3)
N(1)	−2091 (8)	−130 (3)	668 (6)	49 (7)
C(1)	−3234 (10)	−259 (4)	1019 (7)	49 (8)
N(11)	605 (7)	−951 (3)	724 (5)	46 (7)
C(12)	−188 (9)	−1259 (4)	1539 (7)	61 (9)
C(13)	149 (9)	−1844 (4)	1952 (7)	62 (9)
C(14)	1345 (10)	−2171 (3)	1469 (8)	55 (9)
C(15)	2200 (10)	−1864 (4)	667 (8)	70 (10)
C(16)	1780 (10)	−1241 (4)	312 (8)	71 (10)
C(17)	1747 (10)	−2831 (4)	1912 (8)	61 (10)
O(18)	1497 (10)	−2957 (3)	2954 (6)	119 (10)
C(19)	2398 (9)	−3320 (3)	1118 (8)	53 (9)
C(20)	3183 (11)	−3815 (4)	1624 (9)	74 (11)
C(21)	3681 (11)	−4319 (4)	912 (11)	88 (13)
C(22)	3445 (12)	−4298 (5)	−295 (11)	96 (13)
C(23)	2653 (12)	−3788 (4)	−792 (10)	80 (12)
C(24)	2137 (11)	−3309 (4)	−89 (9)	68 (10)
N(31)	723 (7)	436 (3)	1700 (6)	56 (7)
C(32)	−65 (9)	341 (4)	2674 (7)	57 (9)
C(33)	347 (10)	575 (3)	3784 (8)	61 (9)
C(34)	1617 (9)	925 (4)	3880 (7)	53 (9)
C(35)	2443 (10)	1012 (4)	2879 (8)	52 (10)
C(36)	1966 (11)	767 (4)	1828 (8)	62 (11)
C(37)	2098 (10)	1128 (4)	5105 (8)	64 (10)
O(38)	1814 (8)	799 (3)	5930 (6)	79 (8)
C(39)	2863 (10)	1766 (4)	5267 (8)	60 (10)
C(40)	2719 (12)	2267 (4)	4492 (9)	69 (12)
C(41)	3386 (13)	2848 (4)	4727 (10)	86 (13)
C(42)	4217 (11)	2915 (5)	5721 (10)	77 (13)
C(43)	4380 (11)	2426 (5)	6494 (9)	66 (13)
C(44)	3695 (11)	1848 (5)	6271 (8)	63 (11)

* $U_{eq} = \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

Table 2. *Atomic coordinates* (×10^4, *for* H ×10^3) *and isotropic thermal parameters* (Å^2 ×10^3) *for* (2) *with e.s.d.'s in parentheses*

	x	*y*	*z*	$\bar{U}_{eq}$*/*U*
Co	0	0	0	64 (1)
N(1)	6 (4)	968 (3)	−2130 (7)	84 (5)
C(2)	−20 (5)	1459 (3)	−3390 (8)	73 (5)
S(3)	−37 (3)	2183 (1)	−5173 (3)	134 (3)
N(10)	1845 (4)	−501 (3)	−576 (6)	60 (4)
C(11)	2696 (5)	−958 (3)	961 (9)	76 (5)
C(12)	3939 (5)	−1211 (4)	819 (9)	77 (6)
C(13)	4338 (4)	−982 (3)	−972 (8)	60 (5)
C(14)	3455 (5)	−517 (3)	−2611 (8)	69 (5)
C(15)	2220 (5)	−299 (4)	−2359 (8)	67 (5)
C(16)	5704 (5)	−1200 (3)	−1201 (9)	64 (5)
O(17)	6098 (4)	−855 (3)	−2608 (7)	88 (5)
C(18)	6550 (6)	−1825 (4)	329 (12)	94 (7)
O(4)	1243 (4)	690 (3)	2665 (6)	74 (4)
H(41)	204 (8)	78 (4)	264 (10)	82 (19)†
H(42)	82 (9)	103 (6)	321 (13)	123 (32)†

* $U_{eq} = \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

† *U* values. Remaining H-atom positions have been deposited.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42310 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The twist in (1) is presumably a steric effect to reduce repulsions between the phenyl rings and the pyridine rings. In (2) the much smaller rotation is likely to be due to both steric effects and the geometric demands of the intermolecular hydrogen bond between this group and a water molecule of an adjacent molecule. Dimensions of this intermolecular hydrogen bond are O(4)···O(17)-(1 − *x*, −*y*, −*z*) 2·765 (6), O(2)···H(17) 1·93 (9) Å, O—H···O 174 (8)°. The other water H atom is also involved in an intermolecular hydrogen bond to the S atom of the thiocyanate, dimensions being O(4)···S(3)(*x*, *y*, 1 + *z*) 3·254 (5), H(42)···S 2·43 (10) Å, O—H···S 174 (8)°. Thus the molecules are connected *via* an array of strong hydrogen bonds. This structure is isomorphous with that of the iron analogue (Long, Galeazzi, Russo, Valle & Calogero, 1983).

There are no other intermolecular contacts of note in (2) and none at all in (1). After determining the structure of (1), we took X-ray powder diffraction patterns of both the orange and buff-coloured samples of this compound. We found that the X-ray powder diffraction patterns were identical which suggests that there is no difference in the basic structure of a centrosymmetric *trans* cobalt octahedral complex. We have no explanation for the difference in colour.

Table 3. *Molecular dimensions in* (1)

Distances in Å, angles in degrees.

Co–N(1)	2·058 (7)	C(22)–C(23)	1·400 (14)
Co–N(11)	2·215 (6)	C(23)–C(24)	1·359 (13)
Co–N(31)	2·217 (6)	N(31)–C(32)	1·325 (11)
S(1)–C(1)	1·630 (9)	N(31)–C(36)	1·329 (11)
N(1)–C(1)	1·141 (11)	C(32)–C(33)	1·392 (12)
N(11)–C(12)	1·332 (10)	C(33)–C(34)	1·367 (12)
N(11)–C(16)	1·311 (11)	C(34)–C(35)	1·367 (12)
C(12)–C(13)	1·343 (11)	C(34)–C(37)	1·507 (12)
C(13)–C(14)	1·393 (12)	C(35)–C(36)	1·359 (13)
C(14)–C(15)	1·353 (12)	C(37)–O(38)	1·184 (11)
C(14)–C(17)	1·510 (11)	C(37)–C(39)	1·512 (12)
C(15)–C(16)	1·414 (11)	C(39)–C(40)	1·369 (12)
C(17)–O(18)	1·224 (11)	C(39)–C(44)	1·367 (13)
C(17)–C(19)	1·482 (12)	C(40)–C(41)	1·380 (13)
C(19)–C(20)	1·377 (12)	C(41)–C(42)	1·354 (16)
C(19)–C(24)	1·380 (13)	C(42)–C(43)	1·351 (15)
C(20)–C(21)	1·399 (14)	C(43)–C(44)	1·380 (15)
C(21)–C(22)	1·377 (18)		

N(1)–Co–N(11)	88·52 (23)	C(22)–C(23)–C(24)	120·2 (10)
N(1)–Co–N(31)	90·45 (25)	C(19)–C(24)–C(23)	120·2 (8)
N(11)–Co–N(31)	88·72 (23)	Co–N(31)–C(32)	119·7 (5)
Co–N(1)–C(1)	173·8 (5)	Co–N(31)–C(36)	123·7 (5)
S(1)–C(1)–N(1)	177·4 (7)	C(32)–N(31)–C(36)	116·4 (7)
Co–N(11)–C(12)	123·6 (5)	N(31)–C(32)–C(33)	123·3 (7)
Co–N(11)–C(16)	119·0 (5)	C(32)–C(33)–C(34)	118·8 (7)
C(12)–N(11)–C(16)	117·4 (6)	C(33)–C(34)–C(35)	117·9 (7)
N(11)–C(12)–C(13)	123·8 (7)	C(33)–C(34)–C(37)	117·7 (7)
C(12)–C(13)–C(14)	119·1 (7)	C(35)–C(34)–C(37)	124·1 (7)
C(13)–C(14)–C(15)	118·4 (7)	C(34)–C(35)–C(36)	119·7 (8)
C(13)–C(14)–C(17)	120·3 (7)	N(31)–C(36)–C(35)	123·9 (8)
C(15)–C(14)–C(17)	121·0 (7)	C(34)–C(37)–O(38)	119·5 (7)
C(14)–C(15)–C(16)	118·1 (7)	C(34)–C(37)–C(39)	119·3 (7)
N(11)–C(16)–C(15)	123·0 (7)	O(38)–C(37)–C(39)	121·1 (8)
C(14)–C(17)–O(18)	117·9 (7)	C(37)–C(39)–C(40)	123·6 (8)
C(14)–C(17)–C(19)	121·7 (7)	C(37)–C(39)–C(44)	117·5 (7)
O(18)–C(17)–C(19)	120·4 (7)	C(40)–C(39)–C(44)	118·8 (8)
C(17)–C(19)–C(20)	118·3 (8)	C(39)–C(40)–C(41)	120·6 (9)
C(17)–C(19)–C(24)	121·1 (7)	C(40)–C(41)–C(42)	119·5 (9)
C(20)–C(19)–C(24)	120·5 (7)	C(41)–C(42)–C(43)	120·9 (9)
C(19)–C(20)–C(21)	119·7 (9)	C(42)–C(43)–C(44)	119·7 (9)
C(20)–C(21)–C(22)	119·5 (9)	C(39)–C(44)–C(43)	120·5 (8)
C(21)–C(22)–C(23)	119·8 (9)		

Table 4. *Molecular dimensions in* (2)

Distances in Å, angles in degrees.

Co–N(1)	2·080 (5)	C(11)–C(12)	1·373 (7)
Co–N(10)	2·195 (4)	C(12)–C(13)	1·367 (7)
Co–O(4)	2·130 (4)	C(13)–C(14)	1·395 (7)
N(1)–C(2)	1·127 (6)	C(13)–C(16)	1·500 (6)
C(2)–S(3)	1·632 (5)	C(14)–C(15)	1·376 (7)
N(10)–C(11)	1·338 (6)	C(16)–O(17)	1·218 (6)
N(10)–C(15)	1·337 (6)	C(16)–C(18)	1·504 (8)

N(1)–Co–N(10)	90·67 (15)	C(11)–C(12)–C(13)	118·63 (48)
N(1)–Co–O(4)	90·12 (17)	C(12)–C(13)–C(14)	118·80 (43)
N(10)–Co–O(4)	88·51 (15)	C(12)–C(13)–C(16)	121·95 (46)
Co–N(1)–C(2)	175·65 (48)	C(14)–C(13)–C(16)	119·24 (45)
N(1)–C(2)–S(3)	178·7 (5)	C(13)–C(14)–C(15)	118·94 (46)
Co–N(10)–C(11)	120·43 (33)	N(10)–C(15)–C(14)	122·31 (47)
Co–N(10)–C(15)	121·46 (33)	C(13)–C(16)–O(17)	118·66 (49)
C(11)–N(10)–C(15)	117·81 (42)	C(13)–C(16)–C(18)	119·10 (48)
N(10)–C(11)–C(12)	123·45 (48)	O(17)–C(16)–C(18)	122·23 (48)

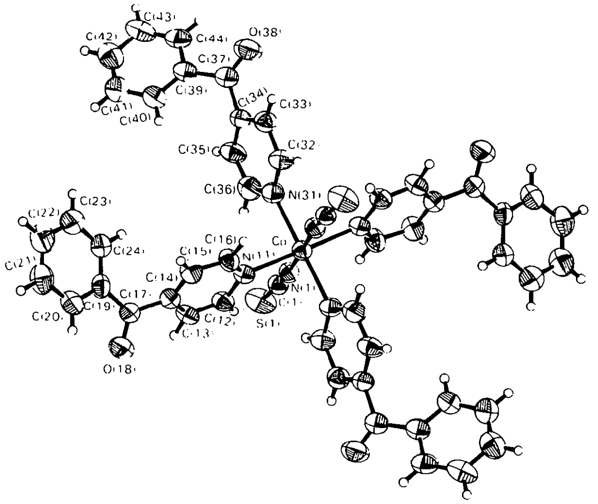

Fig. 1. The structure of (1) – thermal ellipsoids are shown at 50% probability.

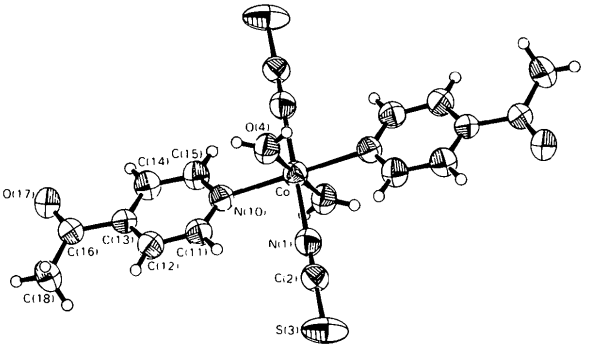

Fig. 2. The structure of (2) – thermal ellipsoids are shown at 50% probability.

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Structure of 4-Methyl-1,2,6-tristibatricyclo[2.2.1.$0^{2,6}$]heptane, $C_5H_9Sb_3$

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Dedicated to Professor Dr Dr h.c. H. Behrens on the occasion of his 70*th birthday*

Abstract. $M_r = 434\cdot4$, monoclinic, $A2/m$, $a = 8\cdot574\,(3)$, $b = 9\cdot576\,(2)$, $c = 11\cdot354\,(3)$ Å, $\beta = 105\cdot26\,(2)^\circ$, $V = 899\cdot4$ Å^3, $Z = 4$, $D_x = 3\cdot20$ g cm^{-3}, $\lambda(\mathrm{Ag}\,K\alpha) = 0\cdot55936$ Å, $\mu = 46\cdot4$ cm^{-1}, $F(000) = 768$, room temperature. Final $R = 0\cdot047$ for 681 unique reflections (unobserveds included). The molecule consists of a three-membered ring of Sb atoms [Sb—Sb 2·796 (3) and 2·817 (3) Å] and an organic group. All bond lengths and angles are in the expected range. In the crystal the molecules are mainly connected by intermolecular Sb···Sb contacts supplemented by van der Waals forces between methyl groups.

Introduction. The title compound is the first all-*cis* cyclic tristibine (Ellermann & Veit, 1982); thus it was necessary to correlate spectroscopic results with an X-ray structure determination.

Differences from the analogous arsenic compound (Thiele, Zoubek, Lindner & Ellermann, 1978), whose structure has already been determined, are important for further investigations.

Experimental. Light-red rhombic plates of crystals grown from tetrahydrofuran; crystal dimensions 0·32 × 0·22 × 0·14 mm; lattice parameters determined by a Guinier photograph, using refinement procedure *GIVER* (Krogmann, 1966); possible space groups: $A2$, Am, $A2/m$; $A2/m$ (non-standard setting of $C2/m$) gave the best agreement in the structure refinement. Intensities collected on a Philips PW 1100 diffractometer, using Ag $K\alpha$ radiation and a graphite monochromator, ω–2θ scans, scan width: $(1\cdot20 + 0\cdot2\tan\theta)^\circ$, $2 \leq 2\theta \leq 18^\circ$, 2861 reflections measured; symmetrically equivalent reflections averaged, 681 unique reflections (unobserveds included), index range $h \pm 9$, k 0/10, l 0/12; $R_{\mathrm{int}} = 0\cdot016$. Three standard reflections used for control of stability. Spherical absorption correction applied with $\mu R = 0\cdot54 \pm 0\cdot20$; Sb positions derived by Patterson techniques; a subsequent Fourier map revealed all C atoms, only some of the H atoms could be found in the ΔF map and no H atoms were included; final least-squares refinement on F used corrections for extinction and anomalous dispersion; weights $w = 1/\sigma^2$. Scattering factors taken from *International Tables for X-ray Crystallography* (1974).

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