plexes $[Mn(\eta^5\text{-}C_6H_6R)(CO)_3]$, R = H, Ph [2·17 (1) Å] (Churchill & Scholer, 1969), as well as with the distance of 2·15 (2) Å in the neutral η^5-cyclopentadienyl complex $[Mn(\eta^5\text{-}C_5H_5)(CO)_3]$ (Berndt & Marsh, 1963).

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Structures of Bis{*N*-[1-(2-pyrrolyl)ethylidene]ethylaminato}zinc(II) and Bis{*N*-[1-(2-pyrrolyl)ethylidene]ethylaminato}copper(II)

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Abstract. $[Zn(C_8H_{11}N_2)_2]$, (1), M_r = 335·75, triclinic, $P\bar{1}$, a = 7·849 (1), b = 9·783 (1), c = 12·097 (2) Å, α = 77·12 (1), β = 87·06 (1), γ = 67·20 (1)°, V = 834·1 Å^3, Z = 2, D_x = 1·337 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, μ = 2·035 mm^{-1}, F(000) = 352, T = 293 (1) K, R = 0·038 for 2660 reflections with $I > 3\sigma(I)$. $[Cu(C_8H_{11}N_2)_2]$, (2), M_r = 333·92, monoclinic, $P2_1/c$, a = 14·405 (4), b = 8·281 (6), c = 14·205 (7) Å, β = 104·49 (3)°, V = 1640·6 Å^3, Z = 4, D_x = 1·352 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·332 mm^{-1}, F(000) = 700, T = 293 (1) K, R = 0·039 for 1929 reflections with $I > 3\sigma(I)$. The geometry around the Zn atom in (1) is distorted tetrahedral with Zn—N distances in the range 1·962 (2)–2·041 (2) Å and N—Zn—N *cis* angles involving N atoms of the same ligand 84·2 (1) and 84·1 (1)° and *trans* angles in the range 118·2 (1)–129·5 (1)°. The Cu atom in (2) has distorted square-planar geometry with Cu—N distances in the range 1·935 (3)–2·000 (4) Å and N—Cu—N *cis* angles 82·8 (2) and 83·0 (2)° involving the N atoms of the same ligand and 102·3 (2) and 103·3 (2)° involving N atoms of the two ligands; the *trans* angles are 152·6 (2) and 156·0 (2)°.

Introduction. We have recently reported the crystal structure of (2-acetylpyrolato){*N*-[1-(2-pyrrolyl)ethylidene]ethylaminato}copper(II), (3) (Parvez & Birdsall, 1988), a mixed-ligand complex containing one ethylamine Schiff base and one 2-acetylpyrrole chelate per Cu^{II} center. This compound formed upon spontaneous hydrolysis of bis{*N*-[1-(2-pyrrolyl)ethylidene]ethylaminato}copper(II), and exhibits a distorted square-planar geometry. The mixed-ligand nature of (3) is unusual, but bond angles and bond distances fall within the range reported for bis-bidentate Schiff-base complexes of Zn^{II} (Kanters, Spek, Postma, van Stein & van Koten, 1983), Co^{II} (Wei, 1972*a*), Ni^{II} (Wei & Einstein, 1972), and two modifications of Cu^{II} with the same ligand (Wei, 1972*b*).

We have now synthesized bis{*N*-[1-(2-pyrrolyl)ethylidene]ethylaminato}zinc(II), (1) and bis{*N*-[1-(2-pyrrolyl)ethylidene]ethylaminato}copper(II), (2). Compound (1) was prepared by published methods (Holm, Chakravorty & Theriot, 1966; Yeh & Barker, 1967). Compound (2) resulted from attempts to hydrolyze the second Schiff-base ligand on (3) to form a bis-bidentate 2-acetylpyrrole Cu^{II} product;

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surprisingly, (3) decomposed upon hydrolysis and reformed as a bis-bidentate Schiff-base complex.

Experimental. For both structures, accurate cell constants and orientation matrices were obtained by least-squares refinement of the setting angles of 25 reflections in the range $10 < \theta < 15°$ on Enraf–Nonius CAD-4 diffractometers using graphite-monochromatized Cu $K\alpha$ and Mo $K\alpha$ radiation for (1) and (2), respectively. In each case intensities were collected by the $\omega/2\theta$ scan method. The stability of each crystal was monitored by measurement of three reflections at regular intervals; the data were corrected for decay by appropriate scaling. The data were corrected for Lorentz and polarization factors and for absorption (North, Phillips & Mathews, 1968) for both structures. A summary of data collection and structure refinement parameters for both structures is given in Table 1.

The structures were solved by the heavy-atom method and refined by full-matrix least-squares calculations on F's, initially with isoptropic and finally with anisotropic temperature factors for the non-H atoms. At intermediate stages in the refinements, difference maps revealed all H atoms in both structures which were included in the subsequent rounds of calculations at fixed positions and with overall isotropic thermal parameters. Refinements converged with $R = 0·038$ and 0·039 and $wR = 0·064$ and 0·072 for (1) and (2), respectively. In the refinement cycles, weights were derived from the counting statistics. Scattering factors were those of Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) and allowance was made for anomalous dispersion (Cromer & Liberman, 1970). The computer programs used were part of the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985). The final fractional coordinates with e.s.d.'s are given in Table 2* and molecular dimensions are in Table 3. Figs. 1 and 2 show the *ORTEP*II (Johnson, 1976) drawings of the molecular structures of (1) and (2), respectively while the crystal packing diagrams are depicted in Figs. 3 and 4.

Table 1. *Summary of data collection and structure refinement parameters*

	(1)	(2)
Crystal size (mm)	0·20 × 0·14 × 0·32	0·27 × 0·18 × 0·32
Radiation	Cu $K\alpha$	Mo $K\alpha$
θ limits (°)	5–70	2–26
Range of indices: h	0 to 9	0 to 17
k	−10 to 11	0 to 10
l	−14 to 14	−16 to 17
Scan width (°)	$(0·75 + 0·14 \tan\theta)$	$(0·60 + 0·35 \tan\theta)$
Variable scan speed (° min^{-1})	1·8–8·2	0·75–3·3
Crystal decay (%)	2·7	4·5
Absorption coefficient: max.	0·9974	0·9977
min.	0·8241	0·9229
Unique data measured	3153	3460
Observed data [$I > 3\sigma(I)$]	2660	1929
R, wR	0·038, 0·064	0·039, 0·072
Data/parameter ratio	10·4	7·5
(Δ/σ) max. in last cycle	< 0·10	< 0·15
Max. and min. $\Delta\rho$ (e Å^{-3})	0·37, −0·24	0·25, −0·44
Weighting scheme	$1/[\sigma^2(F) + 0·080F^2]$	$1/[\sigma^2(F) + 0·080F^2]$
S	1·366	1·507

Table 2. *Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = (1/3)\textstyle\sum_i\sum_j B_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Compound (1)				
Zn	0·39257 (4)	0·24483 (3)	0·73215 (3)	4·721 (7)
N(1)	0·3717 (3)	0·4534 (2)	0·7235 (2)	5·06 (5)
N(2)	0·2915 (3)	0·3274 (2)	0·5682 (2)	4·46 (4)
N(3)	0·6117 (3)	0·0527 (2)	0·7676 (2)	4·92 (5)
N(4)	0·2700 (3)	0·1308 (3)	0·8467 (2)	5·21 (5)
C(1)	0·4020 (4)	0·5459 (3)	0·7817 (2)	6·00 (7)
C(2)	0·3605 (5)	0·6880 (3)	0·7139 (3)	6·42 (7)
C(3)	0·3035 (4)	0·6858 (3)	0·6076 (3)	5·74 (7)
C(4)	0·3125 (3)	0·5395 (3)	0·6159 (2)	4·60 (5)
C(5)	0·2730 (3)	0·4680 (3)	0·5342 (2)	4·41 (5)
C(6)	0·2151 (4)	0·5567 (3)	0·4151 (2)	5·45 (7)
C(7)	0·2540 (4)	0·2467 (3)	0·4899 (2)	5·33 (6)
C(8)	0·2535 (5)	0·0979 (3)	0·5517 (3)	7·05 (8)
C(9)	0·7871 (4)	−0·0184 (4)	0·7394 (3)	5·99 (7)
C(10)	0·8517 (5)	−0·1704 (4)	0·7907 (3)	7·31 (9)
C(11)	0·7112 (5)	−0·1942 (3)	0·8515 (3)	6·78 (8)
C(12)	0·5633 (4)	−0·0560 (3)	0·8380 (2)	5·03 (6)
C(13)	0·3784 (4)	−0·0090 (3)	0·8797 (2)	5·17 (5)
C(14)	0·3209 (5)	−0·1233 (3)	0·9595 (3)	6·91 (8)
C(15)	0·0796 (4)	0·1868 (4)	0·8865 (3)	7·53 (9)
C(16)	0·0027 (5)	0·3558 (5)	0·8747 (3)	7·89 (9)
Compound (2)				
Cu	0·78557 (4)	0·48828 (6)	0·55091 (4)	3·96 (1)
N(1)	0·9072 (3)	0·4001 (5)	0·6223 (3)	3·9 (1)
N(2)	0·8637 (3)	0·6840 (5)	0·5478 (3)	4·1 (1)
N(3)	0·6807 (3)	0·5101 (5)	0·4347 (3)	5·1 (1)
N(4)	0·6914 (3)	0·3491 (5)	0·5954 (3)	4·7 (1)
C(1)	0·9482 (4)	0·2610 (6)	0·6611 (4)	4·7 (1)
C(2)	1·0473 (4)	0·2765 (7)	0·6875 (4)	5·1 (1)
C(3)	1·0681 (3)	0·4302 (7)	0·6614 (3)	4·7 (1)
C(4)	0·9809 (3)	0·5050 (6)	0·6220 (3)	4·2 (1)
C(5)	0·9535 (3)	0·6622 (6)	0·5820 (3)	3·7 (1)
C(6)	1·0302 (4)	0·7864 (7)	0·5845 (4)	5·4 (1)
C(7)	0·8246 (4)	0·8402 (6)	0·5074 (4)	5·7 (1)
C(8)	0·7509 (5)	0·8964 (8)	0·5556 (5)	7·0 (2)
C(9)	0·6590 (4)	0·5765 (8)	0·3452 (4)	6·2 (1)
C(10)	0·5696 (5)	0·5237 (8)	0·2932 (5)	7·1 (2)
C(11)	0·5368 (4)	0·4183 (8)	0·3508 (5)	6·8 (1)
C(12)	0·6059 (3)	0·4110 (6)	0·4399 (4)	4·9 (1)
C(13)	0·6137 (3)	0·3274 (6)	0·5293 (4)	4·8 (1)
C(14)	0·5315 (4)	0·2230 (7)	0·5398 (5)	6·7 (2)
C(15)	0·7067 (4)	0·2814 (7)	0·6929 (4)	6·2 (1)
C(16)	0·7191 (4)	0·4075 (10)	0·7684 (4)	7·0 (2)

Discussion. The Zn atom in (1) is coordinated to two bidentate ligands and has a distorted tetrahedral geometry with two types of Zn—N bond distances; significantly shorter bonds involve pyrrole N atoms [1·962 (2) and 1·971 (2) Å] as compared with those involving ethylaminato N atoms [2·023 (2) and 2·041 (2) Å]. These distances agree well with those reported for a closely related complex, $[Zn(C_9H_{15}N_2)_2]$ (Kanters, Spek, Postma, van Stein & van Koten, 1983). The bond distances and angles in the ligand are in excellent agreement with those

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52773 (52 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Molecular dimensions with e.s.d.'s in parentheses for* (1) *and* (2)

	(1)	(2)		(1)	(2)
M—N(1)	1·962 (2)	1·935 (3)	C(1)—C(2)	1·371 (4)	1·388 (7)
M—N(2)	2·041 (2)	1·980 (4)	C(2)—C(3)	1·390 (6)	1·380 (8)
M—N(3)	1·971 (2)	1·948 (4)	C(3)—C(4)	1·386 (5)	1·387 (7)
M—N(4)	2·023 (2)	2·000 (4)	C(4)—C(5)	1·439 (4)	1·436 (7)
N(1)—C(1)	1·357 (4)	1·347 (6)	C(5)—C(6)	1·497 (3)	1·504 (7)
N(1)—C(4)	1·374 (3)	1·373 (6)	C(7)—C(8)	1·482 (4)	1·476 (9)
N(2)—C(5)	1·297 (3)	1·275 (6)	C(9)—C(10)	1·376 (5)	1·386 (8)
N(2)—C(7)	1·467 (5)	1·469 (6)	C(10)—C(11)	1·360 (6)	1·360 (10)
N(3)—C(9)	1·346 (3)	1·348 (7)	C(11)—C(12)	1·381 (4)	1·401 (7)
N(3)—C(12)	1·370 (4)	1·371 (7)	C(12)—C(13)	1·443 (4)	1·426 (8)
N(4)—C(13)	1·282 (3)	1·282 (6)	C(13)—C(14)	1·500 (4)	1·503 (8)
N(4)—C(15)	1·475 (4)	1·458 (8)	C(15)—C(16)	1·499 (6)	1·475 (11)

	(1)	(2)		(1)	(2)
N(1)—*M*—N(2)	84·2 (1)	82·8 (2)	C(1)—C(2)—C(3)	107·7 (4)	106·9 (4)
N(1)—*M*—N(3)	129·5 (2)	152·6 (2)	C(2)—C(3)—C(4)	105·7 (2)	106·5 (4)
N(1)—*M*—N(4)	125·5 (1)	102·3 (2)	N(1)—C(4)—C(3)	110·3 (3)	109·8 (4)
N(2)—*M*—N(3)	118·2 (1)	103·3 (2)	N(1)—C(4)—C(5)	117·9 (2)	116·0 (4)
N(2)—*M*—N(4)	120·0 (1)	156·0 (2)	C(3)—C(4)—C(5)	131·8 (2)	134·1 (5)
N(3)—*M*—N(4)	84·1 (1)	83·0 (2)	N(2)—C(5)—C(4)	117·6 (3)	115·6 (4)
M—N(1)—C(1)	144·2 (2)	141·7 (3)	N(2)—C(5)—C(6)	123·1 (3)	125·6 (4)
M—N(1)—C(4)	109·8 (2)	111·0 (3)	C(4)—C(5)—C(6)	119·4 (2)	118·9 (4)
C(1)—N(1)—C(4)	106·0 (2)	106·5 (4)	N(2)—C(7)—C(8)	110·8 (2)	110·3 (5)
M—N(2)—C(5)	110·4 (2)	113·9 (3)	N(3)—C(9)—C(10)	110·3 (3)	109·8 (6)
M—N(2)—C(7)	128·8 (1)	124·4 (4)	C(9)—C(10)—C(11)	106·8 (3)	107·3 (5)
C(5)—N(2)—C(7)	120·7 (2)	121·8 (4)	C(10)—C(11)—C(12)	107·6 (3)	107·2 (6)
M—N(3)—C(9)	143·7 (2)	141·7 (4)	N(3)—C(12)—C(11)	108·9 (3)	108·5 (5)
M—N(3)—C(12)	109·2 (2)	110·6 (3)	N(3)—C(12)—C(13)	117·6 (2)	116·7 (4)
C(9)—N(3)—C(12)	106·5 (2)	107·1 (4)	C(11)—C(12)—C(13)	133·6 (3)	134·8 (5)
M—N(4)—C(13)	110·9 (2)	112·8 (4)	N(4)—C(13)—C(12)	117·8 (2)	116·4 (5)
M—N(4)—C(15)	128·4 (2)	124·7 (3)	N(4)—C(13)—C(14)	122·7 (2)	124·9 (5)
C(13)—N(4)—C(15)	120·6 (3)	122·7 (5)	C(12)—C(13)—C(14)	119·6 (3)	118·6 (4)
N(1)—C(1)—C(2)	110·4 (3)	110·4 (5)	N(4)—C(15)—C(16)	111·5 (3)	112·4 (5)

reported for similar complexes, *e.g.* [$Co(C_9H_{15}N_2)_2$] (Wei, 1972*a*), [$Cu(C_9H_{15}N_2)_2$] (Wei, 1972*b*) and [$Ni(C_9H_{15}N_2)_2$] (Wei & Einstein, 1972).

The most notable change in the conversion of (3) to (2) is that *cis* pyrrole groups in (3) become *trans* in (2). Both structures have distorted square-planar geometries, but (2) deviates more from planarity than (3) with *trans* N(1)—Cu—N(3) and N(2)—Cu—N(4) angles of 152·6 (2) and 156·0 (2)°, respectively, while (3) contains *trans* O—Cu—N and N—Cu—N angles of 169·5 (1) and 173·5 (1)°, respectively. Decomposition and reformation of (3) to the *trans* pyrrole complex (2) occurs rather than the hydrolysis of (3) to a bis 2-acetylpyrrole complex with *cis* pyrrole groups. Since (3) itself is a decomposition product of an unstable bis(ethylamine) Schiff-base adduct of 2-acetylpyrrole, the unstable parent complex may undergo hydrolysis to a mixed-ligand product because of non-bonded interactions between ethyl groups on *cis* imine N atoms. Compound (3), therefore, can be viewed as the product of an unstable *cis* Schiff-base bidentate adduct, while compound (2) is a stable decomposition product of (3) containing *trans* bidentate Schiff-base ligands. The Zn adduct (1) is stable because strain between ethyl groups on imine N atoms is not a factor in this tetrahedral complex. Ligand bond distances and angles for (2) agree well with those reported for similar Cu^{II} complexes containing *tert*-butylamine Schiff-base bidentate groups (Wei, 1972*b*).

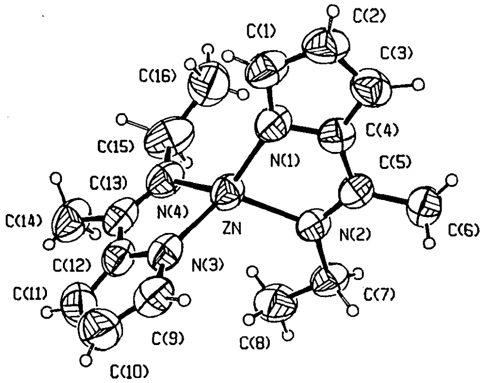

Fig. 1. *ORTEP* drawing of the molecular structure of (1).

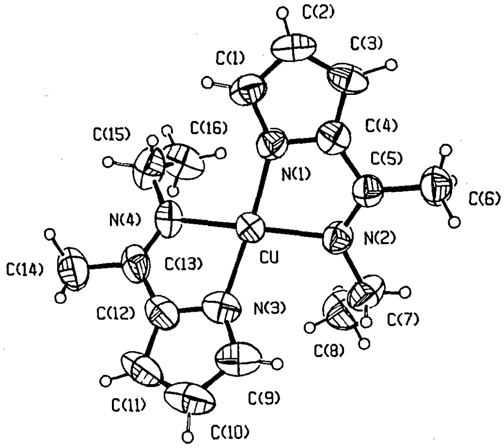

Fig. 2. *ORTEP* drawing of the molecular structure of (2).

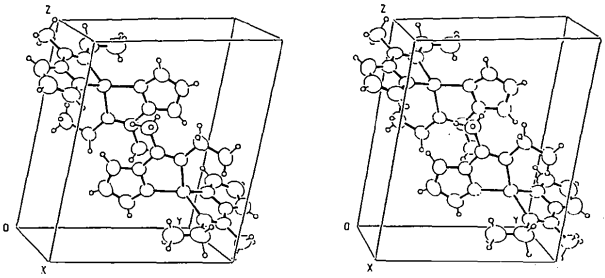

Fig. 3. Stereoscopic view of the crystal packing diagram of (1).

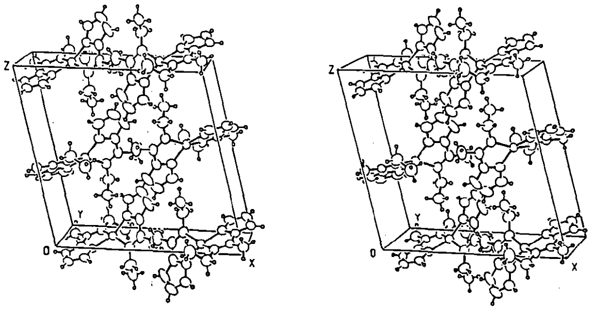

Fig. 4. Stereoscopic view of the crystal packing diagram of (2).

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Structure of Bis(benzene-1,2-dithiolato)gold(IV)

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Abstract. $[Au(C_6H_4S_2)_2]$, $M_r = 477\cdot44$, monoclinic, $P2_1/n$, $a = 12\cdot435\ (2)$, $b = 3\cdot750\ (1)$, $c = 14\cdot495\ (1)$ Å, $\beta = 111\cdot88\ (1)^\circ$, $V = 627\cdot2\ (2)$ Å^3, $Z = 2$, $D_x = 2\cdot52$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 123\cdot0$ cm^{-1}, $F(000) = 446$, $T = 298$ K. Final $R = 0\cdot024$ for 1167 observed reflections. Au has square-planar coordination to four S atoms forming planar units which are regularly stacked along the *b* axis, which makes an angle of 18·5 (1)° with the normal of the complex. The average Au—S distance is 2·300 (1) Å. A weak superstructure is observed but has been ignored in the present analysis.

Introduction. The recent discoveries of superconductivity in (TTF)$[Ni(dmit)_2]_2$ (TTF = tetrathiafulvalene, dmit = 4,5-dimercapto-1,3-dithiole-2-dithione) (Brossard, Ribault, Bousseau, Valade & Cassoux, 1986) and $[(CH_3)_4N]\ [Ni(dmit)_2]$ (Kobayashi, Kim, Sasaki, Kato, Kobayashi, Moriyama, Nishio, Kajita & Sasaki, 1987) have stimulated renewed interest in the structure and properties of related compounds containing a central metal atom (Ni, Pt, Pd or Au) bonded to four S atoms in a nearly square-planar coordination. Such compounds have previously been intensively studied because of their magnetic properties involving a spin-Peierls transition (see, for example, Bray, Interrante, Jacobs & Bonner, 1983). The planar metal complexes, which are often stacked in the crystal structure, may be charged or uncharged corresponding to different formal oxidation states of the metal atom. The present study deals with the neutral gold(IV) complex $[Au(bdt)_2]^0$ (bdt = benzene-1,2-dithiolato) which may be reduced to the anion $[Au(bdt)_2]^-$ corresponding to the well known oxidation state of +3 for Au. A similar compound formally containing gold(IV), bis(5,6-dihydro-1,4-dithiin-2,3-dithiolato)-gold(IV) was reported earlier (Schultz, Wang, Soderholm, Sifter, Williams, Bechgaard & Whangbo, 1987). This material exhibits a pronounced dimerization of the radicaloid gold(IV) species. Such dimerization is not found in the title compound, which contains uniform stacks of formally neutral gold(IV) radicals.

Experimental. Crystals of $[Au(bdt)_2]^0$ were obtained by electrochemical oxidation (3 μA) in a standard H cell of the $(C_4H_9)_4N^+$. $[Au(bdt)_2]^-$ salt. A platinum wire, which could be adjusted to keep only the tip in the electrolyte, was used as the anode. The crystals of best quality were obtained when grown from the tip of the electrode only throughout the entire electrolysis. A crystal of dimensions 0·30 × 0·08 × 0·03 mm was selected for study on an Enraf–Nonius CAD-4 diffractometer. The unit cell was derived from 25 reflections ($12\cdot5 < \theta < 17\cdot5^\circ$). X-ray intensity data