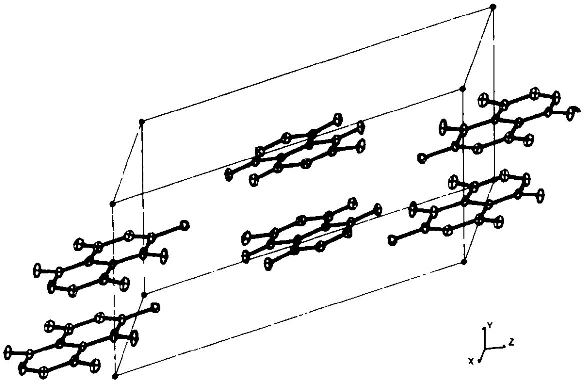

Fig. 2. The packing arrangement of the molecules.

of the lengths of different bond types with those observed in 9,10-anthraquinone (Lonsdale, Milledge & El Sayed, 1966) and 1,5-dihydroxy-9,10-anthraquinone (Hall & Nobbs, 1966) (two similar compounds which have been studied accurately) shows them to be generally in good agreement. Each molecule possesses a planar naphthalene residue with maximum deviations from the mean planes of 0·022 and 0·011 Å respectively ($\sigma = 0{\cdot}014$ Å); some of the substituent atoms are displaced as much as 0·05 Å from coplanarity. There are no intermolecular O···O contacts less than 3·3 Å, and thus the hydrogen bonding is solely intramolecular.

We are indebted to Professor D. E. Williams for providing the program *PCK*5, and for valuable discussions relating to the procedures used.

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The Crystal and Molecular Structure of a Bridged and Intramolecularly Alkylated Cobaloxime, $C_{25}H_{40}N_5O_4Co$

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$C_{25}H_{40}N_5O_4Co$ crystallizes in space group $P2_1/c$, with unit-cell dimensions $a = 9{\cdot}275$ (3), $b = 23{\cdot}308$ (6), $c = 12{\cdot}905$ (4) Å, $\beta = 114{\cdot}29$ (1)°, $Z = 4$. The atomic parameters were refined to a final R value of 0·073 for 2307 observed reflexions. The molecules, in which the bridge is arranged in the *cis* configuration, have no mirror plane.

Introduction

Bis(dimethylglyoximato)cobalt complexes, 'cobaloximes', have been used as model compounds for the more complex cobalamines (Schrauzer, 1968, 1971). Among the cobalamines the vitamin B_{12} coenzyme is of particular interest. Its structure was determined by Lenhert & Hodgkin (1961) and Lenhert (1968). Looking for better model compounds for the coenzyme B_{12} enzymatic reactions, Rétey (1971) synthesized bridged glyoxime cobalt complexes which, for convenience, will also be called cobaloximes. For one of these compounds, $C_{23}H_{37}N_5O_4CoCl.CH_2Cl_2$, where the bridge consists of 12 methylene groups, Bartlett & Dunitz (1971) determined the crystal structure. In a further step, Flohr, Kempe, Pannhorst & Rétey (1976) succeeded in synthesizing an intramolecularly alkylated cobaloxime with the molecular formula $C_{25}H_{40}N_5O_4Co$. This is thought to be a model for the active site of the coenzyme B_{12} containing enzymes, and actually the first model in which, by intramolecular alkylation of the cobalt, the situation on the enzyme is simulated. The exact structure of this complex could not be established by physicochemical techniques and therefore a structure determination was performed. Some results of the structure determination have already been published in a preliminary note (Flohr, Kempe, Pannhorst & Rétey, 1976).

Experimental

Single crystals were kindly provided by H. Flohr and were synthesized according to the method described by Flohr, Kempe, Pannhorst & Rétey (1976). Preliminary unit-cell parameters and space-group information were obtained from precession and Weissenberg photographs taken with Cu radiation. For data collection a plate with typical monoclinic shape was used; the b axis was parallel to the normal of the plate and the crystal had approximate dimensions of 0·25 × 0·20 × 0·05 mm, $\beta \simeq 115°$. The data were collected with a Picker automatic four-circle diffractometer equipped with a Mo tube and a graphite monochromator.

Accurate cell parameters were determined from 16 reflexions with 2θ between 10 and 25° by a least-squares technique. Intensity data were collected with the θ–2θ scanning method and a scan speed of 1·0° min^{-1}. The scan width was 1·15° plus a dispersion correction. Stationary background counts were measured before and after each scan for 40 s. Three reference reflexions were measured after every 47 reflexions and were used to scale the observed intensities. Intensity data were collected for the hkl and $\bar{h}kl$ reflexions over the range $3° \leq 2\theta \leq 50°$. A second set of data was collected for the $h\bar{k}l$ and $\bar{h}\bar{k}l$ reflexions over the range $3° \leq 2\theta \leq 45°$. Averaging of these two sets of data gave 4625 symmetrically independent reflexions, of which 2307 had $I \geq 3\sigma(I)$ and were thereby classified as 'observed'. Lorentz and polarization corrections were applied to all reflexions, but no corrections were made for absorption. The main crystallographic data are given in Table 1.

Table 1. *Crystallographic data*

$C_{25}H_{40}N_5O_4Co$
Monoclinic
$a = 9{\cdot}275\ (3)$ Å
$b = 23{\cdot}308\ (6)$
$c = 12{\cdot}905\ (4)$
$\beta = 114{\cdot}29\ (1)°$
$Z = 4$

$M_r = 533{\cdot}4$
Space group $P2_1/c$
$V = 2543$ Å^3
λ(Mo $K\alpha$) = 0·7093 Å
μ (Mo $K\alpha$) = 7·4 cm^{-1}
$D_{\text{calc}} = 1{\cdot}42$ g cm^{-3}
$D_{\text{exp}} = 1{\cdot}39\ (1)$ (by flotation)

Structure determination

The position of the Co atom was derived from a three-dimensional Patterson map. The positions of all remaining non-hydrogen atoms were deduced from three successive difference Fourier maps that were calculated on the basis of the known part of the structure. The structure was refined by the block-diagonal least-squares method, and the calculations were based on the observed reflexions only. Refinement of the positional parameters and the anisotropic temperature factors of the non-hydrogen atoms led to $R = 0{\cdot}073$ with unit weights.

A difference Fourier map showed electron density maxima between 0·2 and 0·6 e Å^{-3}, corresponding to the H atoms in the structure. The positions of the H atoms, except those of the two methyl groups, were located from two successive difference Fourier maps.

Table 2. *Fractional coordinates* (× 10^4) *for the non-hydrogen atoms, with estimated standard deviations in parentheses*

	x	y	z
Co	940·4 (15)	1198·1 (6)	7880·0 (11)
O(1)	3345 (8)	2050 (3)	8670 (7)
O(2)	−385 (8)	552 (3)	9135 (6)
O(3)	−1344 (8)	298 (3)	7114 (6)
O(4)	2096 (9)	1889 (3)	6590 (7)
N(1)	2460 (9)	1682 (4)	8944 (7)
N(2)	669 (9)	955 (3)	9181 (7)
N(3)	−566 (9)	706 (4)	6810 (7)
N(4)	1102 (10)	1462 (4)	6551 (7)
N(5)	2695 (9)	583 (3)	8140 (6)
C(1)	3811 (13)	1981 (5)	10951 (10)
C(2)	2619 (12)	1639 (4)	10015 (9)
C(3)	1516 (11)	1223 (5)	10129 (8)
C(4)	1388 (13)	1118 (5)	11247 (8)
C(5)	628 (13)	1639 (5)	11605 (9)
C(6)	−1156 (13)	1592 (5)	11280 (9)
C(7)	−2179 (13)	1590 (5)	9965 (9)
C(8)	−2114 (14)	2153 (5)	9400 (10)
C(9)	−3010 (12)	2131 (5)	8082 (9)
C(10)	−2314 (11)	1706 (4)	7483 (8)
C(11)	−3314 (12)	1731 (5)	6178 (8)
C(12)	−4760 (13)	1343 (6)	5769 (9)
C(13)	−5471 (13)	1268 (7)	4444 (10)
C(14)	−4201 (14)	1027 (6)	4049 (9)
C(15)	−3575 (13)	431 (6)	4574 (10)
C(16)	−1792 (12)	346 (5)	4825 (10)
C(17)	−733 (12)	746 (5)	5756 (9)
C(18)	242 (12)	1200 (5)	5608 (8)
C(19)	258 (15)	1375 (6)	4482 (10)
C(20)	−586 (11)	1862 (4)	7745 (9)
C(31)	4060 (11)	737 (5)	8065 (9)
C(32)	5307 (13)	360 (5)	8310 (10)
C(33)	5156 (14)	−201 (5)	8631 (10)
C(34)	3774 (14)	−365 (5)	8692 (10)
C(35)	2552 (13)	37 (5)	8443 (9)

Calculations showed that a refinement of the positional parameters of the H atoms would lead to high estimated standard deviations (>0·1 Å), so that the information obtained would be of little value for chemical considerations. Therefore, no attempt was made to refine the positional parameters of the H atoms. When the H atoms were included in the structure factor calculations with a constant isotropic temperature factor of $B_H = 5{\cdot}0$ Å^2 the R value reduced to 0·062.

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1968). For the Co atom a dispersion correction was introduced with real and imaginary anomalous dispersion terms, as given by Cromer & Liberman (1970). All computations were performed on the Univac 1108 computer at the University of Karlsruhe Computer Center, with the XRAY 70 program system (Stewart, Kundell & Baldwin, 1970). The refined atomic coordinates of the non-hydrogen atoms are given in Table 2.*

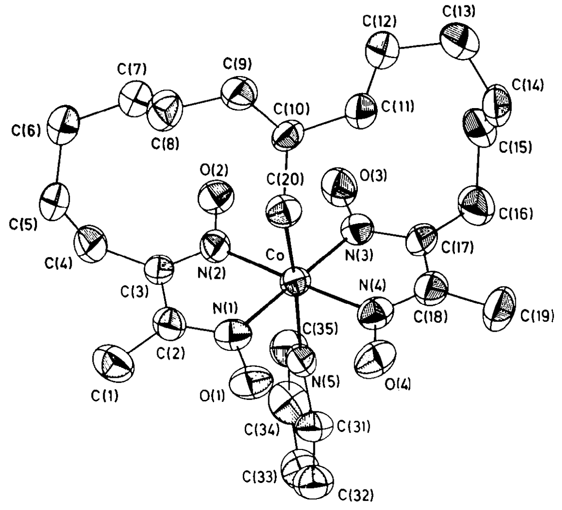

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule showing the atomic labelling. Atoms are represented as 50% probability thermal ellipsoids.

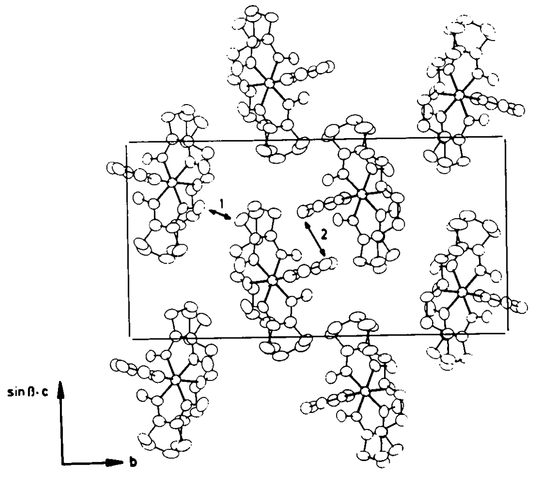

Fig. 2. Packing of the molecules as seen along **a**. The arrows indicate the shortest intermolecular distances.

Table 3. *Intramolecular bond distances* (Å) *and bond angles* (°)

(*a*) Coordination polyhedron

Co–N(1)	1·883 (8)	N(4)–Co–N(1)	98·8 (4)
Co–N(2)	1·885 (9)	N(1)–Co–C(20)	83·9 (4)
Co–N(3)	1·893 (7)	N(2)–Co–C(20)	87·8 (4)
Co–N(4)	1·885 (10)	N(3)–Co–C(20)	96·8 (4)
Co–N(5)	2·090 (8)	N(4)–Co–C(20)	89·2 (4)
Co–C(20)	2·056 (10)	N(1)–Co–N(5)	89·6 (3)
		N(2)–Co–N(5)	91·8 (4)
N(1)–Co–N(2)	82·2 (4)	N(3)–Co–N(5)	89·7 (3)
N(2)–Co–N(3)	98·0 (4)	N(4)–Co–N(5)	91·2 (4)
N(3)–Co–N(4)	81·1 (4)		

(*b*) Equatorial ligands

O(1)–N(1)	1·33 (1)	O(1)–N(1)–C(2)	120 (1)
O(2)–N(2)	1·34 (1)	N(1)–C(2)–C(1)	122 (1)
N(1)–C(2)	1·33 (2)	N(1)–C(2)–C(3)	112 (1)
N(2)–C(3)	1·31 (1)	C(1)–C(2)–C(3)	126 (1)
C(1)–C(2)	1·49 (1)	C(2)–C(3)–C(4)	122 (1)
C(2)–C(3)	1·46 (2)	C(2)–C(3)–N(2)	113 (1)
C(3)–C(4)	1·52 (2)	C(4)–C(3)–N(2)	124 (1)
O(3)–N(3)	1·35 (1)	C(3)–N(2)–O(2)	122 (1)
O(4)–N(4)	1·34 (1)	O(4)–N(4)–C(18)	122 (1)
N(3)–C(17)	1·31 (2)	N(4)–C(18)–C(19)	122 (1)
N(4)–C(18)	1·30 (1)	N(4)–C(18)–C(17)	133 (1)
C(19)–C(18)	1·52 (2)	C(19)–C(18)–C(17)	125 (1)
C(18)–C(17)	1·46 (2)	C(18)–C(17)–C(16)	125 (1)
C(17)–C(16)	1·52 (1)	C(18)–C(17)–N(3)	112 (1)
		C(16)–C(17)–N(3)	123 (1)
		C(17)–N(3)–O(3)	120 (1)

(*c*) Pyridine group

N(5)–C(31)	1·36 (2)	C(35)–N(5)–C(31)	118 (1)
C(31)–C(32)	1·38 (2)	N(5)–C(31)–C(32)	122 (1)
C(32)–C(33)	1·40 (2)	C(31)–C(32)–C(33)	119 (1)
C(33)–C(34)	1·37 (2)	C(32)–C(33)–C(34)	119 (1)
C(34)–C(35)	1·40 (2)	C(33)–C(34)–C(35)	119 (1)
C(35)–N(5)	1·36 (1)	C(34)–C(35)–N(5)	122 (1)

(*d*) Methylene bridge

C(4)–C(5)	1·57 (2)	C(3)–C(4)–C(5)	112 (1)
C(5)–C(6)	1·54 (2)	C(4)–C(5)–C(6)	115 (1)
C(6)–C(7)	1·57 (1)	C(5)–C(6)–C(7)	114 (1)
C(7)–C(8)	1·52 (2)	C(6)–C(7)–C(8)	113 (1)
C(8)–C(9)	1·56 (2)	C(7)–C(8)–C(9)	113 (1)
C(9)–C(10)	1·55 (2)	C(8)–C(9)–C(10)	115 (1)
C(10)–C(11)	1·55 (1)	C(9)–C(10)–C(11)	109 (1)
C(11)–C(12)	1·52 (2)	C(10)–C(11)–C(12)	114 (1)
C(12)–C(13)	1·57 (2)	C(11)–C(12)–C(13)	111 (1)
C(13)–C(14)	1·57 (2)	C(12)–C(13)–C(14)	111 (1)
C(14)–C(15)	1·55 (2)	C(13)–C(14)–C(15)	112 (1)
C(15)–C(16)	1·56 (2)	C(14)–C(15)–C(16)	112 (1)
		C(15)–C(16)–C(17)	112 (1)
C(10)–C(20)	1·54 (2)	C(9)–C(10)–C(20)	110 (1)
		C(11)–C(10)–C(20)	109 (1)
		C(10)–C(20)–Co	117 (1)

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* Lists of structure factors, anisotropic thermal parameters and the positional parameters of the H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32465 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Results and discussion

In Fig. 1 the projection of one molecule is shown, together with the labelling of the atoms. Intramolecular bond distances and bond angles are given in Table 3, while Fig. 2 shows the packing of the molecules in the crystal.

The crystal structure of $C_{25}H_{40}N_5O_4Co$ consists of discrete molecules in which the bridge is arranged in the *cis* configuration. For molecules with the *cis* configuration, a mirror plane through atoms N(5), Co, C(20) and C(10) might be expected, but the symmetry of the molecules is 1. The centrosymmetric structure contains both enantiomorphic forms which are related by symmetry operations of the space group.

The distances and angles within the equatorial* plane are those found in other dimethylglyoxime cobalt complexes (see Palenik, Sullivan & Naik, 1976). The two almost planar glyoxime groups (Table 4) are rotated against each other by an angle of 3·7° about the line bisecting the angles N(1)—Co—N(2) and N(3)—Co—N(4). According to the difference syntheses the H atoms of the two intramolecular hydrogen bonds are attached to O(1) and O(3). Within the system O(1)—H(O1)···O(4) the distances are O(1)—H(O1) = 1·01 Å and H(O1)—O(4) = 1·47 Å, while for the system O(3)—H(O3)···O(2) values of O(3)—H(O3) = 1·01 Å and H(O3)—O(2) = 1·45 Å are obtained. The estimated errors for these values are about 0·1 Å.

Table 4. *Least-squares planes and deviations* (Å $\times 10^3$) *of atoms from the planes*

(*a*) Least-squares planes. The equations of the planes are expressed in direct space as $PX + QY + RZ = S$.

	P	*Q*	*R*	*S*
Glyoxime group 1	−6·372	16·331	1·373	2·402
Glyoxime group 2	−6·798	15·383	1·937	2·766
Pyridine	−0·207	6·524	11·407	9·603

(*b*) Deviations (Å $\times 10^3$) of atoms from the planes. The plane-defining atoms are marked by an asterisk.

Glyoxime group 1

N(1)*	54	N(2)*	−8	O(1)*	4	O(2)*	−2
C(2)*	−20	C(3)*	20	C(1)	−91	C(4)	83
Co	37	N(3)	47	N(4)	183	C(3)	−82
O(4)	253	C(16)	−32	C(17)	73	C(18)	173
C(19)	294						

Glyoxime group 2

N(3)*	24	N(4)*	3	O(3)*	−16	O(4)*	−8
C(17)*	−6	N(18)*	2	C(16)	−80	C(19)	42
Co	−36	N(1)	−119	N(2)	27	O(1)	−208
O(2)	114	C(1)	−188	C(2)	−86	C(3)	47
C(4)	189						

Pyridine

N(5)*	7	C(31)*	−7	C(32)*	1	C(33)*	5
C(34)*	−5	C(35)*	−1				

The planar pyridine group (Table 4) deviates slightly from a symmetrical position with respect to the equatorial ligand system. The angles between the pyridine group and the planes through N(1), N(3), N(5), Co and N(2), N(4), N(5), Co are 53·1 and 45·5°.

The most interesting part of this model compound of vitamin B_{12} coenzyme is the geometry around the C(20) atom, since the corresponding C atom of the coenzyme plays a crucial role in the course of the catalytic processes. The Co—C(20) bond distance is 2·06 Å and agrees well with the corresponding distance in the coenzyme, 2·05 Å (Lenhert, 1968). The N(1–4)—Co—C(20) angles vary between 84 and 97°, and thereby deviate significantly from those of a regular octahedron. Similar deviations from octahedral symmetry are observed in the coenzyme, where the N—Co—C angles vary between 86 and 95°. With respect to the least-squares planes through the glyoxime groups 1 and 2, the line Co—C(20) forms angles of 7·4 and 8·7°. C(20) attaches the methylene bridge to the Co atom with an angle of 117° for Co—C(20)—C(10), which is somewhat smaller than the corresponding angle of 125° in the coenzyme.

From the geometry of the bridge, as well as from the location of the H atoms, it follows that the tertiary H(C10) atom in the middle of the bridge points towards the equatorial plane. The positions of the H atoms as derived from the difference syntheses give C—H bond distances between 0·86 and 1·20 Å, while the H—C—H angles vary between 97 and 132°.

The packing of the molecules in the crystal is determined by van der Waals forces only. The shortest intermolecular distances are O(4)—C′(1) = 3·36 Å and C(33)—C′(34) = 3·44 Å, which are indicated in Fig. 2 by arrows 1 and 2.

The author is indebted to Professor J. Rétey and H. Flohr for suggesting the problem and for their kind interest during the course of this work.

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* The two glyoxime units 1 [O(1), O(2), N(1), N(2), C(2), C(3)] and 2 [O(3), O(4), N(3), N(4), C(17), C(18)] and the atoms Co, C(1), C(4), C(16), C(19) form an almost planar system, which is called the equatorial plane of the molecule.

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5-(*N*-Benzyl-*N*-methyliminioprop-2-enyl)-3,7,8,10-tetramethyl-1,5-dihydroisoalloxazine Perchlorate, a Model Compound of a Flavoprotein Inhibitor Complex

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5-(*N*-Benzyl-*N*-methyliminioprop-2-enyl)-3,7,8,10-tetramethyl-1,5-dihydroisoalloxazine perchlorate, $C_{25}H_{28}N_5O_2^+ClO_4^-.CH_3OH$, M_r = 562·03, is monoclinic, $P2_1/c$, with a = 7·742 (5), b = 17·890 (5), c = 18·830 (5) Å, β = 98·06 (1)°, D_c = 1·36 g cm^{-3}, Z = 4. Spectral and chemical evidence strongly suggests that this photochemically obtained flavin adduct is to be regarded as a relevant model compound of a monoamine oxidase inhibitor complex. The crystal structure confirms that the propargylamine-type inhibitor is covalently bonded to the N(5) position in the flavin nucleus, yielding a dihydroflavin-5-trimethinecyanine structure. The molecular packing is influenced by the presence of an intermolecular NH···O hydrogen bond which links the molecules in pairs across a centre of symmetry.

Introduction

The present investigation was undertaken to elucidate the structure of a photochemically obtained adduct from 3-methyllumiflavin and *N*-methyl-*N*-benzylpropargylamine (Gärtner, Hemmerich & Zeller, 1976). Propargylamine-type compounds are known to react irreversibly with, and to bond covalently to, the flavin component of mitochondrial monoamine oxidase, giving stable inhibitor complexes (Hellerman & Erwin, 1968). It has been shown (Chuang, Patek & Hellerman, 1974) that these very characteristic chromophores are identical with those of isolated synthetic photoadducts of 3-methyllumiflavin and related propargylamine compounds (Zeller, Gärtner & Hemmerich, 1972). By chemical analysis, dihydroflavin-5-trimethinecyanine structures were assigned to these products (Gärtner & Hemmerich, 1975; Maycock, 1975).

The pale-yellow crystals of the photoadduct were synthesized by Dr B. Gärtner, University of Konstanz, Germany (Gärtner *et al.*, 1976). The space group and preliminary cell parameters were determined by photographic methods. At room temperature the decomposition was too rapid to permit a collection of reliable data. Even at low temperature (−125 °C) decomposition was observed, especially along **a**. A single crystal, 0·33 × 0·12 × 0·12 mm, was mounted on an automatic Siemens diffractometer (AED), and the X-ray intensity data ($\theta \le 57°$) were collected at −125 ± 5 °C with graphite-monochromatized Cu $K\alpha$ radiation (λ = 1·54184 Å). The θ–2θ scan technique was used.

2385 reflexions with $\sigma(I_{\rm net})/I_{\rm net} \le 0·25$ were used in the subsequent calculations. The net intensities were corrected for Lorentz, polarization and absorption (μ = 17·44 cm^{-1}) effects.

The crystal structure was solved by direct methods (Karle & Karle, 1966), with a program package based on a linear, weighted, phase-sum formula for phase determination (Norrestam, 1972). The E map based on the set of phases for the best solution revealed the position of the Cl atom together with 11 non-hydrogen atoms. The position of the Cl atom agreed with that found from a three-dimensional Patterson function. After a full-matrix least-squares refinement with isotropic thermal parameters, the remaining non-hydrogen atoms were located from a difference synthesis. The H atoms