

value of 0.0020 \AA^2 , calculated from the estimated standard deviation of the β_{I_j} of the least-squares refinement, indicates that the rigid-body model is only moderately satisfactory.

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The Crystal Structure of $\text{Bi}_6\text{O}_7\text{FCl}_3$

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The structure of $\text{Bi}_6\text{O}_7\text{FCl}_3$ has been determined by symbolic addition and Fourier methods and refined to $R=0.055$ for 1214 independent counter intensities. The crystals are orthorhombic, space group *Pnma*, with $a=20.105$ (6), $b=3.892$ (2), $c=15.432$ (5) Å, $Z=4$. The Bi atoms are of two types, one four-coordinated by O or O and F which form a nearly square plane, the other five-coordinated by two F and three O, all on one side of Bi. The Bi–O, F distances range between 2.19 and 2.51 Å. The fourfold coordination can be described as a square pyramid with the lone pair of electrons at its apex, and the five-coordination as an octahedron with the lone pair at one corner. The coordination polyhedra are linked by sharing edges and corners to form infinite layers parallel to [010]. Between the nets, trigonal prism columns of chloride ions run in the same direction.

Introduction

By heating BiOCl in air above 600°C , it was totally transformed into a new well defined phase, $\text{Bi}_{24}\text{O}_{31}\text{Cl}_{10}$

(Sillén & Edstrand, 1942). The substitution of O^{2-} by F^- in BiOCl is useful in preparing compounds with simple and predictable structures, related to the above phase. These substances can serve as models for the

study of principles of syntheses, the non-stoichiometry, and the possible mechanisms of the reactions. Preparative and structural studies are in progress on the systems Bi_2O_3 – BiOCl and BiOCl – BiOF – Bi_2O_3 . The present paper reports the crystal structure of $\text{Bi}_6\text{O}_7\text{FCl}_3$, a new compound in the latter system.

Experimental

For the preparation of $\text{Bi}_6\text{O}_7\text{FCl}_3$, a mixture of Bi_2O_3 , BiOCl and BiOF in the mole ratio 1:3:1 was heated in a sealed gold capsule for one day at 770°C. The product consisted of colourless needle-shaped crystals. Preliminary Weissenberg photographs indicated orthorhombic symmetry with the systematic absences $0kl$ and $hk0$ for $k+l$ and h odd, respectively, indicating the space groups $Pnma$ (No. 62) and $Pn2_1a$ (No. 33*). Powder photographs were taken in a Guinier–Hägg camera with $\text{Cu } K\alpha$ radiation and KCl ($a=6.2909 \text{ \AA}$) added as an internal standard. Refinement of the cell parameters was performed by least-squares calculations on 27 lines according to Werner (1970). Some crystal data are given in Table 1. Intensities were collected on a Pailred diffractometer with $\text{Mo } K\alpha$ radiation monochromatized by reflexion off the (002) planes of a graphite crystal, the monochromator angle being 6.08° . The single crystal ($0.018 \times 0.022 \times 0.32 \text{ mm}$) was mounted along the needle axis parallel to b . The reflexions $h0l$ – $h4l$ were collected for the Mo range, $\sin \theta/\lambda < 0.80$, by equi-inclination and ω -scan techniques, with a scan rate of $1.0^\circ \text{ min}^{-1}$. The scan range was 3.0° for all reflexions. The stationary background counts were measured for 40 s at each end of the scan interval. The aperture size of the detector was 2.0° . The intensity of one standard reflexion was measured at regular intervals. The largest variation in its intensity was 2.8%.

Reflexions for which the two measured background values differed by more than 3.09 times the e.s.d. of their difference were omitted. The integrated peak counts I were calculated from the total integrated peak counts, the background counts, and the counting time in the usual way. If $\sigma(I)/I \geq 0.387$, the reflexion was considered unobserved. For each layer line both hkl and $\bar{h}kl$ were measured, a total of 3924 reflexions. The intensities were corrected for Lorentz, polarization and absorption effects; the transmission factors varied from 0.159 to 0.276. The unobserved 1839 reflexions were deleted in the refinement. In the final step, mean values of the intensities of the planes hkl and $\bar{h}kl$ were taken, resulting in 1224 independent reflexions.

Structure determination and refinement

The positions of $\text{Bi}(1)$ – $\text{Bi}(6)$ were determined by symbolic addition. The positions of all Cl and $\text{O}(\text{F})$ atoms could be deduced from a difference map. The centric

Table 1. *Crystal data*

$\text{Bi}_6\text{O}_7\text{FCl}_3$, F.W. 1491.23

$Pnma$, $a=20.105$ (6), $b=3.892$ (2), $c=15.432$ (5) \AA
 $V=1207.8 \text{ \AA}^3$, $Z=4$
 $D_m=8.27$, $D_x=8.20 \text{ g cm}^{-3}$
 $\mu(\text{Mo } K\alpha)=835 \text{ cm}^{-1}$

space group $Pnma$ and the non-centric $Pn2_1a$ are both possible, in view of the systematic absences. The least-squares refinement in $Pnma$ progressed normally. As a reasonable structure and reasonable thermal parameters were obtained for $Pnma$, a refinement in $Pn2_1a$ was not performed. All ligands except Cl were treated as if they were O atoms in the calculations. In the structure there are two possible positions for the F atoms, according to Pauling's second rule in calculating $\sum s$ (the sum of the strength of the electrostatic valence bonds) (Pauling, 1929). However, R was equal for both positions.

Table 2. *Positional and thermal parameters obtained in the final least-squares refinement*

The anisotropic thermal parameters are based on the expression $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2kl\beta_{23} + 2hl\beta_{13})]$. By symmetry $\beta_{12} = \beta_{23} = 0$. The β_{ij} values are multiplied by 10^4 . Estimated standard deviations are given in parentheses.

	x	y	z	B (\AA^2)
$\text{Bi}(1)$	−0.06855 (9)	$\frac{1}{2}$	0.0478 (2)	[1.91]
$\text{Bi}(2)$	−0.24070 (7)	$\frac{1}{2}$	−0.09002 (9)	[0.64]
$\text{Bi}(3)$	0.09231 (9)	$\frac{1}{2}$	0.16117 (11)	[1.36]
$\text{Bi}(4)$	0.26934 (7)	$\frac{1}{2}$	0.28481 (9)	[0.75]
$\text{Bi}(5)$	0.55672 (7)	$\frac{1}{2}$	0.06361 (10)	[0.78]
$\text{Bi}(6)$	0.43114 (10)	$\frac{3}{4}$	0.21717 (13)	[2.16]
$\text{Cl}(1)$	0.1390 (5)	$\frac{3}{4}$	0.5617 (8)	1.4 (2)
$\text{Cl}(2)$	0.3176 (5)	$\frac{3}{4}$	0.4369 (7)	1.0 (1)
$\text{Cl}(3)$	0.1418 (5)	$\frac{3}{4}$	0.3129 (7)	1.0 (1)
$\text{O}(1)$	0.2092 (13)	$\frac{1}{4}$	0.1516 (17)	0.5 (4)
$\text{O}(2)$	0.4969 (13)	$\frac{1}{4}$	−0.0604 (19)	0.6 (4)
$\text{O}(3)$	0.0419 (12)	$\frac{1}{4}$	0.0319 (16)	0.8 (3)
$\text{O}(4)$	0.3782 (17)	$\frac{1}{4}$	0.2862 (23)	1.7 (5)
$\text{O}(5)$	−0.2914 (12)	$\frac{1}{4}$	−0.2167 (23)	1.6 (5)
$\text{O}(6)$	0.4829 (22)	$\frac{3}{4}$	0.3431 (31)	3.0 (8)
$\text{O}(7)$	−0.1283 (17)	$\frac{3}{4}$	−0.0944 (24)	1.7 (6)
F	0.5032 (14)	$\frac{1}{4}$	0.1900 (20)	0.9 (4)
	β_{11}	β_{22}	β_{33}	β_{13}
$\text{Bi}(1)$	4.7 (0.4)	283 (14)	72.7 (1.6)	8.6 (0.6)
$\text{Bi}(2)$	4.3 (0.3)	103 (9)	6.3 (0.5)	−0.2 (0.3)
$\text{Bi}(3)$	11.5 (0.4)	258 (12)	9.7 (0.7)	−2.8 (0.4)
$\text{Bi}(4)$	6.1 (0.3)	123 (9)	6.1 (0.5)	−0.4 (0.3)
$\text{Bi}(5)$	3.9 (0.3)	108 (9)	12.7 (0.6)	−1.8 (0.4)
$\text{Bi}(6)$	10.4 (0.4)	703 (19)	15.4 (0.7)	2.4 (0.5)

The F atom was assumed to be in the position given in Table 2. The final refinement in $Pnma$ by the least-squares method with anisotropic temperature factors for Bi and isotropic for the light atoms, gave $R=0.055$ and $R_w=0.071$, where $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ and $R_w = [\sum w_i(|F_o| - |F_c|)^2 / \sum w_i |F_o|^2]^{1/2}$. Cruickshank's weighting scheme, with $a=300$, $c=0$ and $d=0$, was used. $[\sum w_i(|F_o| - |F_c|)^2 / (m-n)]^{1/2}$, where m and n are the number of observations and parameters varied, was

* Orientation different from that given in *International Tables for X-ray Crystallography* (1965).

Table 4. *Interatomic distances and angles*

Interatomic distances (Å) in $\text{Bi}_6\text{O}_7\text{FCl}_3$ compared with the corresponding distances in BiOF and BiOCl (Aurivillius, 1964a. The estimated standard deviations are given in parentheses.

	$\text{Bi}_6\text{O}_7\text{FCl}_3$	BiOF	BiOCl		$\text{Bi}_6\text{O}_7\text{FCl}_3$	BiOF	BiOCl
Bi-Bi	≥ 3.584 (3)	≥ 3.694 (3)	≥ 3.71 (1)	Bi(4)-2O(5)	2.25 (2)		
Bi(1)-2Cl(2)	3.168 (8)			Bi(5)-2O(2)	2.23 (1)		
Bi(2)-2Cl(1)	3.135 (9)			Bi(5)-O(2)	2.26 (3)		
Bi(2)-2Cl(2)	3.212 (9)			Bi(6)-2O(4)	2.46 (2)		
Bi(3)-2Cl(3)	3.203 (9)		3.49 (4)	Bi(6)-O(6)	2.20 (5)		
Bi(4)-2Cl(2)	3.143 (9)		3.07 (2)	Bi(1)-2O(6)	2.77 (3)		
Bi(4)-2Cl(3)	3.247 (8)			Bi(3)-2O(6)	2.94 (3)		
Bi(5)-2Cl(1)	3.204 (10)			Bi(5)-F	2.22 (3)		
Bi(5)-2Cl(3)	3.217 (8)			Bi(6)-2F	2.46 (2)	2.75 (2)	
Bi(1)-O(7)	2.51 (4)			Cl-O	3.32 (3)-3.43 (2)		≥ 3.25 (3)
Bi(1)-2O(3)	2.36 (1)			F-O	≥ 2.79 (3)	2.75 (5)	
Bi(1)-O(3)	2.24 (2)			F-F	3.892 (=b)	3.44 (8)	
Bi(2)-2O(1)	2.26 (1)			Cl-Cl	3.84 (2)-3.90 (2)		3.48 (5)
Bi(2)-O(5)	2.21 (3)			O(1)-2O(5)	2.74 (3)		
Bi(2)-O(7)	2.26 (3)			O(1)-2O(7)	2.69 (3)		
Bi(3)-O(1)	2.36 (3)	2.273 (2)	2.309 (4)	O(2)-2O(2)	2.70 (4)		
Bi(3)-O(3)	2.24 (2)			O(3)-2O(7)	2.78 (3)	2.6494 (4)	2.7457 (5)
Bi(3)-2O(7)	2.31 (2)			O(3)-2O(3)	2.76 (3)		
Bi(4)-O(1)	2.37 (3)			O(4)-2O(5)	2.82 (4)		
Bi(4)-O(4)	2.19 (3)			O(4)-2O(6)	3.00 (4)		

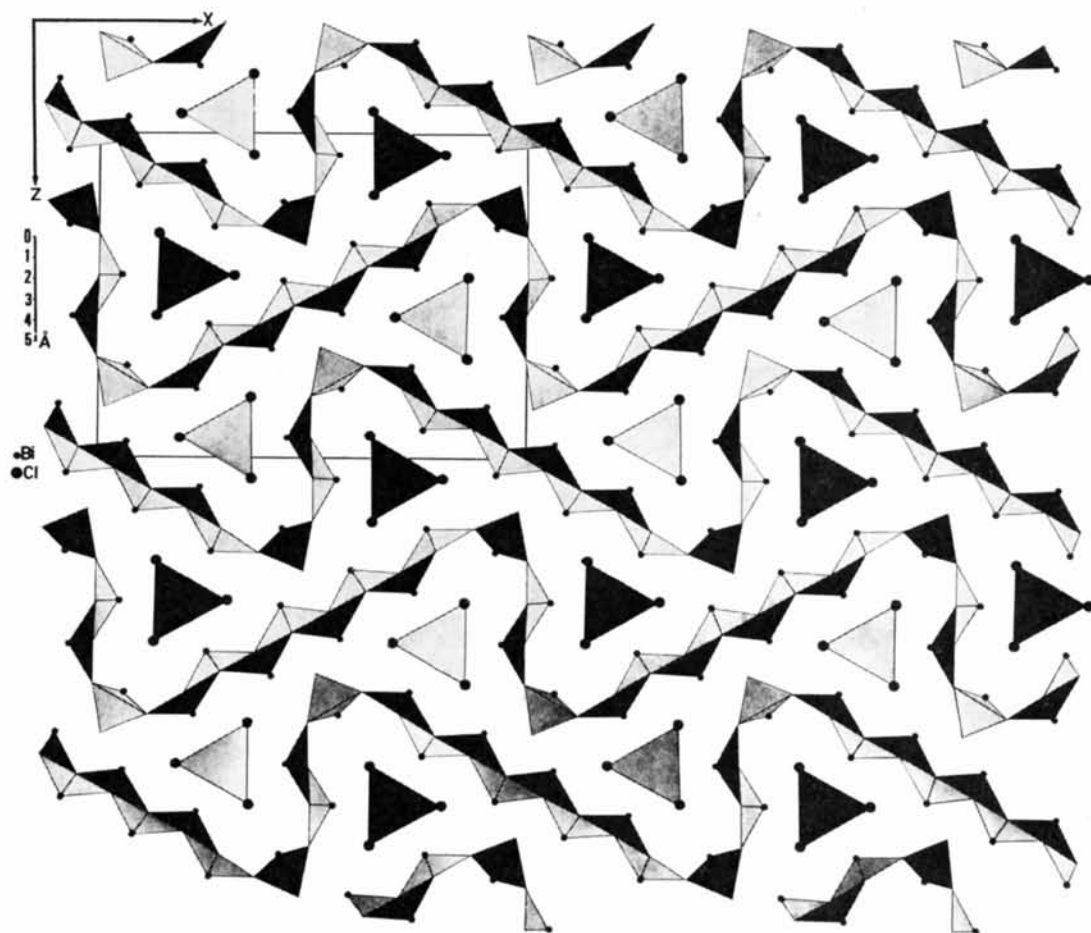


Fig. 1. The structure of $\text{Bi}_6\text{O}_7\text{FCl}_3$ projected along the y axis. The bismuth atoms are coordinated to oxygen (fluorine) atoms in the form of square pyramids and octahedra, connected to infinite layers parallel to $[010]$. The heavier and the lighter polyhedra are $b/2$ apart. The chloride ions form trigonal prisms, running in the same direction. All atoms are at the heights $y = \frac{1}{4}$ or $\frac{3}{4}$.

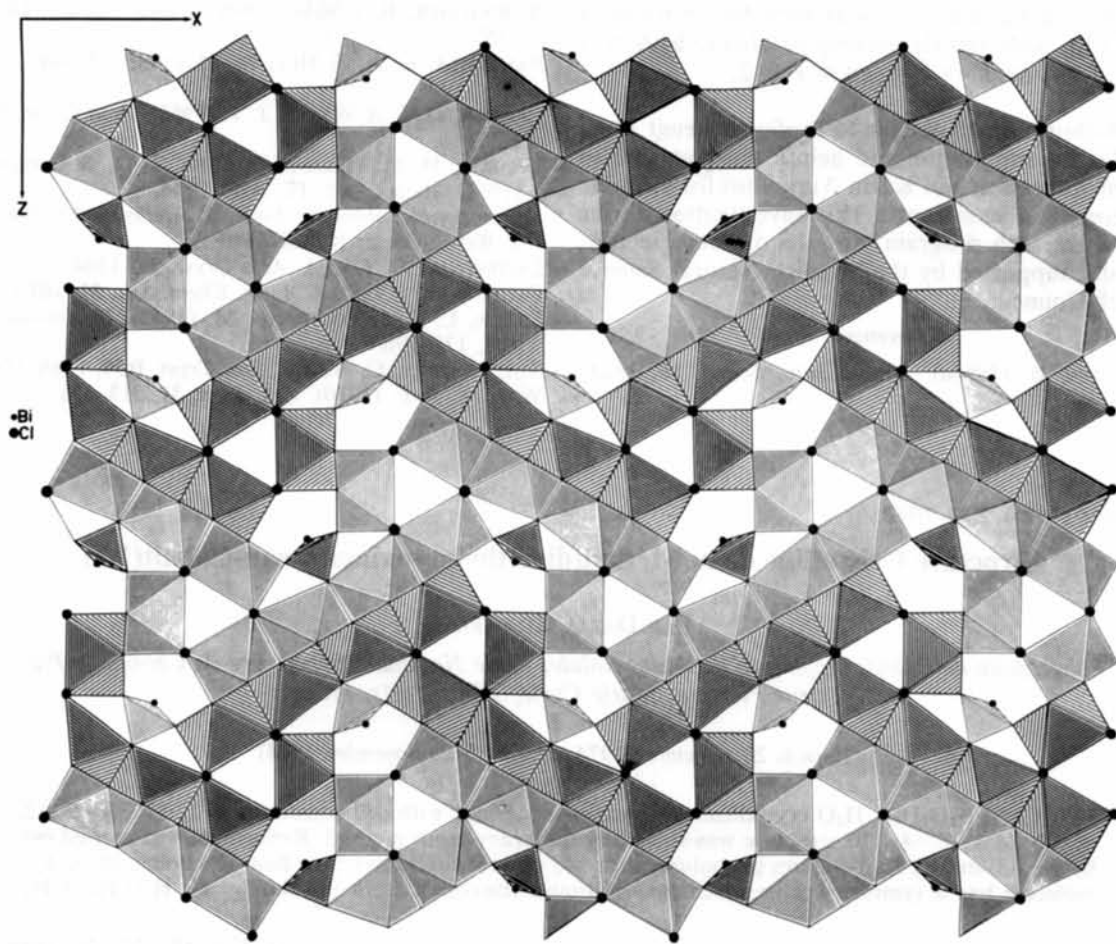


Fig. 2. The structure of $\text{Bi}_6\text{O}_7\text{FCl}_3$ projected along the y axis. The coordination polyhedra of bismuth are described as octahedra and square antiprisms, the bismuth atoms in the latter case coordinated to eight ligands (oxygen, fluorine and chlorine). The polyhedra are fused to a three-dimensional network, interleaved by tunnels running along $[010]$. The heavier and lighter polyhedra are $b/2$ apart. All atoms are at the heights $y = \frac{1}{4}$ or $\frac{3}{4}$.

Table 4 (cont.)

Selected angles ($^\circ$) in $\text{Bi}_6\text{O}_7\text{FCl}_3$

The square plane

O(5)–O(1)–O(5)	90.4 (1.3)	O(7)–O(3)–O(7)	88.7 (1.2)
O(7)–O(1)–O(7)	92.7 (1.4)	O(7)–O(3)–O(3)	90.8 (0.6)
O(2)–O(2)–O(2)	92.3 (1.8)	O(5)–O(4)–O(5)	87.4 (1.4)
O(2)–O(2)–F	89.5 (0.7)	O(4)–O(5)–O(1)	91.1 (0.6)
F–O(2)–F	88.6 (1.2)	O(1)–O(7)–O(3)	89.3 (0.5)
O(3)–O(3)–O(3)	89.7 (1.4)		

In the octahedron

O(4)–O(4)–F	90.00 (0.0)	O(4)–O(6)–O(4)	81.0 (1.4)
F–F–O(4)	90.00 (0.0)	O(4)–O(6)–F	57.2 (1.0)
F–O(6)–F	78.0 (1.3)		

The trigonal prism

Cl(1)–Cl(2)–Cl(3)	59.5 (0.3)
Cl(1)–Cl(3)–Cl(2)	61.0 (0.3)
Cl(2)–Cl(1)–Cl(3)	59.5 (0.3)

BiOF (Aurivillius, 1964a). $\text{Bi}(6)$ (Table 2), on the other hand, is surrounded by two F and three O atoms, all on one side. The lone pair of electrons completes a

distorted octahedron (Table 4). The square pyramids are joined by sharing edges, forming layers which are connected to the octahedra by sharing corners. The nets of formula $[\text{Bi}_6\text{O}_7\text{F}^{3+}]_n$ zigzag through the structure parallel to b . Between them are trigonal prism columns of chloride ions, running in the same direction. A projection of the described layer structure is given in Fig. 1. In the structure of red PbO (Leciejewicz, 1961), there are similar layers consisting of square pyramids.

The coordination around $\text{Bi}(1)–\text{Bi}(5)$ can also be described as a square antiprism. $\text{Bi}(1)$, $\text{Bi}(3)$ are each surrounded by two Cl and six O atoms, $\text{Bi}(2)$, $\text{Bi}(4)$ by four Cl and four O atoms and $\text{Bi}(5)$ by four Cl one F and three O atoms. This type of coordination polyhedron of bismuth has also been found in the structure of BiOHCrO_4 (Aurivillius 1964b). The square antiprisms are joined by sharing faces and edges with other antiprisms, forming blocks, which are connected by sharing corners with the described above octahedra. The structure can in this way be described as being

built up of a three-dimensional network of formula $[\text{Bi}_6\text{O}_7\text{FCl}_3]_n$ with tunnels running parallel to **b**. A projection of this network is given in Fig. 2.

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Structure Cristalline de la Méthyl(diméthylglyoximato)eau-Cobalt(III)

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$\text{CH}_3 \cdot \text{Co}(\text{C}_4\text{N}_2\text{O}_2\text{H}_7)_2 \cdot \text{H}_2\text{O}$ crystallizes in space group *Pnma*, with cell constants $a=13.136$, $b=9.112$, $c=12.114$ Å, $Z=4$. The structure was solved by the heavy-atom method. Refinement was carried out by the full-matrix least-squares procedure, with 946 observed reflexions; the final *R* value is 10%. The molecule has a symmetry plane which passes through the cobalt atom and the ligands H_2O and CH_3 .

Introduction

La méthyl(diméthylglyoximato)eau-cobalt(III) fait partie de la série des méthylcobaloximes synthétisées et étudiées par Benlian & Hernandez (1971). Nous avons entrepris l'étude cristallographique du composé pour examiner s'il y a une influence *trans* sensible du ligand H_2O sur le ligand CH_3 .

Partie expérimentale

Les cristaux sont des aiguilles rouge-brun qui cristallisent dans l'eau. Le groupe spatial et les dimensions de la maille ont été déterminés par enregistrement en chambre de Weissenberg. Les valeurs des paramètres de la maille et les intensités des réflexions ont été mesurées au laboratoire de recherche Philips à Eindhoven sur le diffractomètre à quatre cercles PW 1100. Les intensités ont été mesurées avec la radiation du molybdène pour les angles θ inférieurs à 25° .

Sur 1356 réflexions indépendantes, 946 réflexions répondant au critère $I > 2\sigma(I)$ ont été utilisées pour la détermination et l'affinement de la structure. Les intensités diffractées ont été corrigées des facteurs de Lorentz et de polarisation. Les effets de l'absorption ont été négligés. Les données cristallographiques sont

les suivantes: $\text{C}_9\text{N}_4\text{O}_5\text{H}_{19}\text{Co}$, $M=322,21$; orthorhombique *Pnma*, $a=13,136$ (5); $b=9,112$ (3); $c=12,114$ (4) Å; $V=1450,1$ Å³; $Z=4$; $D_m=1,45$; $D_c=1,48$ g cm⁻³; $\mu=12,53$ cm⁻¹ pour λ (Mo $K\alpha$).

Détermination de la structure et affinement

La structure a été résolue par la technique de l'atome lourd. L'étude de la fonction de Patterson tridimensionnelle a permis de localiser l'atome de cobalt ainsi que l'atome de carbone de la liaison $\text{Co}-\text{CH}_3$ et l'atome d'oxygène de la liaison $\text{Co}-\text{H}_2\text{O}$, ceux-ci sont en position spéciale sur les plans de symétrie de la maille. Les positions des autres atomes ont été déterminées par des synthèses de Fourier successives.

L'affinement des coordonnées atomiques et des coefficients de température a été réalisé par la méthode des moindres carrés (matrice complète). Pour tous les atomes avec des facteurs d'agitation thermique isotrope, le facteur *R* est égal à 0,12 ($R = \sum | \Delta F | / \sum | F_o |$).

L'affinement de la structure a été poursuivi avec des facteurs d'agitation thermique anisotrope sans les atomes d'hydrogène, car ceux-ci ne sont pas repérables avec précision sur la série de densité électronique diffrérence. En fin d'affinement, nous avons utilisé un schéma de pondération de la forme: $w = 5 - 0,125 | F_o | +$