Distance and angle program: *ORFFE* (Busing, Martin & Levy, 1964)

The last three programs were used from the SUNY Buffalo crystallographic computer programs, a system developed under the guidance of P. Coppens.

The authors wish to thank the Danish International Development Agency for financial aid, and the Facultad de Quimica de Montevideo for a leave of absence to Raul A. Mariezcurrena. They are much indebted to Dr J. Danielsen for use of his program *SYMBAD* and for advice on direct methods. Mrs R. Grønbæk Hazell is thanked for helpful advice on computing and for helpful discussions.

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The Crystal Structure of Potassium Boromalate ($KBC_8H_8O_{10}.H_2O$)

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The crystal and molecular structure of the title compound has been determined by X-ray diffraction techniques. The space group is $P2_1$, with two formula units per unit cell. The cell parameters are $a=5{\cdot}54$ (2), $b=11{\cdot}98$ (2), $c=10{\cdot}74$ (2) Å, $\beta=113{\cdot}2$ (5)°. Intensities h,k,l, $k=0$ to 14, were measured using a linear diffractometer equipped with a crystal monochromator and a scintillation counter. 1680 independent reflexions were obtained. The structure was solved by direct methods combined with Patterson methods and chemical knowledge. It was refined by full-matrix least-squares methods to a final R value of 0·056 using 1638 independent reflexions considered to be observable. The two malate groups showed differences in the thermal parameters and in bond lengths. A refinement constraining the two malate groups to have identical geometry gave an R value of 0·087, and confirmed the differences in the thermal parameters. The potassium and the boron atoms are coordinated to oxygen atoms of the carboxyl and hydroxyl groups. The boron atom is tetrahedrally coordinated, and the potassium atom exhibits five-coordination.

Introduction

The acidity of orthoboric acid, $B(OH)_3$, is greatly increased by the addition of polyhydroxy compounds like glycerol, mannitol, *etc.* through the formation of complex compounds. Studies of acid–base equilibria and of electrolytic conductance have confirmed the existence of negative complex ions containing boron. Few such crystalline compounds have been characterized and to our knowledge only one, boromycin, has been subjected to X-ray structure analysis (Dunitz, Hawley, Miklos̆, White, Berlin, Marus̆ić & Prelog, 1971). The classical work of Böeseken and his collaborators showed that α-hydroxy-acids can also take part in complex formation with boric acid, and potassium boromalate is one of the few crystalline complexes of boron with hydroxyl compounds which has been reported. It is described in a paper by Jones (1933) and we obtained crystals suitable for X-ray crystallography by following his procedure.

The aim of our investigation was the determination of the geometry of a boric acid complex and the study of boron–oxygen distances in a compound of this kind.

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Experimental

Crystal data

$a=5{\cdot}54$ (2), $b=11{\cdot}98$ (2), $c=11{\cdot}74$ (2) Å, $\beta=113{\cdot}2$ (5)°. $V=656{\cdot}2$ Å^3, formula $KBC_8H_8O_{10}.H_2O$, F.W. 331·9, $D_m=1{\cdot}680$, $D_c=1{\cdot}681$ g cm^{-3}, $Z=2$, $\mu=4{\cdot}6$ cm^{-1} (Mo $K\alpha$).

Oscillation, Weissenberg and precession photographs were taken with a crystal mounted along the unique b axis using Mo $K\alpha$ radiation ($\lambda=0{\cdot}7107$ Å). The space group ($P2_1$) was determined from the systematic absences, $0k0$ absent for k odd, and from the knowledge that the molecule is optically active ($[\alpha]_D^{26}=5{\cdot}05°$ cm^2 g^{-1}).

Lattice constants were determined from the photographic data and were confirmed by diffractometer measurements. The measured density of 1·680 g cm^{-3} agrees well with the value of 1·681 g cm^{-3} calculated assuming two formula units per unit cell. A sphere of 0·60 mm diameter was ground from a large crystal. The crystal was mounted along the b axis. A linear diffractometer of the Arndt–Phillips design was used for data collection. Graphite monochromated radiation was employed. A scintillation counter in conjunction with a pulse-height analyser was used for intensity measurements. Intensities hkl, $k=0$ to 14, were recorded. Averaging symmetry-related pairs, 1680 independent reflexions were obtained. Of these 42 had a calculated intensity which was less than twice their standard deviations, estimated as the square root of the total number of counts in an intensity measurement. These weak reflexions were considered unobserved.

Structure determination and refinement

The structure contains one heavy atom per asymmetric unit. Consequently, electron density maps phased on the heavy atom only exhibit a pseudo mirror plane parallel to (010). This false symmetry can in principle be broken by adding light atoms to constitute a non-centrosymmetric partial structure as an initial structure.

The three-dimensional Patterson function readily yielded the position of the potassium atom and a number of partial structures were established using superposition methods. Tangent refinement was used for phasing $|E|$ values for electron density maps, but

Table 1. *Fractional coordinates and thermal parameters*

(*a*) Non-hydrogen atoms. All coordinates are $\times 10^5$.

Thermal parameters, U^{ij} (Å^2) $\times 10^4$ are as they appear in the Debye–Waller expression: $\exp(-2\pi^2\sum a_i^* a_j^* h_i h_j U^{ij})$; e.s.d.'s in the last significant figure are given in parentheses.

	x/a	y/b	z/c	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K	103174 (20)	25000	13447 (10)	334 (10)	476 (43)	301 (48)	−58 (44)	129 (37)	16 (43)
O(1)	44714 (68)	30830 (33)	37960 (42)	253 (13)	446 (13)	516 (20)	−51 (13)	121 (13)	−141 (16)
O(1′)	73167 (84)	14611 (59)	−6572 (44)	389 (20)	980 (36)	361 (20)	−171 (24)	23 (15)	175 (24)
O(2)	66802 (79)	46646 (26)	44725 (46)	406 (18)	237 (11)	596 (24)	31 (13)	190 (17)	−56 (14)
O(2′)	40453 (105)	20147 (60)	−25258 (50)	552 (26)	882 (34)	454 (24)	−115 (28)	12 (20)	187 (25)
C(1)	65128 (91)	35882 (46)	42833 (46)	302 (21)	300 (16)	274 (21)	−50 (15)	131 (16)	−10 (15)
C(1′)	50360 (108)	14114 (54)	−14357 (52)	407 (25)	549 (23)	302 (21)	−49 (28)	111 (18)	13 (21)
C(2)	91998 (86)	30558 (40)	47468 (49)	232 (18)	360 (19)	278 (21)	−48 (16)	58 (15)	−25 (17)
C(2′)	31068 (137)	6216 (84)	−12514 (61)	495 (32)	477 (54)	299 (26)	−310 (36)	105 (12)	−124 (32)
C(3)	92135 (88)	18866 (39)	42343 (44)	229 (19)	397 (18)	277 (20)	−42 (16)	96 (15)	−27 (16)
C(3′)	40387 (138)	1299 (59)	1907 (63)	562 (33)	660 (32)	400 (29)	−422 (31)	248 (25)	−248 (26)
O(3)	75465 (80)	8890 (35)	57136 (38)	474 (20)	460 (17)	304 (18)	−128 (16)	176 (14)	−2 (15)
O(3′)	73609 (102)	−11647 (50)	1358 (70)	1797 (80)	468 (21)	805 (40)	45 (36)	791 (49)	−247 (28)
C(4)	75648 (93)	10348 (37)	46041 (47)	334 (22)	315 (16)	253 (20)	−14 (16)	139 (16)	6 (15)
C(4′)	66329 (167)	−4307 (49)	6997 (68)	938 (51)	389 (22)	470 (32)	14 (30)	445 (35)	−60 (25)
O(4)	62082 (73)	4511 (25)	35210 (32)	493 (18)	290 (10)	283 (15)	−120 (13)	184 (13)	−60 (12)
O(4′)	81241 (90)	−672 (31)	19080 (42)	599 (25)	419 (14)	422 (19)	177 (17)	223 (17)	−65 (10)
O(5)	82561 (73)	18377 (26)	27888 (32)	427 (17)	289 (11)	261 (16)	−61 (12)	161 (13)	−6 (11)
O(5′)	43023 (69)	10135 (34)	11486 (34)	353 (17)	553 (18)	266 (15)	71 (16)	95 (12)	−63 (14)
B	66975 (108)	8589 (49)	23022 (52)	323 (23)	445 (22)	231 (23)	37 (20)	138 (17)	−30 (19)
O(W)	71616 (123)	35544 (60)	73427 (64)	576 (50)	737 (29)	708 (36)	115 (29)	240 (29)	199 (29)

(*b*) Hydrogen atoms

All coordinates are $\times 10^4$ and U_{iso}'s are $\times 10^3$.

	x/a	y/b	z/c	U_{iso}
H(C4,1)	1951 (113)	36359 (51)	45260 (60)	23 (6)
H(C4,2)	99490 (161)	31116 (73)	57542 (92)	54 (7)
H(C2′,1)	11420 (154)	9794 (70)	−15204 (78)	52 (8)
H(C2′,2)	30663 (171)	1634 (83)	−17834 (80)	27 (8)
H(O2′)	54801 (123)	25252 (47)	−26421 (61)	60 (10)
H(O3′)	29009 (103)	−2396 (42)	1966 (50)	12 (9)
H(O2)	49519 (129)	53004 (80)	41071 (60)	15 (8)
H(C3)	8192 (114)	16378 (42)	47014 (55)	32 (10)
H(W,1)	89786 (129)	37782 (67)	79069 (81)	13 (8)
H(W,2)	56607 (152)	40143 (55)	69086 (66)	46 (10)

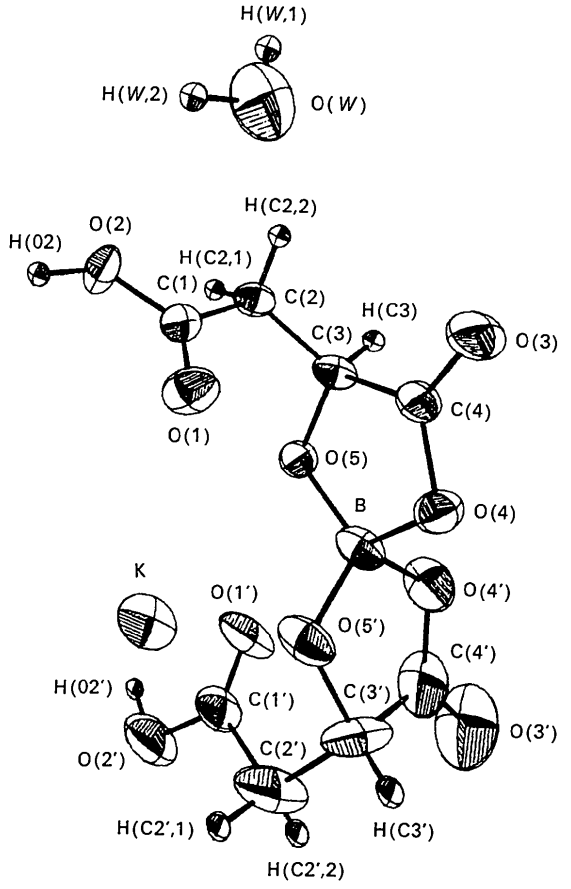

Fig. 1. Perspective drawing of a boromalate group and a water molecule. The thermal vibration ellipsoids are shown on a 50% probability scale.

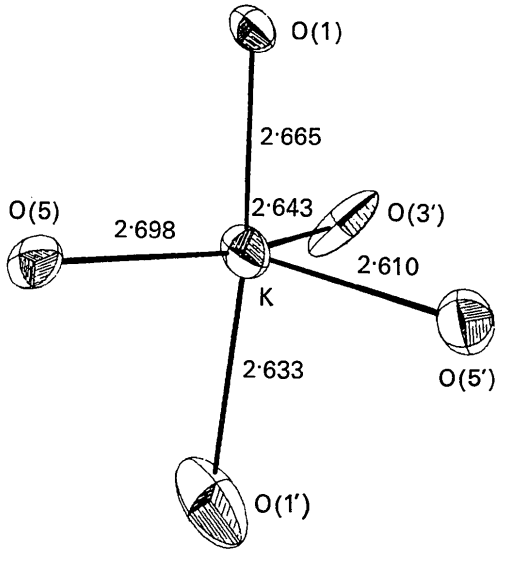

Fig. 2. The configuration of oxygen atoms around a potassium ion. Interatomic distances are given in Å.

we were unable to expand the partial structures mainly because two enantiomorphous images always appeared in the maps. The symbolic addition method was also employed. Two different programs were used: *SYMBAD* (Danielsen, 1971), and *MULTAN* (Germain, Main & Woolfson, 1971). Several attempts to find a suitable reflexion for fixing the enantiomorph failed. All electron density maps contained two enantiomorphous images. Using the combined evidence from several attempts and chemical considerations about bond-lengths and stereochemistry a partial structure was finally selected, which led to an iterative structure solution based on structure-factor calculations and electron density maps. When 10 out of 21 light atoms plus the potassium atom were used as a partial structure, the electron density map still showed two enantiomorphous images of roughly equal peak heights. When all atoms except hydrogen atoms had been located, refinement continued using full-matrix least-squares methods. Hydrogen atoms were located from a difference map using reflexions with $\sin\lambda/\theta < 0{\cdot}35$ Å^{-1}. Refinement was continued using isotropic temperature factor parameters for the hydrogen atoms and assuming anisotropic thermal movements for all other atoms. An isotropic extinction parameter was also refined (Zachariasen, 1967). The largest correction was about 4% of F and the computed extinction factor was $6{\cdot}1\times10^{-6}$. The weighting scheme employed was: $w=1/\mu_F^2$ where $\mu_F=[\sigma(F^2\text{ count})+k|F|^2]^{1/2}-|F|$. The parameter k was varied so that $\langle w|F_{obs}^2-F_{calc}^2|\rangle$ was independent of the magnitude of F_{obs}. The value of k for the last cycle was 1·04. In the last cycle of refinement the maximum coordinate shift for non-hydrogen atoms was $0{\cdot}15\sigma$ and for hydrogen atoms $0{\cdot}5\sigma$. The standard deviation of an observation of unit weight was 0·98. The conventional R value was 0·056.

Scattering factors were taken from *International Tables for X-ray Crystallography* (1962) for the heavy atoms, and for hydrogen atoms the contracted hydrogen atom scattering factors reported by Stewart, Davidson & Simpson (1965) were used.

The scattering factor of potassium was corrected for the real component of anomalous dispersion ($\Delta f'=+0{\cdot}2$). A table of observed and calculated structure factors is available.*

Discussion of the structure

The identification of the atoms and the illustration of the 50% probability thermal ellipsoids are shown in Fig. 1. The fractional atomic coordinates, the atomic thermal parameters (anisotropic for the heavy atoms, isotropic for the hydrogen atoms) and the estimated standard deviations of each are given in Table 1.

* This table has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30060. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Corresponding malate group atoms are labelled with the same number.

Table 1 shows that significant differences apparently exist between the thermal movements of corresponding atoms in the two malate groups. Abnormally large differences between some equivalent bond lengths were also observed, as shown in Table 2. No chemical or structural explanations appeared to explain the differences satisfactorily. To test validity of the results a constrained refinement was carried out using a method and program described by Pawley (1971). The two malate groups were constrained to be of identical geometry but all atoms were allowed independent thermal movements. After four cycles of refinement convergence was reached at an R value of 0·087. No detailed statistics is necessary to prove that the extra parameters allowed in the conventional least-squares refinement carry a high statistical significance. As the final parameters are probably of little general interest, only the bond distances are reported in Table 2. The thermal parameters showed the same trend as in the conventional least-squares refinement. One malate group, the same in both types of refinement, apparently has much larger thermal movements than the other. We are at a loss to explain this difference in thermal movements. The R value based on one malate group alone (plus the potassium atom) without introducing thermal parameters is 0·303 for the group with the smaller thermal movements and 0·378 for the other group. Although the latter group on average contributes less to the intensities than the former, this difference hardly points to any explanation. From the chemical point of view the structure appears plausible. One hydroxyl- and one carboxyl-oxygen atom are bound to boron from each malate group. The boron–hydroxyl-oxygen distance, 1·43 Å, is significantly shorter than the boron–carboxyl-oxygen distance, 1·52 Å. The boron atom enters into two five-membered rings which are each nearly planar. The dihedral angle between the two planes is 85·9°. Details about the two planes are given in Table 3.

Table 2. *Bond lengths obtained by the constrained refinements are compared with those obtained by a conventional refinement*

	Conventional refinement		Constrained refinement	
Bonding atoms	Bond length, l (Å)	Average, l (Å)	Bond length, l (Å)	
C(1)–O(1)	1·203	1·208	1·213	
C(1′)–O(1′)	1·213			
C(1)–O(2)	1·311	1·305	1·311	
C(1′)–O(2′)	1·299			
C(1)–C(2)	1·511	1·505	1·486	
C(1′)–C(2′)	1·499			
C(2)–C(3)	1·507	1·523	1·527	
C(2′)–C(3′)	1·539			
C(3)–C(4)	1·542	1·513	1·511	
C(3′)–C(4′)	1·484			
C(3)–O(5)	1·430	1·436	1·425	
C(3′)–O(5′)	1·443			
C(4)–O(3)	1·208	1·216	1·210	
C(4′)–O(3′)	1·224			
C(4)–O(4)	1·311	1·310	1·318	
C(4′)–O(4′)	1·308			
				Average l
B–O(4)	1·519	1·518	1·511	1·508
B–O(4′)	1·517		1·506	
B–O(5)	1·429	1·428	1·462	1·438
B–O(5′)	1·428		1·415	

Table 3. *The planarity of the two five-membered rings containing the boron atom*

The planes are described by equations: $Ax+By+Cz=D$. The coefficients are determined by the least-squares methods.

Ring 1	Distance from plane (Å)
C(3′)	−0·06
C(4′)	0·04
O(4′)	−0·02
O(5′)	0·06
B	−0·04

$A=3·4282$, $B=7·8413$, $C=-6·9186$, $D=1·4141$

Ring 2	Distance from plane (Å)
C(3)	0·07
C(4)	−0·03
O(4)	−0·01
O(5)	−0·08
B	0·06

$A=4·2107$, $B=7·4984$, $C=-1·4304$, $D=1·7857$

The dihedral angle between the two planes is 85·9°.

The coordination around the boron atom is described in more detail in Table 4. The environment of the boron atom is approximately tetrahedral. The

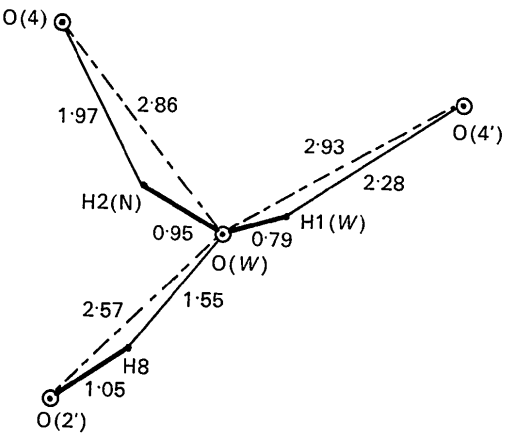

Fig. 3. The geometry of the hydrogen-bonding system formed by the water molecule viewed down the c axis. Interatomic distances are given in Å.

deviations of the BO_4 tetrahedron from $\bar{4}$ symmetry are small but significant. The boron–oxygen distances fall within the range observed in a number of borates. This is in accordance with the chemical observations that the boromalate complex is a stable species at least in neutral and in alkaline environments.

Table 4. *Coordination around the boron atom*

Bond lengths (Å)	
B–O(4) } B–O(4′) }	1·518 (3)
B–O(5) } B–O(5′) }	1·429 (3)
Bond angles (°)	
O(5′)–B–O(5)	115·7 (4)
O(5)–B–O(4′)	113·3 (3)
O(4′)–B–O(4)	107·2 (4)
O(4)–B–O(5′)	111·7 (4)
O(4′)–B–O(5′)	105·2 (3)
O(5)–B–O(4)	103·7 (3)

The potassium ion is five-coordinated with potassium–oxygen distances in the range 2·61–2·70 Å in accordance with the predominantly ionic character of the compound. Details about bond lengths involving potassium are given in Table 5. The coordination polyhedron is shown in Fig. 2.

Table 5. *Coordination around the potassium ion*

Bond lengths (Å)	
K–O(1)	2·665 (3)
K–O(1′)	2·633 (4)
K–O(3′)	2·643 (3)
K–O(5)	2·698 (4)
K–O(5′)	2·610 (3)

The boromalate ions are joined by electrostatic forces to the potassium ion and are also tied together by hydrogen bonds. The water molecule forms three hydrogen bonds to non-equivalent oxygen atoms of three different boromalate ions. The surroundings of the water molecule are depicted in Fig. 3, where relevant bond distances are also shown. As water is a very weak acid, the two protons of the water molecule do not approach the fairly neutral O(4) and O(4′) atoms very closely and these two hydrogen bonds are weak as in most similar cases. The basic character of the water molecule is more pronounced and the fairly acidic proton, H(8), from a carboxyl group can therefore approach the water molecule fairly closely as indicated in Fig. 3. The contents of a unit cell are shown in Fig. 4.

The other carboxyl group also forms a hydrogen bond through its proton H(7), which forms a hydroxyl group with O(2). This proton is linked with the oxygen

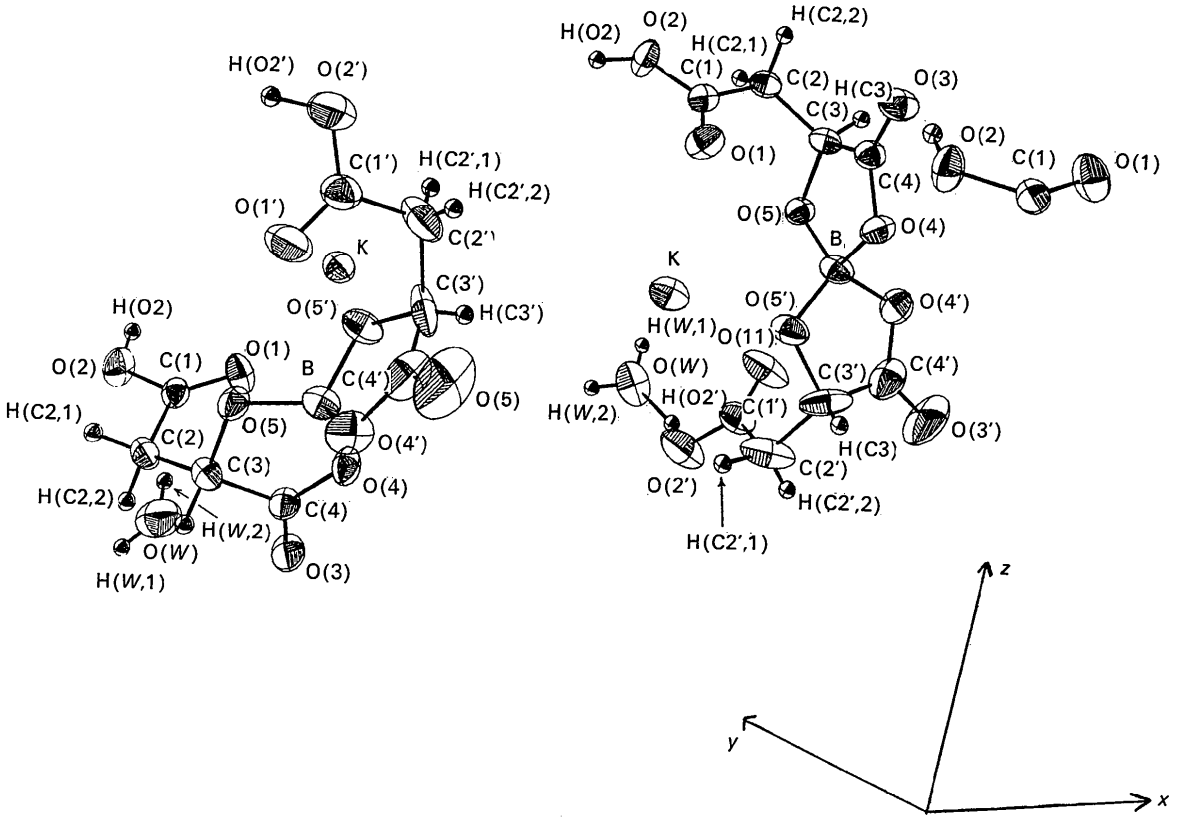

Fig. 4. The contents of a unit cell. The directions of the axes are indicated.

atom O(3) of an adjacent ion, the distance O(3)–H(7) being 1·63 Å. The corresponding O(3)–O(2) distance is 2·706 Å. The oxygen atoms which are not involved in hydrogen bonding form bonds with the potassium ion.

Computation

All calculations were carried out on a CDC6400 computer at the Computing Center of Aarhus University. The following programs were used:

Data reduction: Program *G*4 (Grønbæk Hazell, 1964).
Direct methods: *SYMBAD* (Danielsen, 1971) and *MULTAN* (Germain, Main & Woolfson, 1971).
Fourier synthesis: *FORDAP* (A. Zalkin, modified by Lundgren and Liminga).
Least-squares refinement: *LINUS*, modification of *ORFLS* (Busing, Martin & Levy, 1962) by W. C. Hamilton, J. A. Ibers and J. Edmonds; constrained refinement: Pawley (1971).
Ellipsoid plot program: *ORTEP* (Johnson, 1965).
Distance and angle program: *ORFFE* (Busing, Martin & Levy, 1964).

The last three programs were used from the SUNY Buffalo crystallographic computer programs, a system developed under the guidance of P. Coppens.

The authors wish to thank the Danish International Development Agency for financial aid, and the Facultad de Quimica de Montevideo for leave of absence to Raul A. Mariezcurrena. We are much indebted to Dr J. Danielsen for use of his program *SYMBAD* and for advice on direct methods. Mrs R. Grønbæk Hazell is thanked for helpful advice on computing and for helpful discussions.

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Etude Structurale de Dérivés du Fer Carbonyle. I. Structure Cristalline et Moléculaire de l'*o*-Aminothiophénol-difer Hexacarbonyle, $(C_6H_4SNH)Fe_2(CO)_6$

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(*Reçu le* 7 *décembre* 1972, *accepté le* 8 *janvier* 1973)

The crystal and molecular structure of *o*-aminothiophenol-bis (tricarbonyliron), $(C_6H_4SNH)Fe_2(CO)_6$, has been determined by a single-crystal X-ray study. The compound crystallizes with four molecules in a unit cell of symmetry $P2_1/c$ with dimensions $a = 8{\cdot}982$ (3), $b = 14{\cdot}443$ (5), $c = 11{\cdot}597$ (5) Å. $\beta = 94{\cdot}39$ (5)°. The intensities of the reflexions were measured on a Nonius CAD-3 automatic diffractometer. The structure has been refined to R and R'' values of 0·035 and 0·045 respectively, for 1058 independent reflexions. The molecule contains two iron tricarbonyl groups bridged through the sulphur and the nitrogen atoms of the organic ligand, with an iron–iron bond of length 2·411 (1) Å. The mean Fe–S and Fe–N distances are 2·283 (2) and 2·002 (4) Å, respectively. A comparison is made with iron carbonyl derivatives in which the iron atoms are doubly bridged through two sulphur or two nitrogen atoms.

Introduction

Alors que de nombreux travaux ont été effectués ces dernières années sur des dérivés du fer carbonyle comportant un squelette Fe_2X_2 (X = S ou N), c'est-à-dire un double pont symétrique entre les deux atomes métalliques, il n'existe pas à notre connaissance d'étude structurale de dérivés présentant un squelette Fe_2XX'