[for C(1′)–C(1), C(2″)–C(2) and C(3″)–C(3)] and 1·545 Å [for C(2′)–C(2), C(3′)–C(3) and C(1″)–C(1)]. Other bond lengths and angles are comparable to the values found in the α modification.

The planes formed by C(3), C(7), C(10) and by C(4), C(6), C(11) are inclined to the plane of the oxygens by 1·02 and 0·73°, respectively. The angle between the plane of the phenyl group and that of the oxygen atoms is 99·6°. The phenyl group is planar within experimental error. The equations of the planes are listed in Table 6.

The average C–C bond length in the phenyl ring is 1·393 Å, which agrees with that in crystalline benzene (Cox, Cruickshank & Smith, 1958). An unusual C–C–C angle of 116·1° is found at C(12), as in the α form (115·9°).

The α and β structures are shown in Fig. 2 viewed down the N–Si axis. In the title compound each carbon atom α to the nitrogen [namely C(4), C(6) and C(11)] is bent out of the plane formed by the N, Si and the proper O and C atoms, in accordance with the rule assumed by Boer & Turley (1969) for similar structures containing short (2·1–2·2 Å) dative Si ← N bonds.

We thank Dr K. Sasvári and Dr A. Kálmán for stimulating discussions and Mr Cs. Kertész for help with the data collection.

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# The Crystal Structure of the Complex Dibromobis-[5-(2-hydroxyethyl)-4-methylthiazole]cobalt(II)

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The crystal structure of the title compound has been solved by direct methods and refined by full-matrix least-squares calculations to $R=0{\cdot}061$ for 1696 independent reflexions. The monoclinic unit cell, space group $P2_1/c$, with $a=16{\cdot}993$ (8), $b=7{\cdot}794$ (5), $c=14{\cdot}807$ (7) Å, $\beta=112{\cdot}1$ (2)°, contains four molecules. The cobalt atom is tetrahedrally bound to two bromine atoms and the nitrogen atoms of the substituted thiazoles. The overall molecular symmetry is approximately twofold and the hydroxyethyl side chains have similar atomic environments. All but one of the hydrogen atoms have been located in difference syntheses. Short S···O and Br···O contacts are observed, the latter indicating O–H···Br hydrogen bonding.

## Introduction

The reaction between transition metal halides and organic molecules containing the thiazole entity produces both ionic structures and organometallic complexes. Compounds of the former class have recently been studied by X-ray diffraction. Among these are 3-(2-diethylammoniumethoxy)-1,2-benzisothiazole tetrachlorocobaltate (Bonamartini, Nardelli & Palmieri, 1972), the corresponding Cu compound (Bonamartini, Nardelli, Palmieri & Pelizzi, 1971) and more recently, (protonated thiamine)$^{2+}$(CuCl$_4$)$^{2-}$ (Caira, Fazakerley, Linder & Nassimbeni, 1973). In the structure reported here, there is direct bonding between the metal atom and the thiazole ligand. Much enzymic activity depends on transition-metal–thiazole interaction and it is therefore of interest to establish the binding site in this heterocyclic system. In addition the ligand 5-(2-hydroxyethyl)-4-methylthiazole, containing S,N and O atoms as possible donors, is of importance, being the thiazole fraction of the vitamin $B_1$ molecule.

## Experimental

Several copper(II) and cobalt(II) complexes of thiamine chloride hydrochloride and some related thiazoles have recently been prepared at this University (Fazakerley & Russell, 1974) and crystals of the title compound were obtained from this source. The dark blue crystals were shown to have the composition Co-[5-(2-hydroxyethyl)-4-methylthiazole]$_2$Br$_2$ by microanalysis. Preliminary X-ray data were obtained by rotation and Weissenberg methods with Ni-filtered Cu $K\alpha$ radiation. Systematic absences $h0l$, $l=2n+1$, and $0k0$, $k=2n+1$, indicated the space group $P2_1/c$. With a single crystal cut to a cube of dimension 0·4 mm, the cell parameters were obtained from a least-squares analysis of the settings of 25 reflexions measured on a four-circle diffractometer. The crystal data are listed in Table 1. The density was measured by flotation in a chlorobenzene–methyl iodide mixture.

Table 1. *Crystal data*

| | |
|---|---|
| Molecular formula | $CoC_{12}H_{18}N_2O_2S_2Br_2$ |
| M.W. | 504·9 |
| Space group | $P2_1/c$ |
| $a =$ 16·993 (8) Å | |
| $b =$ 7·794 (5) | $D_m = 1·84$ g cm$^{-3}$ |
| $c =$ 14·807 (7) | $D_c = 1·85$ g cm$^{-3}$ for $Z=4$ |
| $\beta =$ 112·1 (2)° | $\mu$(Mo $K\alpha$) = 58·6 cm$^{-1}$ |
| $V =$ 1817·18 Å$^3$ | $F(000) = 996$ |

Intensities were measured on a Philips PW 1100 computer-controlled four-circle diffractometer, operating in the $\omega$–$20$ scan mode (scan speed 0·04°$\theta$ s$^{-1}$, scan width 1·2°$\theta$). With graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0·7107$ Å), 2433 reflexions up to $2\theta = 44°$ were recorded. With the criterion $I_{\rm rel} > 1·65\sigma(I_{\rm rel})$ for an observed reflexion, 737 reflexions including systematic absences were omitted as unobserved, leaving 1696 independent reflexions which were used in the analysis. Three reference reflexions monitored every hour remained constant to within ±3%. The value of $\mu R$ for the crystal was 1·2 but no corrections for absorption were made.

Table 2. *Fractional atomic coordinates and thermal parameters and their e.s.d.'s for the complex*

(*a*) Heavy atoms. Coordinates are × 10$^4$. Thermal parameters are of the form

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*) \times 10^3\right].$$

| | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Co | 2458 (1) | 6371 (2) | 668 (1) | 37 (1) | 27 (1) | 32 (1) | 4 (1) | 13 (1) | 0 (1) |
| Br(1) | 1655 (1) | 8217 (2) | 1284 (1) | 62 (1) | 49 (1) | 64 (1) | 24 (1) | 35 (1) | 3 (1) |
| Br(2) | 3383 (1) | 7996 (2) | 115 (1) | 52 (1) | 41 (1) | 64 (1) | −6 (1) | 28 (1) | 6 (1) |
| N(3) | 3162 (7) | 4827 (14) | 1798 (7) | 27 (7) | 30 (7) | 28 (7) | −5 (5) | 5 (5) | −6 (5) |
| C(2) | 3275 (9) | 5184 (18) | 2692 (10) | 40 (9) | 34 (8) | 41 (9) | 4 (7) | 13 (7) | 4 (7) |
| S(1) | 3893 (3) | 3785 (5) | 3527 (3) | 54 (3) | 54 (3) | 32 (2) | 17 (2) | 15 (2) | 8 (2) |
| C(4) | 3593 (8) | 3344 (17) | 1741 (9) | 26 (8) | 23 (7) | 37 (8) | −1 (6) | 9 (6) | 0 (6) |
| C(5) | 4026 (9) | 2635 (18) | 2604 (10) | 39 (9) | 29 (8) | 49 (9) | 7 (7) | 22 (8) | 10 (7) |
| C(6) | 3566 (11) | 2784 (19) | 791 (11) | 81 (12) | 29 (8) | 45 (9) | 9 (8) | 26 (9) | −7 (7) |
| C(7) | 4546 (9) | 995 (21) | 2809 (11) | 33 (9) | 50 (10) | 58 (10) | 4 (8) | 15 (7) | 6 (8) |
| C(8) | 4175 (12) | −428 (23) | 3141 (17) | 60 (13) | 46 (11) | 125 (17) | 13 (9) | 57 (12) | 19 (12) |
| O(9) | 4183 (15) | −47 (23) | 4072 (17) | 262 (26) | 91 (13) | 231 (24) | 56 (15) | 196 (22) | 70 (15) |
| N(31) | 1759 (7) | 4799 (14) | −439 (7) | 39 (8) | 38 (7) | 21 (6) | −1 (6) | 11 (5) | −0 (5) |
| C(21) | 1874 (9) | 4780 (18) | −1260 (10) | 31 (9) | 36 (8) | 38 (9) | −4 (7) | 7 (7) | 1 (7) |
| S(11) | 1441 (2) | 3102 (5) | −1990 (3) | 46 (2) | 52 (2) | 31 (2) | −7 (2) | 17 (2) | −5 (2) |
| C(41) | 1287 (8) | 3386 (18) | −368 (10) | 32 (8) | 32 (8) | 40 (8) | 4 (7) | 17 (7) | 6 (7) |
| C(51) | 1065 (8) | 2361 (19) | −1145 (10) | 27 (8) | 47 (9) | 34 (8) | 7 (7) | 10 (6) | −4 (7) |
| C(61) | 1080 (12) | 3210 (22) | 497 (12) | 89 (14) | 54 (11) | 63 (11) | −22 (10) | 47 (10) | −10 (9) |
| C(71) | 553 (10) | 686 (20) | −1288 (12) | 41 (10) | 43 (10) | 42 (9) | −7 (8) | 12 (8) | −7 (8) |
| C(81) | 876 (11) | −770 (23) | −1722 (13) | 46 (12) | 45 (10) | 59 (12) | 1 (9) | 10 (8) | −0 (9) |
| O(91) | 766 (9) | −315 (18) | −2666 (9) | 127 (12) | 73 (9) | 69 (9) | −31 (9) | 57 (9) | −24 (7) |

Table 2 (*cont.*)

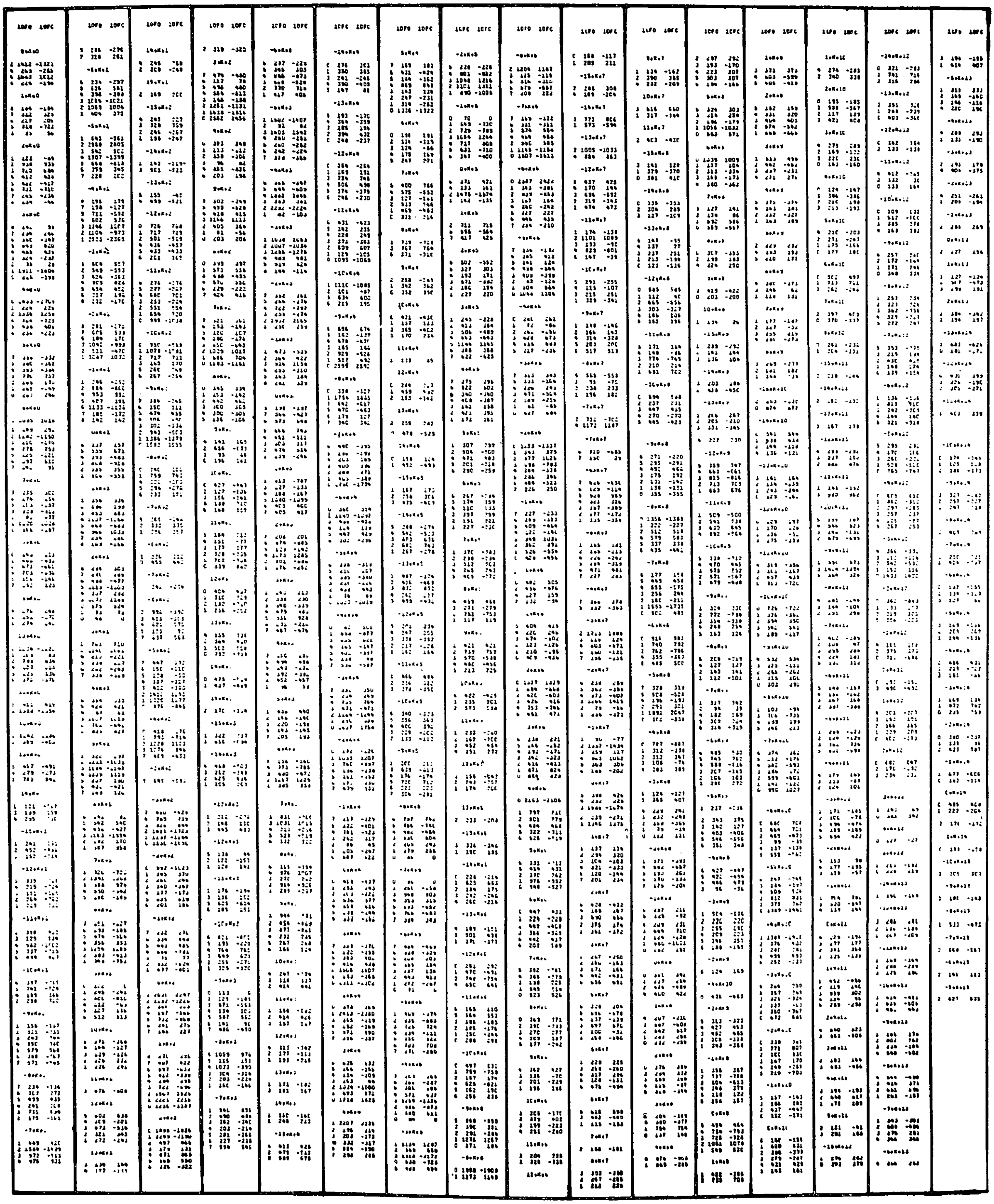
(*b*) Fractional atomic coordinates ( × 10$^3$) and their e.s.d.'s for the hydrogen atoms

| | $x$ | $y$ | $z$ |
|---|---|---|---|
| H(2) | 308 (8) | 634 (17) | 285 (9) |
| H(61) | 305 (9) | 211 (19) | 47 (10) |
| H(62) | 359 (9) | 327 (19) | 35 (10) |
| H(63) | 377 (9) | 193 (19) | 81 (10) |
| H(71) | 460 (7) | 70 (17) | 215 (9) |
| H(72) | 489 (8) | 113 (17) | 323 (9) |
| H(81) | 363 (9) | −66 (21) | 311 (11) |
| H(82) | 456 (9) | −209 (21) | 352 (11) |
| H(21) | 214 (7) | 605 (16) | −134 (8) |
| H(611) | 95 (9) | 397 (20) | 68 (10) |
| H(612) | 71 (9) | 258 (19) | 46 (10) |
| H(613) | 159 (9) | 293 (19) | 110 (10) |
| H(711) | 54 (8) | 25 (18) | −64 (10) |
| H(712) | −4 (9) | 98 (18) | −179 (10) |
| H(811) | 127 (10) | −76 (21) | −124 (11) |
| H(812) | 52 (8) | −183 (19) | −164 (9) |
| H(91) | 107 (10) | −123 (23) | −307 (12) |

## Solution and refinement of the structure

A Patterson map showed only one broad peak on the Harker section $0,\frac{1}{2}-2y,\frac{1}{2}$ in spite of the presence of four heavy atoms in general positions. This implied near equality of the $y$ coordinates of two or more of the heavy atoms. In addition there were nine large peaks, some of them multiple, in the section $2x, \frac{1}{2}, \frac{1}{2}+2z$ which led to several possible solutions for the heavy-atom coordinates. Fourier maps based on some of these possibilities could not be interpreted. It was thus decided to use the Karle–Hauptman $\sum_2$ relationship to solve the structure. $E$ values for the reflexions were calculated from the normalized structure factors (X-

Table 3. *Observed and calculated structure factors*



RAY program system, 1972). 235 reflexions with $E > 1·5$ were phased from 50 generators. An $E$ map based on these reflexions revealed the heavy atoms. The initial $y$ coordinates of the cobalt and the two bromine atoms were 0·64, 0·81 and 0·80, which correlated with the results found in the Patterson map.

The positions and isotropic temperature factors of the cobalt and bromine atoms were given one cycle of refinement. With unit weights $R$ was 0·42. The light atoms were located in subsequent Fourier and difference syntheses. After 3 cycles of refinement $R$ was 0·11. On introducing anisotropic temperature factors for all atoms, 3 cycles of refinement brought $R$ to 0·067. All the hydrogens (except one of a hydroxylic oxygen) were located in a difference synthesis; they were assigned the isotropic temperature factors of the atoms to which they were bonded and their positions were refined. In the last cycle of refinement the average $\Delta/\sigma$ for the heavy atoms was 0·04 and 0·15 for the hydrogen atoms. The final $R$ was 0·061 and a difference synthesis with all atoms subtracted showed no prominent electron density. Scattering factors for Br, C, N, O and S were those of Cromer & Mann (1968); for H, those of Stewart, Davidson & Simpson (1965) and for Co, those of Hanson, Herman, Lea & Skillman (1964). Anomalous dispersion corrections were applied to Co and Br (*International Tables for X-ray Crystallography*, 1968). Table 2 lists the final atomic coordinates and thermal parameters. Observed and calculated structure factors are in Table 3.

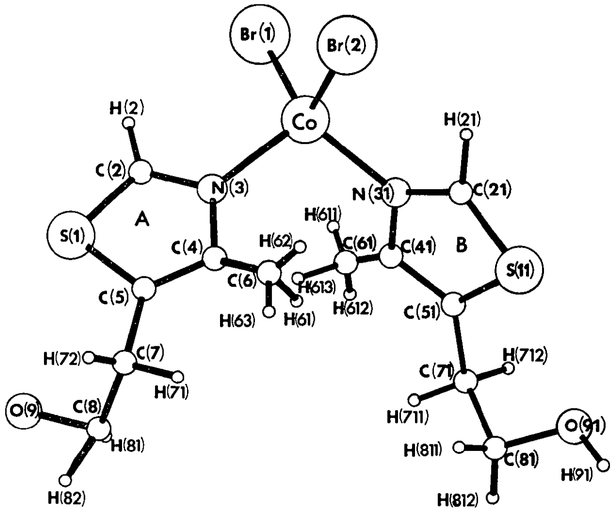


Fig. 1. The molecule viewed approximately parallel to **a*** showing atomic nomenclature and ring labelling.

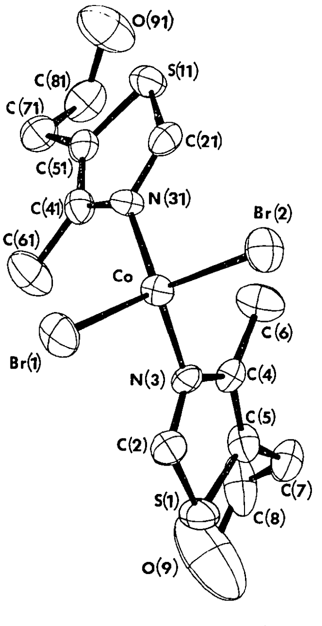


Fig. 2. The molecule viewed down **b** showing thermal ellipsoids of the non-hydrogen atoms at the 50% probability level.

### Description of the structure and discussion

Bond lengths involving heavy atoms and those involving hydrogens are listed in Tables 4 and 5 respectively. All calculations were performed with the X-RAY program system (1972). Atomic nomenclature which is standard in the case of one ligand, $A$, is shown in Fig. 1, a diagram of the molecule viewed approximately parallel to **a***. For ligand $B$, chemically equivalent atoms are similarly labelled but suffixed with the number 1. All hydrogen atoms which were located are labelled.

Table 4. *Bond lengths involving heavy atoms* (Å) *and their e.s.d.'s*

| | | | |
|---|---|---|---|
| Co——Br(1) | 2·388 (3) | C(4)—N(3) | 1·39 (2) |
| Co——Br(2) | 2·391 (3) | C(41)-N(31) | 1·39 (2) |
| Co——N(3) | 2·04 (1) | C(4)—C(6) | 1·46 (2) |
| Co——N(31) | 2·03 (1) | C(41)-C(6) | 1·46 (3) |
| N(3)—C(2) | 1·29 (2) | C(5)—C(7) | 1·52 (2) |
| N(31)-C(21) | 1·30 (2) | C(51)-C(71) | 1·54 (2) |
| C(2)—S(1) | 1·69 (1) | C(7)—C(8) | 1·45 (3) |
| C(21)-S(11) | 1·68 (1) | C(71)-C(81) | 1·51 (3) |
| S(1)—C(5) | 1·72 (2) | C(8)—O(9) | 1·41 (4) |
| S(11)-C(51) | 1·71 (2) | C(81)-O(91) | 1·38 (2) |
| C(5)—C(4) | 1·33 (2) | | |
| C(51)-C(41) | 1·33 (2) | | |

Table 5. *Bond lengths involving hydrogen atoms* (Å)

E.s.d.'s are approximately 0·1 Å.

| | | | |
|---|---|---|---|
| C(2)-H(2) | 1·0 | C(21)-H(21) | 1·1 |
| C(6)-H(61) | 1·0 | C(61)-H(611) | 0·7 |
| C(6)-H(62) | 0·8 | C(61)-H(612) | 0·8 |
| C(6)-H(63) | 0·7 | C(61)-H(613) | 1·0 |
| C(7)-H(71) | 1·0 | C(71)-H(711) | 1·0 |
| C(7)-H(72) | 0·7 | C(71)-H(712) | 1·0 |
| C(8)-H(81) | 1·0 | C(81)-H(811) | 0·8 |
| C(8)-H(82) | 1·1 | C(81)-H(812) | 1·1 |
| | | O(91)-H(91) | 1·2 |

As expected from the dark-blue colour and spectral studies of the complex (Fazakerley & Russell, 1974) the cobalt atom has a tetrahedral environment. The bromine atoms are at an average distance of 2·39 Å from the cobalt atom while the nitrogen atoms of the substituted thiazole ligands are bound to the metal at an average distance of 2·04 Å. It is evident from Table 6, which lists the bond angles, that the tetrahedral geometry about the metal atom is not significantly distorted, the greatest deviation from the ideal being that of angle Br(1)–Co–N(31), 115·2(4)°. Although the atomic positions for the asymmetric unit are general, the overall molecular symmetry is approximately twofold, the rotation axis being the bisector of the Br(1)–Co–Br(2) angle. This is evident from Fig. 2 (*ORTEP*: Johnson, 1965) which views the molecule down **b**, which is almost parallel to the bisector. This pseudosymmetry is displayed with respect to both the ring orientations and the configurations of the ethylhydroxy side chains. Equivalent bond lengths and angles in the ligands agree closely and are within the expected ranges. Comparing the present structure with the thiamine hydrochloride–copper(II) complex (Caira *et al.*, 1973), in which the thiazole N atom also has a coordination number of three, there are no significant differences in the bond angles and bond lengths in the environment of the N atom. Least-squares planes were calculated (Table 7) for the ring atoms of the substituted thiazole ligands *A* and *B*, both of which are planar, the r.m.s. deviations of the atoms from the respective planes being 0·006 and 0·003 Å.

Table 6. *Intramolecular bond angles* (°) *and their e.s.d.'s*

| | | | |
|---|---|---|---|
| Br(1)–Co–Br(2) | 110·8 (1) | S(1)–C(5)–C(4) | 111 (1) |
| Br(1)–Co–N(3) | 106·6 (4) | S(11)–C(51)–C(41) | 112 (1) |
| Br(1)–Co–N(31) | 115·2 (4) | C(5)–C(4)–N(3) | 114 (1) |
| Br(2)–Co–N(3) | 109·6 (3) | C(51)–C(41)–N(31) | 113 (1) |
| Br(2)–Co–N(31) | 107·6 (4) | N(3)–C(4)–C(6) | 119 (1) |
| N(3)–Co–N(31) | 106·9 (4) | N(31)–C(41)–C(61) | 119 (1) |
| Co–N(3)–C(4) | 126·9 (9) | C(5)–C(4)–C(6) | 127 (1) |
| Co–N(31)–C(41) | 126·5 (9) | C(51)–C(41)–C(61) | 128 (1) |
| Co–N(3)–C(2) | 121·8 (9) | C(4)–C(5)–C(7) | 128 (1) |
| Co–N(31)–C(21) | 120·9 (9) | C(41)–C(51)–C(71) | 126 (1) |
| C(2)–N(3)–C(4) | 111 (1) | S(1)–C(5)–C(7) | 121 (1) |
| C(21)–N(31)–C(41) | 111 (1) | S(11)–C(51)–C(71) | 122 (1) |
| N(3)–C(2)–S(1) | 115 (1) | C(5)–C(7)–C(8) | 115 (2) |
| N(31)–C(21)–S(11) | 115 (1) | C(51)–C(71)–C(81) | 115 (2) |
| C(2)–S(1)–C(5) | 89·2 (7) | C(7)–C(8)–O(9) | 109 (2) |
| C(21)–S(11)–C(51) | 89·1 (8) | C(71)–C(81)–O(91) | 108 (1) |

Table 7. *Least-squares planes*

The equations of the planes are expressed in orthogonalized space as $PI+QJ+RK=S$.

Plane I: through atoms N(3), C(2), S(1), C(5), C(4) of thiazole ring *A*.
Equation: $0{\cdot}83166I+0{\cdot}54836J+0{\cdot}08743K=5{\cdot}91525$

| Atoms included in the calculation | Distance from plane | Atoms not included in calculation | Distance from plane |
|---|---|---|---|
| N(3) | −0·002 Å | Co | 0·053 Å |
| C(2) | 0·006 | H(2) | 0·113 |
| S(1) | −0·006 | | |
| C(5) | −0·007 | C(7) | −0·030 |
| C(4) | 0·005 | C(6) | 0·043 |

Plane II: through atoms N(31), C(21), S(11), C(51), C(41) of thiazole ring *B*.
Equation: $0{\cdot}73313I-0{\cdot}52282J+0{\cdot}43494K=0{\cdot}15164$

| Atoms included in the calculation | Distance from plane | Atoms not included in calculation | Distance from plane |
|---|---|---|---|
| N(31) | 0·001 Å | Co | 0·44 Å |
| C(21) | −0·003 | H(21) | −0·20 |
| S(11) | 0·004 | | |
| C(51) | −0·004 | C(71) | 0·014 |
| C(41) | 0·003 | C(61) | −0·021 |

Table 8. *Short intramolecular and intermolecular approaches* (Å)

Symmetry code: i $x$ $\frac{1}{2}-y$ $\frac{1}{2}+z$
ii $x$ $\frac{1}{2}-y$ $-\frac{1}{2}+z$

| | | | |
|---|---|---|---|
| S(1)···O(9) | 3·08 (2) | O(91)···Br($1^{ii}$) | 3·40 (2) |
| S(11)···O(91) | 2·92 (1) | C(2)···Br(1) | 3·63 (1) |
| O(9)···Br($2^{i}$) | 3·33 (3) | C(21)···Br(2) | 3·62 (1) |

Interatomic distances less than the van der Waals sum have been found for certain atoms (Table 8) and these are of interest. On the one hand, there are short intramolecular S···O distances between S(1) and O(9), 3·08(2), and between S(11) and O(91), 2·92, the van der Waals sum being 3·25 Å. These arise from the near *cis*-configurations of the side chains. For ligand *A*, the torsion angle defined by S(1)–C(5)–C(7)–C(8) is 68° and by C(5)–C(7)–C(8)–O(9) is also 68°, the directions of torsion being such as to bring S(1), C(5), C(8) and O(9) into near coplanarity, with C(7) 0·67 Å out of the plane. The corresponding torsion angles in ligand *B* are 41 and 66°, resulting in a similar *cis*-configuration with S(11), C(51), C(81) and O(91) roughly coplanar and C(71) 0·55 Å out of the plane. The geometry of both side chains is shown in Fig. 3, in which the figures to the left of each atom are their normal distances in Å from the least-squares planes through S(1), C(5), C(8), O(9) and S(11), C(51), C(81), O(91) respectively. An analogous short S···O intramolecular contact has been observed in the structure of 2,7-dimethylthiachromine-8-ethanol dihydrate (Pletcher, Sax, Yoo, Chu & Power, 1974). The other short contacts observed in the cobalt complex are intermolecular and involve O and Br atoms (Fig. 3). In one case, O(9) and Br($2^{i}$) (Table 8) are separated by 3·33 Å (van der Waals sum = 3·35 Å) while in the other, the O(91)···Br($1^{ii}$) distance is 3·40 Å. Although H(91) attached to O(91) was located in the analysis, the hydroxylic hydrogen of O(9) was not found. The latter failure can be attributed to the heavy-atom environment of O(9) and the fact that O(9) has the largest thermal vibration of all the atoms in the structure (Table 2). The short intermolecular H(91)···Br($1^{ii}$)

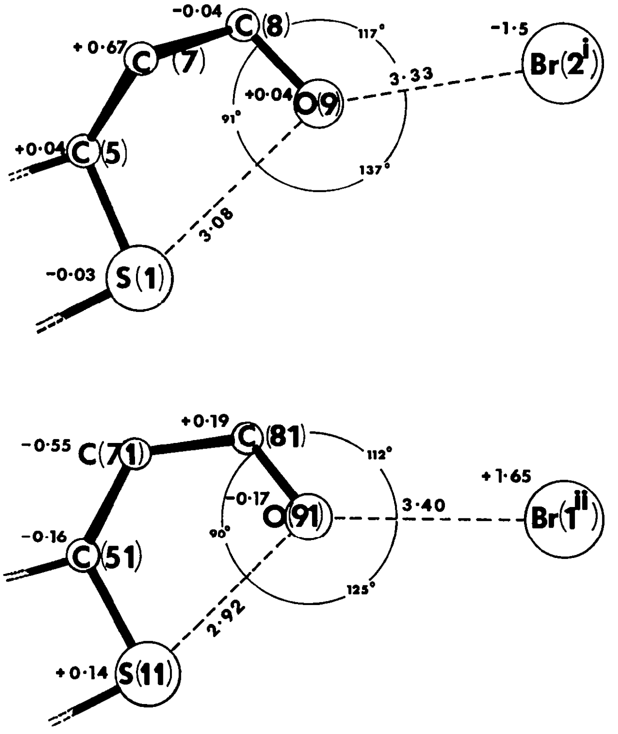


Fig. 3. The configurations of the hydroxyethyl side-chains showing short contacts (Å). Figures to the left of each atom are their normal distances (Å) from the least-squares planes through atoms S(1), C(5), C(8), O(9) (upper diagram) and through atoms S(11), C(51), C(81), O(91) (lower diagram).

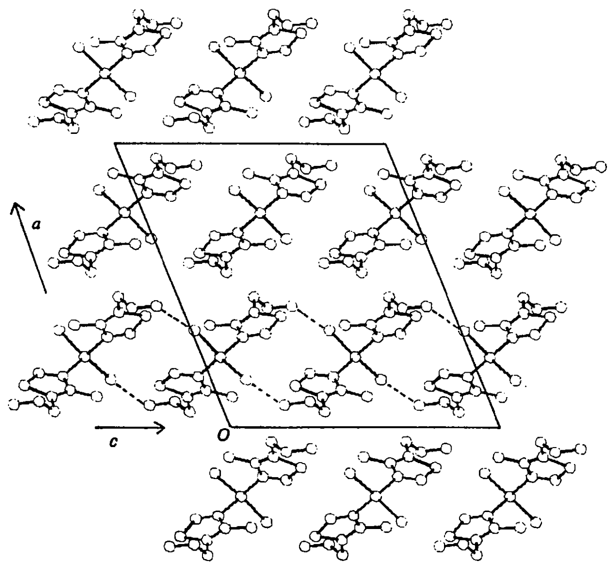


Fig. 4. The (010) projection of the structure. Short O···Br contacts for a row of molecules are represented by dashed lines.

distance of 2·2(2) Å and the O(91)–H(91)···Br($1^{ii}$) angle of 175(14)° indicate hydrogen bonding between these atoms. O(91) is thus involved in two interactions, an intramolecular S···O coulombic interaction and an O–H···Br hydrogen bond. The similar geometry involving O(9) and Br($2^{i}$) suggests analogous H···Br interaction. Further possible hydrogen-bonding sites are the ring methine groups, as observed in the structures of *N*-benzyl-4-methylthiazolium bromide (Power, Pletcher & Sax, 1970) involving C–H···$Br^-$ and in thiamine hydrochloride (Kraut & Reed, 1962) involving C–H···$Cl^-$ hydrogen bonds. In the cobalt complex, the distances H(2)···Br(1) and H(21)···Br(2) are 3·1(1) and 2·8(1) Å respectively, indicating similar interactions, although the respective C(2)···Br(1) and C(21)···Br(2) distances are 3·63(1) and 3·62(1) Å, which just exceed the sum of the C–H hydrogen-bond radius of 1·64 Å (Power *et al.*, 1970) and the acceptor radius of a bromine atom, 1·95 Å. Weak C–H···Br interactions are therefore suggested by these distances and by the favourable ring orientations which make the respective angles C(2)–H(2)···Br(1) and C(21)–H(21)···Br(2), 120(8) and 127(7)°.

In Fig. 4, the (010) projection of the structure, short Br···O distances are represented by dashed lines.

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