Für Arsen ist keine analoge trimere Struktur bekannt. Die Deformation kann am (AsF_4O_2)-Oktaeder aufgrund des Platzbedarfs der 6 Liganden nicht so gross wie am (SbF_4O_2)-Oktaeder sein. Das zeigt auch schon der Vergleich der Abstände. Für das $(As_2F_8O_2)^{2-}$-Anion wurden mittlere As–F-Abstände von 1,739(16) Å und mittlere As–O-Abstände von 1,806(17) Å gemessen (Haase, 1974*a*), für $(Sb_3F_{12}O_3)^{3-}$ 1,88(2) und 1,92(2) Å entsprechend Sb–F- und Sb–O-Abstände. Danach ist für Arsen die Ausbildung eines $(As_3F_{12}O_3)^{3-}$-Anions mit 6-Ring-Struktur gerade nicht mehr, für Antimon die Ausbildung eines $(Sb_3F_{12}O_3)^{3-}$-Anions aber gerade schon möglich.

Bemerkenswert ist, dass die Anionen $(As_2F_{10}O)^{2-}$ und $(Sb_2F_{10}O)^{2-}$ demgegenüber (nahezu) gleiche Gestalt besitzen. Es liegt die Annahme nahe, dass die Ausbildung von As–O–As- bzw. Sb–O–Sb-Brücken mit einem $\angle \sim 135°$ unter wahrscheinlicher Doppelbindungsbetätigung der Normalfall für die hier diskutierten Strukturen ist.

Die IR- und Raman-Spektren von $Cs_3(Sb_3F_{12}O_3)$, am Pulver gewonnen, wurden bereits mitgeteilt (Haase, 1972). Aufgrund der C_s-Symmetrie des Anions treten, im Gegensatz zum zentrosymmetrischen Anion $(As_2F_8O_2)^{2-}$, keine Alternativverbote auf.

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The Crystal Structure of {5-(2-Hydroxyethyl)-4-methylthiazolium} Tribromo-{5-(2-hydroxyethyl)-4-methyl-3-thiazolo}cuprate (II)

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Crystals of $[C_6H_{10}NOS]^+[CuBr_3[C_6H_9NOS)]^-$ are monoclinic, space group $P2_1$, with $a=8{\cdot}016$ (4), $b=17{\cdot}190$ (8), $c=7{\cdot}322$ (4) Å, $\beta=103{\cdot}34$ (6)° at 20°C; $D_c=2{\cdot}00$ g cm^{-3} for $Z=2$. The structure was solved by Patterson and Fourier methods and refined by least-squares calculations to an R of 0·041. Each asymmetric unit consists of a $[CuBr_3(C_6H_9NOS)]^-$ anion and a $[C_6H_{10}NOS]^+$ cation. Rows of anions are linked to rows of cations by hydrogen bonds.

Introduction

The importance of the thiazole group in biological systems has led to the preparation of a wide variety of metal complexes having the thiazole group as a ligand. Fazakerley & Russell (1974) have prepared a number of copper and cobalt complexes in which the metal atom is coordinated to thiamine and thiazole compounds. In particular they have reported the formation of the complex ($CuBr_3$ thiaz thiaz H) where thiaz= 5-(2-hydroxyethyl)-4-methylthiazole.

The infrared spectrum of this compound had a strong band at 238 cm^{-1} and a weak band at 285 cm^{-1} which were assigned to ν(Cu–Br) and ν(Cu–N) respectively. The reflectance spectrum gave evidence of a tetrahedrally coordinated copper atom. A magnetic moment of 1·89 B.M. indicated that there was no

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Cu–Cu interaction. It was postulated that this complex might have a distorted tetrahedral anion $[CuBr_3\ \text{thiaz}]^-$ with an uncoordinated thiazolium cation $[\text{thiaz H}]^+$. The fact that three halogen atoms and one thiazole group might be coordinated to the copper atom was considered unusual. In order to establish the coordination of the copper atom and to investigate the coordinating properties of the thiazole group, crystals of this compound were submitted to us for structure analysis.

Experimental

Dark red crystals of the compound were kindly provided by Miss J. C. Russell of the University of Cape Town. Weissenberg photographs indicated that the crystals were monoclinic with systematic absences $0k0$ for $k=2n+1$, consistent with either $P2_1$ or $P2_1/m$.

The cell parameters were determined at 20°C by a least-squares refinement of the angular settings of 25 high-order reflexions. The cell data for $Z=2$ are: $a=8·016$ (4), $b=17·190$ (8), $c=7·322$ (4) Å, $\beta=103·34$ (6)°, $\mu=78·32$ cm^{-1}, $D_c=2·00$ g cm^{-3}. The crystal density was measured as 2·01 g cm^{-3} by flotation in a mixture of CH_3I and CCl_4.

A 0·08 × 0·13 × 0·45 mm crystal was chosen for data collection. With graphite-monochromated Mo $K\alpha$ radiation 1236 non-equivalent reflexions were measured by the ω–2θ scan technique between $\theta=3$ and 22°. A Philips P.W. 1100 diffractometer of the National Physical Research Laboratory, C.S.I.R., Pretoria was employed. The scan width was 1·2°θ and the scan speed 0·04°θ s^{-1}. The intensities of three strong reflexions measured periodically throughout the data collection showed that the crystal was stable to X-rays. The background was counted for half the scanning time on both sides of each reflexion. 108 reflexions were considered to be unobserved according to the criterion $I<1·65\sigma(I)$, where $\sigma(I)=[(0·02N_o)^2+N_o+K^2N_b]^{1/2}$. N_o is the gross count, N_b the background count and K the ratio of scan to background times.

The intensities were corrected for Lorentz and polarization effects but not for absorption. Scattering factors for the non-hydrogen atoms were those of Cromer & Mann (1968) and for hydrogen those of Stewart, Davidson & Simpson (1965). Anomalous dispersion corrections for bromine and copper were obtained from *International Tables for X-ray Crystallography* (1968). Calculations used for solving and refining the structure were carried out with the programs of the X-RAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

Solution of the structure

The distribution of E values was acentric. The space group was therefore assigned as $P2_1$. This assumption was verified by the final structural results.

The structure was solved by the heavy atom method. The x and z coordinates of the four heavy atoms, namely the copper and three bromines, were obtained from the Harker section $(U,\frac{1}{2},W)$ of a Patterson map. One of these bromine atoms was arbitrarily given a value of $y=0·25$ and the y coordinates of the other three atoms were then deduced from the V coordinates of the cross-vector peaks. A Fourier synthesis based on the positions of these atoms yielded the coordinates of the remaining non-hydrogen atoms. In order to fix the origin the y coordinate of the copper atom was held

Table 1. *Positional and thermal parameters with e.s.d.'s in parentheses*

(a) Heavy atoms. Positional parameters are given as fractional coordinates (×10^4) and thermal parameters (×10^3) are given according to the expression $T=\exp[-2\pi^2(a^{*2}h^2U_{11}+b^{*2}k^2U_{22}+c^{*2}l^2U_{33}+2a^*b^*hkU_{12}+2a^*c^*hlU_{13}+2b^*c^*klU_{23})]$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}	Isotropic B(Å^2)
Cu	2849 (3)	6031 (0)	2058 (3)	23 (1)	21 (1)	27 (1)	1 (1)	2 (1)	−4 (1)	1·58
Br(1)	2202 (3)	7364 (2)	1225 (3)	54 (1)	24 (1)	53 (1)	8 (1)	−10 (1)	2 (1)	3·54
Br(2)	3915 (3)	5057 (2)	296 (3)	35 (1)	36 (1)	32 (1)	5 (1)	10 (1)	−10 (1)	2·67
Br(3)	209 (3)	5487 (2)	2317 (3)	28 (1)	46 (1)	50 (1)	−8 (1)	14 (1)	−14 (1)	3·14
S(1)	8057 (7)	6438 (3)	5703 (8)	30 (2)	37 (3)	33 (3)	−2 (2)	4 (2)	4 (2)	2·43
N(3)	4967 (18)	6239 (8)	3965 (21)	17 (8)	17 (9)	32 (9)	1 (6)	1 (6)	1 (6)	1·52
C(2)	6506 (26)	6186 (11)	3761 (30)	39 (12)	5 (10)	53 (14)	−4 (8)	9 (10)	12 (9)	1·74
C(4)	4892 (26)	6514 (11)	5827 (25)	42 (12)	13 (10)	15 (10)	−3 (9)	10 (9)	1 (8)	2·26
C(5)	6483 (25)	6640 (10)	6918 (26)	39 (12)	15 (10)	6 (9)	0 (8)	−8 (8)	−1 (7)	1·47
C(6)	3242 (26)	6553 (13)	6384 (37)	23 (11)	38 (15)	72 (18)	11 (10)	19 (11)	−13 (12)	2·87
C(7)	6911 (31)	6882 (13)	8964 (30)	54 (14)	31 (13)	29 (13)	−13 (11)	9 (10)	7 (10)	2·59
C(8)	7344 (32)	7772 (16)	9310 (35)	54 (16)	48 (15)	51 (16)	2 (13)	15 (13)	−1 (13)	2·08
O(9)	8681 (19)	7993 (10)	8431 (22)	41 (9)	53 (10)	51 (9)	−11 (8)	4 (7)	19 (8)	2·62
S(11)	2465 (6)	9635 (3)	1580 (7)	30 (2)	29 (2)	31 (2)	1 (2)	8 (2)	6 (2)	2·33
N(31)	518 (19)	9159 (8)	3617 (23)	23 (9)	6 (7)	42 (11)	5 (6)	8 (8)	−3 (7)	1·82
C(21)	484 (28)	9436 (13)	1867 (29)	45 (13)	30 (12)	26 (11)	4 (10)	17 (10)	1 (9)	2·21
C(41)	2134 (23)	9086 (11)	4683 (28)	25 (11)	11 (10)	36 (12)	3 (8)	7 (9)	−3 (8)	1·62
C(51)	3393 (24)	9314 (10)	3820 (25)	30 (11)	5 (8)	22 (10)	5 (8)	5 (8)	4 (7)	1·35
C(61)	2429 (35)	8783 (15)	6701 (36)	86 (18)	63 (16)	37 (15)	27 (14)	29 (13)	31 (13)	3·50
C(71)	5290 (23)	9294 (12)	4500 (26)	22 (10)	34 (12)	22 (10)	−8 (9)	1 (8)	−12 (9)	1·34
C(81)	6143 (36)	8550 (17)	4013 (55)	14 (13)	52 (20)	52 (14)	12 (11)	5 (11)	−6 (14)	2·86
O(91)	7876 (17)	8520 (9)	4715 (19)	13 (7)	50 (8)	43 (8)	9 (6)	15 (6)	15 (7)	2·37

invariant. Several cycles of isotropic and anisotropic temperature refinement reduced R to 0·043, where $R=\sum w||F_o|-|F_c||/\sum w|F_o|$. The refinement was carried out by minimizing $\sum w(|F_o|-|F_c|)^2$ and unit weights were applied.

At this stage a difference map was computed in an attempt to locate the 19 hydrogen atoms. Only nine of these could be located in satisfactory positions. The hydrogen atoms were given isotropic temperature factors equal to the B values of the atoms to which they were attached. These temperature factors were held invariant during the last two cycles of refinement. The final R was 0·041. The positional and thermal parameters of the atoms are listed in Table 1.*

Determination of the absolute configuration

The absolute configuration of the molecule was determined by accepting the configuration which yielded the lowest weighted R after anisotropic temperature refinement had been executed on all the non-hydrogen atoms. With a weight $w=1/\sigma(F_o)$ for each reflexion, values of the weighted residual for the two configurations $R'(hkl)$ and $R'(h\bar{k}l)$ were 0·050 and 0·054 respectively. Hamilton's (1965) significance test was applied to the weighted R factor ratio, $R'(h\bar{k}l)/R'(hkl)=1{\cdot}080$. For the one-dimensional hypothesis, *viz* that the correct configuration applied to the $h\bar{k}l$ data, interpolation in Hamilton's table indicated a ratio of about 1·004 at the 0·5% significance level for 1038 degrees of freedom (1236 reflexions and 198 parameters). This implied that the above hypothesis could be safely rejected and that the correct configuration is defined by the coordinates listed in Table 1. These coordinates apply to a left-handed set of axes as this axial system was used for indexing the hkl data. The correct configuration of the structure is illustrated in Fig. 1.

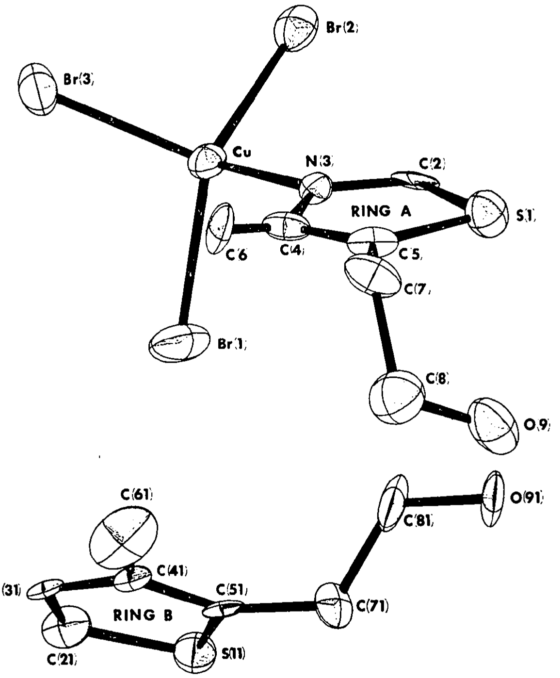

Fig. 1. The labelling scheme for the molecule. The thermal ellipsoids are drawn at the 50% probability level (Johnson, 1965).

Table 1 (*cont.*)

(b) Hydrogen atoms. Fractional coordinates ($\times 10^3$) and thermal parameters

	x	y	z	Isotropic B (Å^2)
H(61)	217 (22)	632 (12)	608 (25)	2·87
H(62)	276 (23)	660 (11)	781 (28)	2·87
H(211)	−36 (21)	940 (10)	71 (24)	2·21
H(611)	346 (27)	844 (14)	736 (28)	3·50
H(612)	345 (26)	898 (12)	786 (29)	3·50
H(711)	538 (20)	934 (10)	573 (21)	1·34
H(712)	590 (20)	972 (9)	398 (21)	1·34
H(811)	573 (24)	848 (12)	260 (29)	2·86
H(31)	−47 (21)	893 (10)	398 (25)	1·82

Description of the structure

The structure and labelling of the atoms are given in Fig. 1. Bond lengths and angles are listed in Tables 2 and 3.

Table 2. *Interatomic distances* (Å) *with their e.s.d.'s in parentheses*

Cu–Br(1)	2·397 (3)	Cu–Br(3)	2·361 (3)
Cu–Br(2)	2·390 (3)	Cu–N(3)	1·96 (1)
Ring *A*		Ring *B*	
N(3)–C(2)	1·28 (3)	N(31)–C(21)	1·36 (3)
C(2)–S(1)	1·71 (2)	C(21)–S(11)	1·68 (2)
S(1)–C(5)	1·74 (2)	S(11)–C(51)	1·73 (2)
C(5)–C(4)	1·36 (3)	C(51)–C(41)	1·36 (3)
C(4)–N(3)	1·46 (2)	C(41)–N(31)	1·35 (2)
C(4)–C(6)	1·47 (3)	C(41)–C(61)	1·53 (3)
C(5)–C(7)	1·52 (3)	C(51)–C(71)	1·49 (3)
C(7)–C(8)	1·58 (4)	C(71)–C(81)	1·53 (4)
C(8)–O(9)	1·42 (3)	C(81)–O(91)	1·37 (3)
C(6)–H(61)	0·93 (18)	C(71)–H(711)	0·89 (16)
C(6)–H(62)	1·20 (22)	C(71)–H(712)	1·00 (17)
C(21)–H(211)	0·96 (15)	C(81)–H(811)	1·02 (21)
C(61)–H(611)	1·04 (21)	N(31)–H(31)	0·97 (19)
C(61)–H(612)	1·08 (19)		

Each asymmetric unit in the structure consists of two main components:

(i) a distorted tetrahedral anion [CuBr$_3$ thiaz]$^-$ in which the copper atom is coordinated to three bro-

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30546 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3. *Intramolecular bond angles* (°) *with their e.s.d.'s in parentheses*

Br(1)–Cu–Br(2)	128·0 (1)	Br(2)–Cu—N(3)	98·8 (5)
Br(2)–Cu–Br(3)	101·8 (1)	Br(3)–Cu—N(3)	131·2 (5)
Br(1)–Cu–Br(3)	104·7 (1)	Cu—N(3)–C(2)	127·0 (14)
Br(1)–Cu–N(3)	96·2 (4)	Cu—N(3)–C(4)	120·5 (12)
Ring *A*		Ring *B*	
C(2)–N(3)–C(4)	112·4 (15)	C(21)–N(31)–C(41)	112·5 (17)
N(3)–C(2)–S(1)	114·8 (16)	N(31)–C(21)–S(11)	111·9 (14)
C(2)–S(1)–C(5)	90·1 (10)	C(21)–S(11)–C(51)	91·6 (10)
S(1)–C(5)–C(4)	111·2 (15)	S(11)–C(51)–C(41)	109·2 (13)
S(1)–C(5)–C(7)	122·4 (15)	S(11)–C(51)–C(71)	120·5 (15)
C(4)–C(5)–C(7)	126·4 (20)	C(41)–C(51)–C(71)	130·2 (16)
N(3)–C(4)–C(5)	111·5 (18)	N(31)–C(41)–C(51)	114·6 (17)
N(3)–C(4)–C(6)	120·5 (16)	N(31)–C(41)–C(61)	120·0 (19)
C(6)–C(4)–C(5)	127·7 (19)	C(61)–C(41)–C(51)	125·3 (17)
C(5)–C(7)–C(8)	114·6 (18)	C(51)–C(71)–C(81)	114·7 (17)
C(7)–C(8)–O(9)	110·3 (21)	C(71)–C(81)–O(91)	114·5 (22)

mines and to the nitrogen atom, N(3), of one thiazole ring,

(ii) an uncoordinated thiazolium cation [thiaz H]$^+$ in which the nitrogen atom N(31) of the thiazole ring is protonated.

The Cu–Br distances vary between 2·361 and 2·397 Å and are normal. The Cu–N length of 1·96 Å is consistent with the lengths in other copper complexes (Caira, Fazakerley, Linder & Nassimbeni, 1973; Fazakerley, Linder, Nassimbeni & Rodgers, 1974; Davey & Stephens, 1971).

Bond lengths and angles in the two thiazole portions of the structure have been compared (Tables 2 and 3). The C(2)–N(3) distance (1·28 Å) in the coordinated thiazole ring (ring *A*) indicates a double-bond character but is noticeably shorter than the C(21)–N(31) distance (1·36 Å) observed in the uncoordinated ring (ring *B*). In an uncoordinated thiazole compound (Fehlman, 1970) this C–N double-bond distance has been reported to have a value of 1·30 Å. The significant lengthening of this bond in the uncoordinated ring of this structure can be attributed to the protonation of N(31). The N(3)–C(4) distance of 1·46 Å appears to be large in comparison with the value of 1·35 Å in the uncoordinated ring. A value of 1·38 Å for this distance has been reported by Fehlman (1970). There are no other significant differences in the bond lengths of the two thiazole fragments of the structure. Corresponding bond angles in the two rings and side chains are generally in good agreement. However O(9) is in a near *cis* configuration with respect to the rest of the side chain as opposed to the *trans* arrangement that O(91) has with the corresponding side chain of the uncoordinated thiazole ring. The two side chains are linked *via* a hydrogen bond between O(9) and O(91) (Fig. 2).

Both thiazole rings are planar. Least-squares planes through the five atoms defining the two rings were computed and deviations of the atoms from these planes were calculated (Table 4).

Hydrogen bonding and molecular packing

The molecular packing is illustrated in the projection down **c*** of the structure (Fig. 3). The effect of the two-

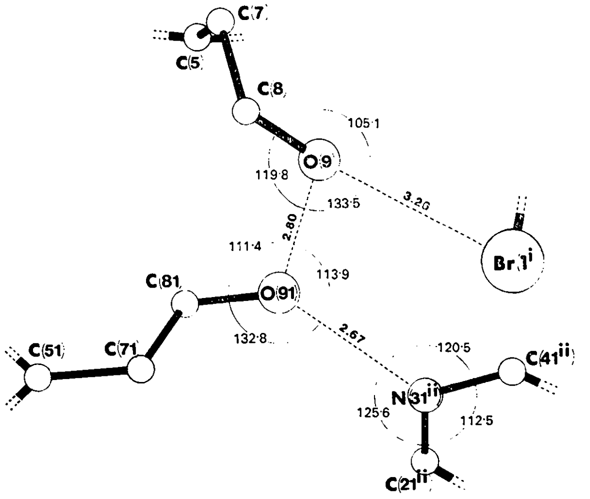

Fig. 2. Proposed hydrogen bonding for the structure. Superscript and symmetry code: (i) $x+1,y,z+1$; (ii) $x+1,y,z$.

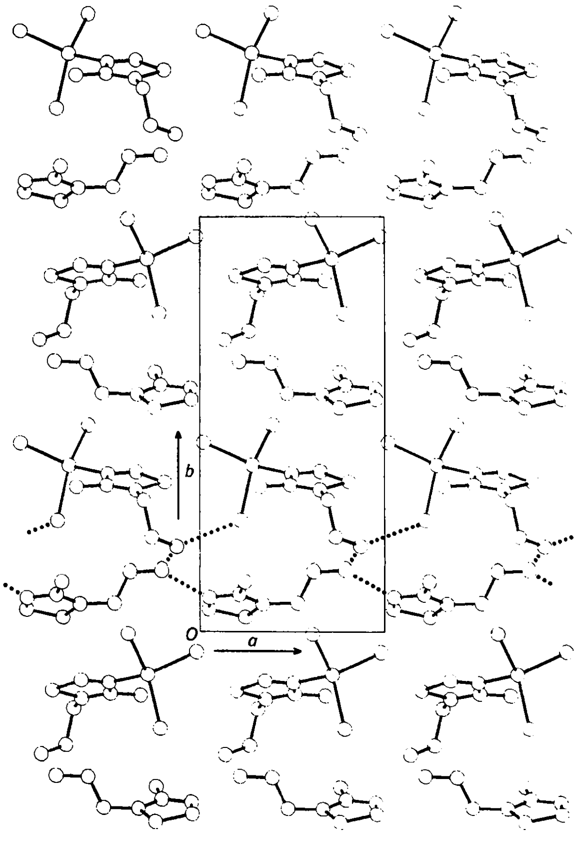

Fig. 3. Projection down **c*** of the structure showing molecular packing. Hydrogen bonds are indicated by dotted lines.

Table 4. *Deviations* (Å) *of atoms from least-squares planes*

(*a*) Deviations of the atoms from the least-squares plane through Ring *A*

S(1)	0·000	Cu*	0·044
C(2)	0·002	C(6)*	−0·112
N(3)	−0·004	C(7)*	−0·077
C(4)	0·004		
C(5)	−0·003		

(*b*) Deviations of the atoms from the least-squares plane through Ring *B*

S(11)	0·009	C(61)*	0·022
C(21)	−0·012	C(71)*	−0·066
N(31)	0·009		
C(41)	0·000		
C(51)	−0·006		

* Denotes atoms not included in the calculation of the least-squares plane.

fold screw axis is clearly visible. Rows of [CuBr$_3$ thiaz]$^-$ anions are linked to rows of [thiaz H]$^+$ cations by hydrogen bonds.

Only one of the nine hydrogens located during the final stages of the refinement was found to be involved in hydrogen bonding. Two other hydrogen bonds have been postulated. The hydrogen-bond scheme is illustrated in Fig. 2.

The protonated hydrogen H(31^{ii}) attached to N(31^{ii}) of the uncoordinated thiazole ring is hydrogen bonded to O(91) of an adjacent molecule. The N···O separation is 2·67 Å and the N–H···O angle is 177·5°. This hydrogen bond links the [thiaz H]$^+$ cations. Although neither of the two hydroxylic hydrogens was located it is postulated that both of these are involved in hydrogen bonding. Both O(9) and O(91) have short contacts which are of the right size for hydrogen-bond formation. The O(9)···Br(1^{i}) separation is 3·26 Å (sum of van der Waals radii = 3·35 Å) and the C(8)–O(9)···Br(1^{i}) angle is 105·1°. It is therefore reasonable to assume that the hydrogen atom H(9) attached to O(9) is hydrogen bonded to Br(1^{i}) and lies on or near the O(9)···Br(1^{i}) line of contact. This hydrogen bond holds the [CuBr$_3$ thiaz]$^-$ anions together.

The O(91)···O(9) separation equals the sum of the van der Waals radii for an O···O contact, *viz* 2·80 Å. The C(81)–O(91)···O(9) angle is 111·4°. It seems likely therefore that the hydroxylic hydrogen H(91) is involved in an interaction with O(9) and lies on or near the O(91)···O(9) line of contact. This interaction binds the rows of anions and cations.

In addition to these hydrogen bonds there are two other short contacts of significance. Both contacts are intramolecular. The first exists between S(1) and O(9), the separation between these two atoms being 3·30 Å (sum of van der Waals radii = 3·25 Å). The second is 3·48 Å between C(2) and Br(2). A similar value has been reported in the structure of *N*-benzyl-4-methylthiazolium bromide (Power, Pletcher & Sax, 1970) in which a hydrogen bond between the bromine and H(2) was reported in view of the polarity of the C(2)–H(2) bond. In this structure no hydrogen bond seems likely as the C(2)–H(2)···Br(2) angle is only 111° assuming that H(2) is 1·00 Å from C(2) and lying in the plane of the thiazole ring. However a weak Br(2)···H(2) interaction cannot be entirely discounted.

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