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# The Crystal and Molecular Structure of 3-(2-Diethylammoniumethoxy)-1,2-benzisothiazole Tetrachlorocuprate

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3-(2-Diethylammoniumethoxy)-1,2-benzisothiazole tetrachlorocuprate, $(C_{13}H_{19}N_2OS)_2^+[CuCl_4]^{2-}$, forms monoclinic crystals ($C2/c$) with cell dimensions $a = 25\cdot24$ (1), $b = 9\cdot38$ (2), $c = 13\cdot58$ (1)Å, $\beta = 91\cdot6(3)°$, $Z = 4$. The organic ion shows no direct coordinative interactions with copper which forms a tetrachlorocuprate ion having a flattened tetrahedral structure (Cu–Cl = 2·259(3), 2·220 (2) Å). The organic cation consists of a planar benzisothiazole system with a side chain containing the tetrahedral protonated diethylammonium group. All hydrogen atoms have been located directly from the last $\Delta F$ synthesis. Packing is determined mainly by hydrogen-bonding interaction (NH$\cdots$Cl = 3·14 Å) involving the tetrachlorocuprate anion and the organic cation and by a short Cl$\cdots$S = 3·34 Å contact.

## Introduction

A series of 1,2-benzisothiazole derivatives has recently been prepared by Vitali, Mossini, Bertaccini & Impicciatore (1968) to study the local anesthetic and antihistaminic properties of the derivatives. Among these products, 3-(2-diethylammoniumethoxy)-1,2-benzisothiazole chloride forms crystalline compounds with $MnCl_2$, $CoCl_2$, $CuCl_2$. This paper reports on the crystal structure of what was originally thought to be a complex between this organic base and copper(II) chloride but which turned out to be the 3-(2-diethylammoniumethoxy)-1,2-benzisothiazole tetrachlorocuprate.

## Experimental

3-(2-Diethylammoniumethoxy)-1,2-benzisothiazole tetrachlorocuprate occurs as yellow monoclinic crystals. When the crystals are examined with plane-polarized light looking down the $a$ and $b$ axes, a dichroic effect is observed: the colour ranges from bright yellow to green-yellow when the electric vector vibrates perpendicular to the $c$ axis and when it vibrates parallel to the $c$ axis respectively.

Crystal data, deduced from rotation and Weissenberg photographs (Cu $K\alpha$, $\bar\lambda = 1\cdot5418$ Å) are as follows (standard deviations given in parentheses are in units of the last decimal figure):

$(C_{13}H_{19}N_2OS)_2[CuCl_4]$, $M = 708\cdot12$, $a = 25\cdot24(1)$,

$b = 9\cdot38$ (2), $c = 13\cdot58$ (1) Å, $\beta = 91\cdot6$ (3)°, $V = 3278$ Å$^3$, $Z = 4$, $D_m = 1\cdot43$ g.cm$^{-3}$, $D_c = 1\cdot41$ g.cm$^{-3}$, $F(000) = 1468$, $\mu = 53\cdot2$ cm$^{-1}$ (Cu $K\alpha$). Space group: $C2/c$ (from systematic absences and structure analysis).

Three-dimensional intensity data were determined photometrically on integrated Weissenberg photographs taken up to the eighth and tenth layers along [010] and [001] respectively (multiple-film technique, Cu $K\alpha$). The total number of independent reflexions was 3532 (possible 3914). No absorption correction was used since the samples were small (mean radii: 0·05 and 0·07 mm). After correction for Lorentz and polarization factors, the intensities were placed on the same relative scale (Rollett & Sparks, 1960) and the absolute scale was established by Wilson's (1942) method.

## Structure analysis and refinement

The structure was solved with the heavy-atom method starting from the three-dimensional Patterson synthesis, which showed quite clearly that copper was on a twofold axis surrounded by four chlorine atoms that could be arranged planarly as well as tetrahedrally. This last arrangement proved to be the correct one; a three-dimensional Fourier synthesis showed all the other non-hydrogen atoms to be well resolved.

Refinement was then carried out, using all the observed reflexions, by block-diagonal and full-matrix least-squares methods. The minimized function was:

Table 1. *Final atomic fractional coordinates* ($\times 10^4$), *thermal parameters* (*) ($\times 10$ Å$^2$) *with e.s.d.'s*

| | $x/a(\sigma)$ | $y/b(\sigma)$ | $z/c(\sigma)$ | $B_{11}(\sigma)$ | $B_{22}(\sigma)$ | $B_{33}(\sigma)$ | $B_{12}(\sigma)$ | $B_{13}(\sigma)$ | $B_{23}(\sigma)$ |
|---|---|---|---|---|---|---|---|---|---|
| Cu | 0 (–) | 2158 (1) | 2500 (–) | 28 (0) | 40 (0) | 33 (0) | – | 0 (0) | – |
| Cl(1) | 334 (1) | 524 (2) | 1465 (1) | 47 (1) | 39 (1) | 51 (1) | 11 (1) | 23 (1) | 11 (1) |
| Cl(2) | 657 (1) | 3702 (2) | 2320 (1) | 32 (1) | 47 (1) | 58 (1) | 14 (0) | 5 (1) | 12 (1) |
| S | 3108 (1) | – 500 (2) | 1920 (1) | 34 (1) | 39 (1) | 26 (0) | –10 (0) | –5 (0) | 2 (0) |
| O | 2027 (1) | 955 (4) | 263 (2) | 23 (1) | 36 (1) | 24 (1) | –4 (1) | –1 (1) | 4 (1) |
| N(1) | 2562 (2) | 452 (5) | 1645 (3) | 30 (2) | 39 (2) | 27 (2) | –4 (1) | 2 (1) | 9 (1) |
| N(2) | 907 (2) | 2328 (5) | –135 (3) | 22 (1) | 30 (2) | 39 (2) | 0 (1) | –1 (1) | –9 (1) |
| C(1) | 3171 (2) | –1185 (5) | 759 (3) | 25 (2) | 28 (2) | 24 (2) | 4 (1) | 2 (1) | 3 (1) |
| C(2) | 3556 (2) | –2076 (6) | 385 (4) | 27 (2) | 33 (2) | 32 (2) | –6 (2) | 4 (1) | –1 (2) |
| C(3) | 3513 (2) | –2463 (6) | –605 (4) | 35 (2) | 31 (2) | 30 (2) | –5 (2) | 3 (2) | 2 (2) |
| C(4) | 3094 (2) | –1945 (6) | –1215 (4) | 39 (2) | 34 (2) | 35 (2) | 6 (2) | 6 (2) | 4 (2) |
| C(5) | 2715 (2) | –1055 (5) | –854 (4) | 32 (2) | 28 (2) | 24 (2) | 2 (2) | 1 (1) | 3 (2) |
| C(6) | 2762 (2) | – 661 (5) | 148 (3) | 22 (2) | 25 (2) | 25 (2) | 7 (1) | –1 (1) | 0 (1) |
| C(7) | 2435 (2) | 281 (5) | 713 (3) | 17 (1) | 32 (2) | 25 (2) | 3 (1) | –2 (1) | 0 (2) |
| C(8) | 1746 (2) | 1922 (6) | 880 (4) | 28 (2) | 41 (2) | 37 (2) | –9 (2) | –2 (2) | 9 (2) |
| C(9) | 1428 (2) | 2889 (6) | 216 (4) | 26 (2) | 31 (2) | 36 (2) | –1 (2) | 0 (2) | –1 (2) |
| C(10) | 956 (2) | 1045 (7) | –751 (5) | 36 (2) | 37 (2) | 50 (3) | 8 (2) | –4 (2) | 2 (2) |
| C(11) | 1270 (3) | 1310 (10) | –1717 (5) | 60 (4) | 79 (5) | 27 (2) | –1 (3) | –1 (2) | 0 (3) |
| C(12) | 596 (3) | 3559 (7) | –580 (6) | 43 (3) | 39 (3) | 66 (4) | –5 (2) | –16 (3) | –12 (3) |
| C(13) | 25 (3) | 3153 (10) | –854 (8) | 40 (3) | 56 (4) | 105 (6) | –15 (2) | –11 (3) | –13 (4) |

* The anisotropic temperature factor is expressed in the form: $\exp\,[-\frac{1}{4}(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^*+2B_{13}hla^*c^*+2B_{23}klb^*c^*)]$.

$\Sigma w|\Delta F|^2$, assuming the weighting scheme: $w=1/(A+BF_o)$ with $A=-24.94$, $B=1.05$ if $|F_o|>|F_o^*|$ and $A=6.0$, $B=0.13$ if $|F_o|<|F_o^*|$, ($F_o^*=30$). Only the overall scale factor was considered in the refinement. Three cycles of isotropic least-squares refinement reduced the conventional residual error index $R$ from 0.23 to 0.13, and the subsequent anisotropic refinement improved it to 0.096. At this point a three-dimensional $\Delta F$ synthesis was sufficient to locate all the hydrogen atoms, including those of the terminal methyl groups which were not defined clearly due to their strong thermal motion. A block-diagonal least-squares isotropic refinement for the hydrogen atoms reduced the conventional $R$ index to the final value of 0.085. No further anisotropic refinement of the non-hydrogen atoms was carried out after the hydrogen atoms were included.

Table 2. *Fractional coordinates* ($\times 10^3$) *and isotropic thermal parameters* (Å$^2$) *for hydrogen atoms*

| | $x/a(\sigma)$ | $y/b(\sigma)$ | $z/c(\sigma)$ | $B(\sigma)$ |
|---|---|---|---|---|
| H(1) | 392 (3) | – 226 (9) | 72 (6) | 3·5 (1·9) |
| H(2) | 380 (3) | – 315 (10) | –82 (6) | 3·4 (1·9) |
| H(3) | 309 (3) | – 231 (10) | – 199 (6) | 3·4 (2·0) |
| H(4) | 239 (3) | – 73 (9) | – 135 (6) | 2·9 (1·9) |
| H(5) | 202 (3) | 259 (10) | 127 (6) | 3·8 (2·0) |
| H(6) | 151 (3) | 109 (10) | 130 (6) | 3·9 (2·0) |
| H(7) | 160 (3) | 312 (10) | – 50 (6) | 3·3 (1·9) |
| H(8) | 136 (3) | 380 (9) | 62 (6) | 3·6 (1·9) |
| H(9) | 73 (3) | 210 (9) | 58 (6) | 3·2 (1·9) |
| H(10) | 118 (3) | 32 (10) | – 58 (7) | 4·3 (2·2) |
| H(11) | 51 (3) | 85 (10) | – 79 (6) | 3·8 (2·2) |
| H(12) | 166 (4) | 142 (11) | – 168 (7) | 4·6 (2·4) |
| H(13) | 101 (4) | 202 (11) | – 203 (7) | 5·2 (2·4) |
| H(14) | 126 (4) | 29 (11) | – 217 (7) | 5·5 (2·4) |
| H(15) | 66 (4) | 455 (11) | – 25 (7) | 5·7 (2·4) |
| H(16) | 75 (4) | 335 (11) | – 133 (7) | 5·4 (2·4) |
| H(17) | 13 (4) | 262 (12) | – 153 (8) | 5·7 (2·8) |
| H(18) | – 18 (4) | 301 (12) | – 17 (8) | 6·8 (2·8) |
| H(19) | – 14 (4) | 418 (13) | – 101 (9) | 6·0 (2·8) |

The final positional and thermal parameters with estimated standard deviations (Cruickshank, 1949, 1956) are given in Tables 1 & 2. A list of observed and calculated structure factors is available from the authors on request. Atomic scattering factors that were used are those of Cromer & Mann (1968).

Data reduction and Fourier calculations were performed on the Olivetti Elea 6001/S computer of the Centro di Calcolo Elettronico dell'Università di Parma, using the programs of Nardelli, Musatti, Domiano & Andreetti (1964, 1965); the least-squares refinement was performed on the CDC 6600 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale, using the programs written by Immirzi (1969).

### Description of the structure and discussion

Fig. 1 shows the structure projected along the $b$ axis. It consists of discrete tetrachlorocuprate anions and 3-(2-diethylammoniumethoxy)-1,2-benzisothiazole cations. Bond distances and angles are given in the figure and in Table 3. These data and the clinographic projection of Fig. 2 reveal that the anion has a flattened tetrahedral structure. The degree of flattening is well indicated by the dihedral angle of 49·5° between the planes defined by Cl(1)–Cu–Cl(1') and Cl(2)–Cu–Cl(2') which would be 90° for a regular tetrahedral arrangement and 0° for a square planar one. The tetrahedron is flatter than that formed for the same ion in $N,N'$-dimethyl-4,4'-bipyridinium tetrachlorocuprate (Russell & Wallwork, 1969) in which the same dihedral angle is 67·8°, four Cl–Cu–Cl angles are about 100° and two are 129°. Also, the tetrahedron is flatter than that formed for the same ion in $Cs_2CuCl_4$ (Morosin & Lingafelter, 1961) in which four of the six Cl–Cu–Cl angles in the anion are about 102° and two are about 125°.
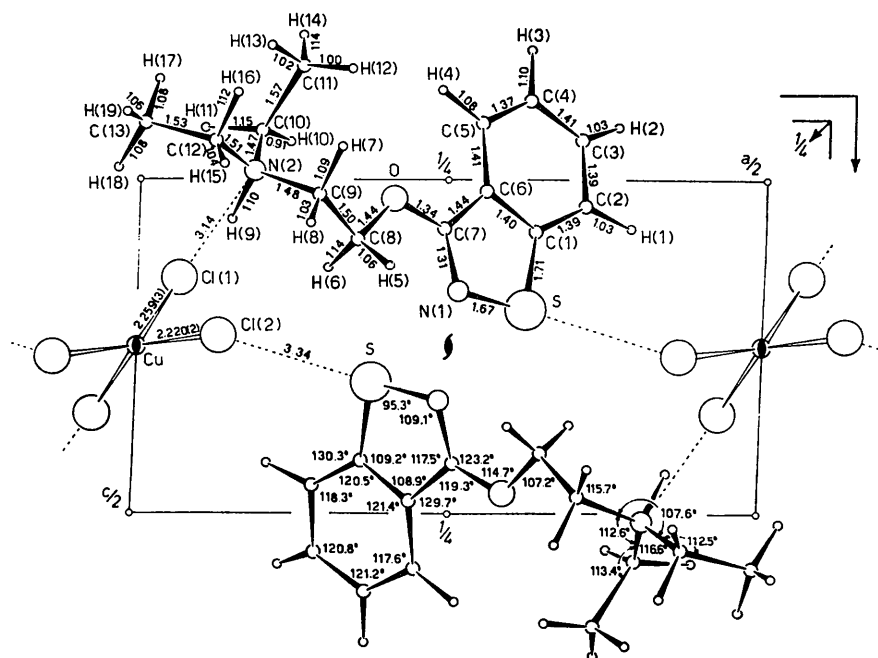
Fig. 1. Projection of $(C_{13}H_{19}N_2OS)_2^+ [CuCl_4]^{2-}$ along (010).



$\bigcirc$S  $\bigcirc$Cl  $\bigcirc$O  $\bigcirc$N  $\bullet$Cu  $\circ$C  $\circ$H
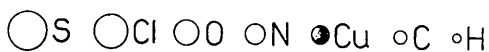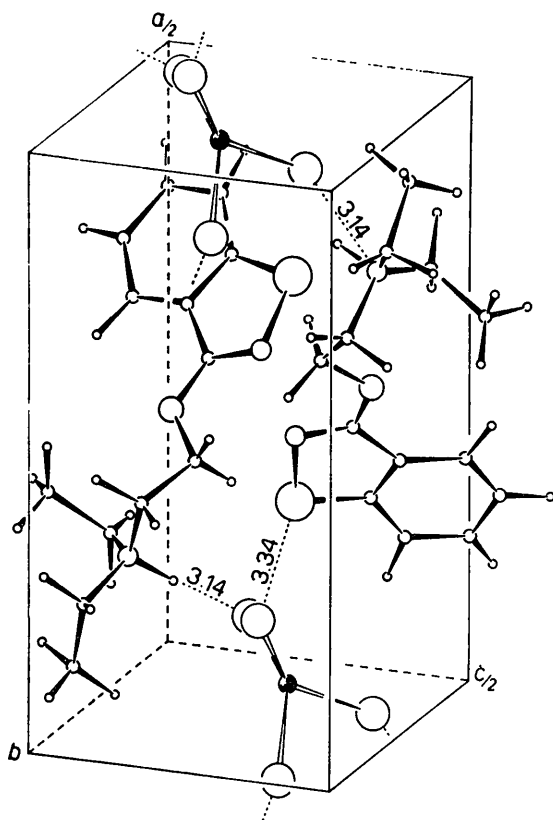
Fig. 2. Clinographic projection of $(C_{13}H_{19}N_2OS)_2^+ [CuCl_4]^{2-}$

The Cu–Cl distances in these compounds are in the range 2·18–2·25 Å; the corresponding distances found in the present determination also fall in this range. The distorted tetrahedral coordination around Cu agrees quite well with the spectral and magnetic properties of the compound. In fact the electronic spectrum shows an enlarged $d$–$d$ band at 850 m$\mu$ (the same band is at 890 m$\mu$ for $Cs_2CuCl_4$, Gill & Nyholm, 1959). Also, the magnetic moment is $\mu_{eff} = 1·76$ B.M., *i.e.* smaller than the value (2·2 B.M.) that could be expected for a perfect tetrahedron for which there is a relatively large orbital contribution (for $Cs_2CuCl_4$, Gill & Nyholm found $\mu_{eff} = 1·96$ B.M.).

Since the anion is on a twofold axis, two independent Cu–Cl distances are significantly different $[\Delta/\sigma = (l_1 - l_2)/(\sigma_1^2 + \sigma_2^2)^{\frac{1}{2}} = 10·8]$: the longer distance (2·259 Å) corresponds to Cl(1) which is engaged in hydrogen bonding with the ammonium nitrogen N(2) and the shorter distance concerns Cl(2) which forms a short contact (3·340 Å) with a sulphur atom of an adjacent cation. The benzisothiazole system is not perfectly planar, but planarity is observed for each ring separately; their least-squares planes are: C(1)–C(2)–C(3)–C(4)–C(5)–C(6) with coordinates $0·5636X' + 0·7909Y' - 0·2383Z' = 3·3793$ and C(1)–C(6)–C(7)–N(1)–S with coordinates $0·5798X' + 0·7782Y' - 0·2410Z' = 3·5113$. The $X'$, $Y'$, $Z'$ are the orthogonal coordinates obtained from the $x, y, z$, monoclinic ones using the matrix|10 cos$\beta$|010|00 sin$\beta$|. The dihedral angle formed by these planes is 178·8°.

Bond distances and angles in the benzene ring are similar to these usually found in this type of ring. In the isothiazole ring the C(1)–S and N(1)–S distances

correspond well with those of single bonds considering the C and N atoms in $sp^2$ states. The double bond is localized mainly between C(7) and N(1) but some $\pi$-delocalization is spread also on C(7)–C(6) and C(7)–O bonds. Distances in the isothiazole ring agree fairly well with those found in 7-chloro-1,2-benzisothiazolin-3-one (Cavalca, Fava Gasparri, Mangia & Pelizzi, 1969), but there are some noticeable differences between the angles in the ring, particularly those on S (88·9°) and on C(7) (108·5°) which are larger and that on N(1) (118·4°) which is narrower. Perhaps these an-

gular deformations are due to some steric hindrance between S and Cl(2) of an adjacent molecule. Indeed, the distance between these two atoms (3·34 Å) is appreciably shorter than the expected value (3·50 Å), assuming 1·70 Å for the van der Waals radius of sulphur (the value 1·85 Å, usually given, is too high as it has been shown in many instances: Donohue, 1950; Ždanov & Zvonkova, 1950; Van der Helm, Lessor & Merritt, 1960; Nardelli, Fava Gasparri, Giraldi Battistini & Domiano, 1966; Andreetti, Cavalca, Manfredotti & Musatti, 1969; Valle, Busetti, Mammi & Carazzolo,

Table 3. *Bond distances and angles*

(a) In [CuCl₄]²⁻

| | | | | |
|---|---|---|---|---|
| Cu–Cl(1) | 2·259 (3) Å | Cl(1)–Cu–Cl(1') (*) | 94·56 (6)° |
| Cu–Cl(2) | 2·220 (2) | Cl(2)–Cu–Cl(2') | 98·59 (6) |
| | | Cl(1)–Cu–Cl(2) | 94·64 (5) |
| | | Cl(2)–Cu–Cl(1') | 143·53 (5) |

\* Primes refer to the $\bar{x}$, $y$, $\frac{1}{2}-z$ position.

(b) In the organic cation

| | | | |
|---|---|---|---|
| S——C(1) | 1·714 (5) Å | N(1)—S——C(1) | 95·3 (2)° |
| S——N(1) | 1·675 (5) | S——N(1)—C(7) | 109·1 (3) |
| N(1)—C(7) | 1·31 (1) | N(1)—C(7)—C(6) | 117·5 (4) |
| C(7)—C(6) | 1·44 (1) | C(7)—C(6)—C(1) | 108·9 (4) |
| C(6)—C(1) | 1·40 (1) | C(6)—C(1)—S | 109·2 (3) |
| C(1)—C(2) | 1·39 (1) | S——C(1)—C(2) | 130·3 (4) |
| C(2)—C(3) | 1·39 (1) | C(2)—C(1)—C(6) | 120·5 (4) |
| C(3)—C(4) | 1·41 (1) | C(1)—C(6)—C(5) | 121·4 (4) |
| C(4)—C(5) | 1·37 (1) | C(6)—C(5)—C(4) | 117·6 (4) |
| C(5)—C(6) | 1·41 (1) | C(4)—C(3)—C(2) | 120·8 (5) |
| C(7)—O | 1·34 (1) | C(3)—C(2)—C(1) | 118·3 (4) |
| O——C(8) | 1·44 (1) | C(5)—C(6)—C(7) | 129·7 (4) |
| C(8)—C(9) | 1·50 (1) | C(3)—C(4)—C(5) | 121·2 (5) |
| C(9)—N(2) | 1·48 (1) | C(6)—C(7)—O | 119·3 (4) |
| N(2)—C(10) | 1·47 (1) | N(1)—C(7)—O | 123·2 (4) |
| C(10)—C(11) | 1·57 (1) | C(7)—O——C(8) | 114·7 (4) |
| N(2)—C(12) | 1·51 (1) | O——C(8)—C(9) | 107·2 (4) |
| C(12)—C(13) | 1·53 (1) | C(8)—C(9)—N(2) | 115·7 (4) |
| | | C(9)—N(2)—C(10) | 112·6 (4) |
| | | C(10)–N(2)—C(12) | 116·6 (5) |
| | | C(12)–N(2)—C(9) | 107·6 (4) |
| | | N(2)—C(12)—C(13) | 112·5 (6) |
| | | N(2)—C(10)—C(11) | 113·4 (5) |

(c) Concerning the hydrogen atoms

| | | | | | |
|---|---|---|---|---|---|
| C(2)—H(1) | 1·03 (8) Å | C(1)—C(2)—H(1) | 124·3 (5·3)° | N(2)—C(10)–H(10) | 121·7 (5·7)° |
| C(3)—H(2) | 1·03 (9) | C(3)—C(2)—H(1) | 115·4 (4·6) | H(10)–C(10)–C(11) | 90·0 (5·6) |
| C(4)—H(3) | 1·10 (9) | C(2)—C(3)—H(2) | 113·4 (4·7) | H(10)–C(10)–H(11) | 120·2 (7·6) |
| C(5)—H(4) | 1·08 (8) | C(4)—C(3)—H(2) | 125·7 (4·7) | H(11)–C(10)–C(11) | 120·7 (6·3) |
| C(8)—H(5) | 1·06 (9) | C(3)—C(4)—H(3) | 115·8 (4·5) | H(11)–C(10)–N(2) | 93·4 (4·9) |
| C(8)—H(6) | 1·14 (9) | C(5)—C(4)—H(3) | 122·9 (4·5) | C(10)–C(11)–H(12) | 119·8 (5·5) |
| C(9)—H(7) | 1·09 (8) | C(4)—C(5)—H(4) | 118·0 (4·3) | H(12)–C(11)–H(14) | 96·5 (7·6) |
| C(9)—H(8) | 1·03 (9) | C(6)—C(5)—H(4) | 124·3 (4·3) | H(14)–C(11)–H(13) | 108·4 (7·5) |
| N(2)—H(9) | 1·10 (8) | O——C(8)—H(5) | 109·9 (4·4) | H(14)–C(11)–C(10) | 108·8 (5·0) |
| C(10)–H(10) | 0·91 (9) | H(5)—C(8)—H(6) | 120·3 (6·4) | C(10)–C(11)–H(13) | 96·3 (5·4) |
| C(10)–H(11) | 1·15 (9) | H(6)—C(8)—C(9) | 115·7 (4·5) | H(12)–C(11)–H(13) | 126·4 (8·1) |
| C(11)–H(12) | 1·00 (9) | H(6)—C(8)—O | 97·6 (4·5) | N(2)—C(12)–H(15) | 116·2 (5·5) |
| C(11)–H(13) | 1·02 (10) | H(5)—C(8)—C(9) | 106·0 (4·9) | H(15)–C(12)–C(13) | 117·2 (5·3) |
| C(11)–H(14) | 1·14 (10) | C(8)—C(9)—H(8) | 106·1 (4·6) | H(15)–C(12)–H(16) | 119·6 (7·6) |
| C(12)–H(15) | 1·04 (10) | H(8)—C(9)—N(2) | 107·6 (4·4) | H(16)–C(12)–N(2) | 92·1 (5·2) |
| C(12)–H(16) | 1·12 (10) | N(2)—C(9)—H(7) | 98·7 (4·3) | H(16)–C(12)–C(13) | 94·9 (4·9) |
| C(13)–H(17) | 1·08 (11) | H(7)—C(9)—H(8) | 112·2 (6·7) | C(12)–C(13)–H(17) | 93·5 (5·7) |
| C(13)–H(18) | 1·08 (11) | H(7)—C(9)—C(8) | 116·3 (4·4) | C(12)–C(13)–H(18) | 106·7 (5·5) |
| C(13)–H(19) | 1·06 (12) | C(9)—N(2)—H(9) | 99·3 (4·2) | C(12)–C(13)–H(19) | 100·0 (6·0) |
| | | H(9)—N(2)—C(12) | 106·6 (4·4) | H(18)–C(13)–H(19) | 95·6 (8·4) |
| | | H(9)—N(2)—C(10) | 112·6 (4·6) | H(18)–C(13)–H(17) | 143·6 (8·7) |
| | | | | H(17)–C(13)–H(19) | 110·6 (8·4) |

1969; Cavalca, Fava Gasparri, Mangia & Pelizzi, 1969; Valle, Cojazzi, Busetti & Mammi, 1970; Meyerhöffer, 1970). This short contact is of the same order of magnitude as that (3·28 Å) found in 7-chloro-1,2-benzoisothiazolin-3-one (Cavalca, Fava Gasparri, Mangia & Pelizzi, 1969).

The oxygen atom lies in the plane of the isothiazole ring (its distance from the plane is 0·06 Å) and this stems from the trigonal $sp^2$ character of the bonds formed by C(7) and from the $\pi$-delocalization along O–C(7)–N(1)–C(6). The C(8)–O distance corresponds to a single bond, so it is possible to have some rotation around it; nevertheless the C(8) atom is only 0·14 Å out of the isothiazole plane, the internal rotation angle being as small as 3·0°. The conformation of the ethoxy group is *gauche* as shown in Fig. 3. The environment of N(2) is tetrahedral as expected for a protonated amine, the hydrogen atom being directed towards Cl(1) and involved in a normal hydrogen bond [N(2)–H(9)··· Cl(1) = 3·139 (5) Å, N(2)–H(9)–Cl(1) = 145·4 (4·3)°].

Table 4. *Van der Waals contacts* (*less than* 3·50 Å)

| | |
|---|---|
| Cl(2)–S(i) | 3·340 (4) Å |
| S——C(4)(ii) | 3·418 (7) |
| N(1)–C(4)(ii) | 3·46 (1) |
| N(1)–C(5)(ii) | 3·45 (1) |
| C(1)–C(5)(iii) | 3·42 (1) |
| C(4)–C(6)(iii) | 3·46 (1) |
| C(5)–C(1)(iii) | 3·42 (1) |
| C(5)–C(6)(iii) | 3·45 (1) |
| C(6)–C(9)(iv) | 3·35 (1) |

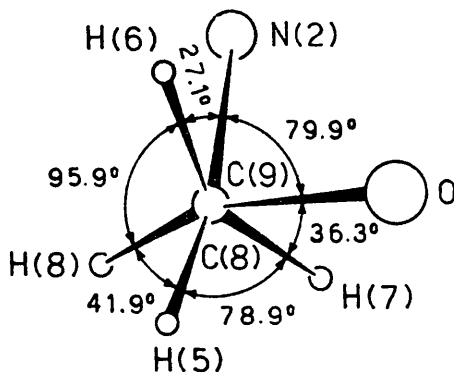| | | |
|---|---|---|
| (i) | $\frac{1}{2}-x,$ | $\frac{1}{2}+y, \frac{1}{2}-z$ |
| (ii) | $x,$ | $\bar{y}, \frac{1}{2}+z$ |
| (iii) | $\frac{1}{2}-x, -\frac{1}{2}-y,$ | $\bar{z}$ |
| (iv) | $\frac{1}{2}-x, \frac{1}{2}-y,$ | $\bar{z}$ |



Fig. 3. Conformation of the ethoxy group.

The two terminal methyl groups show rather high thermal factors that suggest a pronounced vibration for these groups; this is indicated also by the shape of the hydrogen-atom peaks which are spread out and elongated as a result of the anisotropic character of the motion; therefore, their location is less reliable.

Packing is mainly determined by the N(2)–H(9)··· Cl(1) hydrogen bond, by the short Cl(2)···S contact (Fig. 2) and by the C···C and C···N contacts listed in Table 4.

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