

Bis(trimethylsulfonium) Hexachlorodicuprate(II) and Bis(trimethylsulfonium) Tetrachlorocuprate(II)

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Abstract. $[(\text{CH}_3)_3\text{S}]_2\text{Cu}_2\text{Cl}_6$, $M_r = 494.2$, monoclinic, $P2_1/n$, $a = 6.172$ (1), $b = 10.162$ (2), $c = 13.499$ (3) Å, $\beta = 97.25$ (2)°, $Z = 2$, $V = 839.9$ (3) Å³, Mo $K\alpha$ ($\lambda = 0.71069$ Å), $\mu = 3.72$ mm⁻¹, $D_x = 1.95$ Mg m⁻³, $T = 295$ K, $F(000) = 492$, 2177 unique observed [$|F| \geq 3\sigma(F)$] reflections refined to $R = 0.0487$ and $wR = 0.0671$ for 83 parameters. The structure consists of quasiplanar symmetrically bridged $\text{Cu}_2\text{Cl}_6^{2-}$ dimeric anions and $(\text{CH}_3)_3\text{S}^+$ cations. Each Cu^{II} ion forms a semi-coordinate bond to a Cl atom in an adjacent dimer [$\text{Cu}\cdots\text{Cl} = 2.699$ (1) Å]. This gives an elongated square-pyramidal coordination geometry for the Cu^{II} species, and leads to the formation of stacks of $\text{Cu}_2\text{Cl}_6^{2-}$ anions running parallel to the a axis. The sixth coordination site for the Cu^{II} ion is occupied by an S atom from a $(\text{CH}_3)_3\text{S}^+$ cation at a distance of 3.646 Å. $[(\text{CH}_3)_3\text{S}]_2\text{CuCl}_4$, $M_r = 359.7$, monoclinic, $P2_1/n$, $a = 15.752$ (4), $b = 12.231$ (2), $c = 17.462$ (3) Å, $\beta = 114.60$ (1)°, $V = 3059.0$ (1) Å³, $Z = 8$, $\mu = 2.37$ mm⁻¹, $D_x = 1.56$ Mg m⁻³, $T = 295$ K, $F(000) = 1464$, 3262 observed reflections refined to $R = 0.0439$ and $wR = 0.0500$ for 271 parameters. The structure consists of isolated tetrahedrally distorted CuCl_4^{2-} anions and $(\text{CH}_3)_3\text{S}^+$ cations. The two crystallographically independent anions have average *trans* Cl—Cu—Cl angles of 128.6 and 135.0°, respectively. Each cation interacts with three Cl⁻ ions ($\text{S}\cdots\text{Cl}$ distances of 3.44–4.07 Å) to form a severely trigonally elongated octahedral environment for each S atom.

Introduction. As part of an interest in new solid-state systems with tribridged CuX_3^- chains consisting of face-shared Jahn–Teller distorted octahedra, we have synthesized a wide variety of $A_x\text{Cu}_y\text{X}_z$ salts, where A is an organic cation, $X = \text{Cl}^-$ or Br^- , and x, y, z are integers (see Bond, 1990; Bond & Willett, 1989; Bond, Willett & Rubenacker, 1990). Since the holo-type structure, found for example in CsNiCl_3 , is hexagonal (Weenk & Harwig, 1977), one synthetic strategy involved is the utilization of cations with C_3 symmetry. In this manner, it was hoped to find analogs of $[(\text{CH}_3)_4\text{N}]\text{CuCl}_3$ (Weenk & Spek, 1976) or $[(\text{CH}_3)_3\text{NH}]_3\text{Cu}_2\text{Cl}_7$ (Clay, Murray-Rust & Murray-

Rust, 1973), both of which contain the desired tri-bridged structure. The former, in particular, undergoes a complex series of phase transitions related to the dynamic Jahn–Teller effect (Willett, Bond, Haije, Soonieus & Maaskant, 1988). With this thought in mind, copper(II) chloride salts of the trimethylsulfonium cation, $(\text{CH}_3)_3\text{S}^+$, were prepared. The structure determination of $(\text{CH}_3)_3\text{SCuCl}_3$ was undertaken and, because of an interesting structural feature associated with the cation–anion interactions, the structure of $[(\text{CH}_3)_3\text{S}]_2\text{CuCl}_4$ was also investigated. The results of these two structure investigations are reported herein.

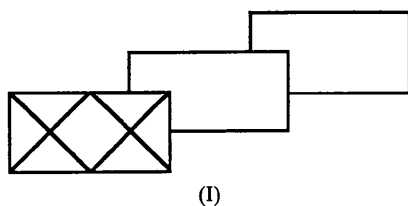
Experimental. Trimethylsulfonium iodide, $(\text{CH}_3)_3\text{SI}$, was prepared by reacting dimethyl sulfide with methyl iodide in a bomb at elevated temperature (~573 K). Metathesis of the product with silver chloride gave $(\text{CH}_3)_3\text{SCl}$. Reaction of $(\text{CH}_3)_3\text{SCl}$ with excess $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ in a concentrated HCl solution, and subsequent reduction of the volume of solution by evaporation, yielded a microcrystalline sample of $(\text{CH}_3)_3\text{SCuCl}_3$. Recrystallization from methanol yielded a mixture of crystals, including red/yellow pleochroic needles of $(\text{CH}_3)_3\text{SCuCl}_3$ and yellow needles of $[(\text{CH}_3)_3\text{S}]_2\text{CuCl}_4$. Diffraction data were collected using standard procedures on a Syntex $P2_1$ diffractometer, upgraded to Nicolet $P3F$ specifications, equipped with a graphite monochromator (Campana, Shepard & Litchman, 1981). Data reduction, structure solution, refinement and analysis were carried out on a Data General Eclipse computer with the *SHELXTL* crystallographic package (Sheldrick, 1985). Data collection and refinement parameters are given in Table 1. Final positional parameters for the two compounds are given in Tables 2 and 3, with selected bond distances and angles listed in Tables 4, 5 and 6.*

* Lists of anisotropic thermal parameters, H-atom parameters and the observed and calculated structure factors, as well as stereoviews of the unit cells, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54879 (48 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *X-ray data-collection parameters*

| | Bis(trimethylsulfonium) hexachloro-dicuprate(II) | Bis(trimethylsulfonium) tetrachlorocuprate(II) |
|--|--|--|
| Type of absorption correction | Empirical, ψ scan | Empirical, ψ scan |
| transmission range | 0.293–0.708 | 0.65–0.94 |
| Data-collection technique | ω scan | ω scan |
| Scan range | 0.9° | 0.9° |
| Scan speed | 6–60° min ⁻¹ | 6–60° min ⁻¹ |
| Check reflections (monitored every 96 reflections) | 111, 002 | 204, 400 |
| Total reflections | 2783 | 4375 |
| 2 θ_{\max} | 60° | 45° |
| Unique reflections | 2449 | 3929 |
| R for equivalent reflections | 0.040 | 0.029 |
| <i>h, k, l</i> | 0 ≤ <i>h</i> ≤ 8 -14 ≤ <i>k</i> ≤ 0 -18 ≤ <i>l</i> ≤ 18 | 0 ≤ <i>h</i> ≤ 16 0 ≤ <i>k</i> ≤ 13 -17 ≤ <i>l</i> ≤ 17 |
| Structure solution package | Nicolet <i>SHELXTL</i> (V. 5.1) | Nicolet <i>SHELXTL</i> (V. 5.1) |
| Structure solution technique | Direct methods | Direct methods |
| R | 0.0487 (0.0526 all data) | 0.0439 (0.0551 all data) |
| wR | 0.0671 (0.0692 all data) | 0.0500 (0.0546 all data) |
| with $w = 1/[\sigma^2(F) + g(F)^2]$ | $g = 0.00335$ | $g = 0.00035$ |
| Function minimized | $\sum w(F_o - F_c)^2$ | $\sum w(F_o - F_c)^2$ |
| Goodness of fit | 0.965 | 1.354 |
| $ \Delta/\sigma _{\max}$ | 0.010 | 0.047 |
| Thermal parameters | Anisotropic on all non-hydrogen atoms | Anisotropic on all non-hydrogen atoms |
| Hydrogen atoms | Constrained to C—H = 0.96 Å; thermal parameters fixed at 1.2 × <i>U</i> (carbon) | Constrained to C—H = 0.96 Å; thermal parameters fixed at 1.2 × <i>U</i> (carbon) |
| Largest peak on final difference map | 1.6 e Å ⁻³ near Cu | 0.7 e Å ⁻³ near S(2) |
| Most negative peak on final difference map | -0.9 e Å ⁻³ | -0.3 e Å ⁻³ |
| Extinction correction | Yes | No |

Discussion. The structure of [(CH₃)₃S]₂Cu₂Cl₆ contains stacks of pseudo-planar CuCl₆²⁻ dimeric anions flanked by two rows of sulfonium cations, as seen in Fig. 1. The structural characteristics of the dimers are consistent with those observed in other quasi-planar copper(II) halide oligomers (Willett, 1987, 1988). The copper(II) ion has an elongated square-pyramidal coordination geometry (Cu—Cl = 2.299 Å, average, basal and Cu...Cl = 2.699 Å, axial) with the distortion of the basal plane intermediate between the idealized square pyramidal and folded geometries. The bridging Cu—Cl distances average 0.055 Å longer than the terminal Cu—Cl distance. The formation of the elongated square-pyramidal geometry results in the development of the aforementioned stacks. If each Cu₂Cl₆²⁻ dimer is represented as an 'envelope', the stack of dimers can be represented by the stacking diagram, (I).



This is just one of a variety of stacking patterns assumed by these dimeric systems (Geiser, Willett, Lindbeck & Emerson, 1986; Bond & Willett, 1989) and is designated a 2(3/2, 1/2) pattern in the Geiser

Table 2. *Atomic coordinates* (× 10⁴) and *isotropic thermal parameters* (Å² × 10³) for [(CH₃)₃S]₂Cu₂Cl₆

The equivalent isotropic *U* is defined as one-third of the trace of the orthogonalized *U_{ij}* tensor.

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>U_{eq}</i> |
|-------|-----------|-----------|-----------|-----------------------|
| Cu | 2353 (1) | 593 (1) | -427 (1) | 29 (1) |
| S | 976 (2) | -493 (1) | -7143 (1) | 40 (1) |
| Cl(1) | 4855 (1) | -400 (1) | -1275 (1) | 35 (1) |
| Cl(2) | 3127 (1) | 2637 (1) | -968 (1) | 41 (1) |
| Cl(3) | 601 (1) | -1423 (1) | -267 (1) | 38 (1) |
| C(1) | 2235 (6) | -1970 (4) | -7496 (3) | 46 (1) |
| C(2) | -1180 (6) | -1118 (4) | -6497 (3) | 46 (1) |
| C(3) | 2878 (7) | 70 (4) | -6128 (3) | 53 (1) |

notation. This stacking arrangement is, in part, dictated by the unusual type of interaction of the sulfonium cation with the stacks. As seen in Fig. 1, the S atom is positioned directly in the sixth coordination site of the Cu^{II} ion, preventing formation of an additional Cu...Cl semi-coordinate interaction. Although the Cu—S distance is long (3.646 Å), it is nearly identical with the more normal (electrostatic) S...Cl(1) interaction distance of 3.660 Å.

The tetrachloro salt consists simply of discrete CuCl₄²⁻ anions and (CH₃)₃S⁺ cations as seen in Fig. 2. The anions show the normal distortion from tetrahedral geometry, exhibiting approximate $\bar{4}m2$ symmetry. The extent of distortion, conveniently measured by the average *trans* Cl—Cu—Cl bond angles, is 128.6° for Cu(1) and 135.0° for Cu(2), with the Cu—Cl bond distances averaging 2.246 Å. The former ion thus has one of the smaller distortions

Table 3. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for $[(\text{CH}_3)_3\text{S}]_2\text{CuCl}_4$

The equivalent isotropic U is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

| | x | y | z | U_{eq} |
|-------|----------|-----------|----------|-----------------|
| Cu(1) | 5810 (1) | 2327 (1) | 1469 (1) | 46 (1) |
| Cl(1) | 5699 (1) | 512 (1) | 1357 (1) | 67 (1) |
| Cl(2) | 6701 (1) | 3483 (1) | 1111 (1) | 65 (1) |
| Cl(3) | 4383 (1) | 2844 (1) | 556 (1) | 69 (1) |
| Cl(4) | 6469 (1) | 2539 (1) | 2878 (1) | 66 (1) |
| Cu(2) | 1014 (1) | 2542 (1) | 1632 (1) | 51 (1) |
| Cl(5) | 1282 (1) | 4346 (1) | 1606 (1) | 74 (1) |
| Cl(6) | 1176 (1) | 2090 (1) | 452 (1) | 76 (1) |
| Cl(7) | -270 (1) | 1530 (1) | 1402 (1) | 73 (1) |
| Cl(8) | 1804 (1) | 2233 (2) | 3011 (1) | 95 (1) |
| S(1) | 348 (1) | 8689 (1) | 1638 (1) | 59 (1) |
| C(1) | 602 (5) | 7265 (4) | 1736 (5) | 81 (4) |
| C(2) | 989 (4) | 9132 (5) | 1059 (4) | 71 (3) |
| C(3) | 1083 (5) | 9157 (6) | 2662 (4) | 85 (4) |
| S(2) | 2674 (1) | 9742 (2) | 367 (1) | 77 (1) |
| C(4) | 3322 (5) | 10442 (6) | 1303 (4) | 80 (3) |
| C(5) | 2650 (6) | 10693 (6) | -410 (4) | 92 (4) |
| C(6) | 3489 (7) | 8772 (7) | 337 (6) | 137 (7) |
| S(3) | 6226 (1) | 6291 (1) | 1479 (1) | 53 (1) |
| C(7) | 6703 (4) | 5694 (5) | 2502 (4) | 66 (3) |
| C(8) | 6082 (5) | 7679 (4) | 1720 (5) | 79 (4) |
| C(9) | 5053 (4) | 5815 (5) | 1070 (4) | 71 (3) |
| S(4) | 8817 (1) | 5096 (1) | 529 (1) | 57 (1) |
| C(10) | 7680 (4) | 5675 (5) | 118 (4) | 83 (4) |
| C(11) | 8959 (4) | 4675 (5) | 1556 (3) | 68 (3) |
| C(12) | 8606 (5) | 3831 (5) | -12 (4) | 90 (4) |

Table 4. Bond lengths (\AA) and angles ($^\circ$) for $[(\text{CH}_3)_3\text{S}]_2\text{Cu}_2\text{Cl}_6$

| | | | |
|-----------|-----------|------------------|-----------|
| Cu—Cl(1) | 2.272 (1) | Cl(1)—Cu—Cl(2) | 93.4 (1) |
| Cu—Cl(2) | 2.272 (1) | Cl(1)—Cu—Cl(3) | 90.5 (1) |
| Cu—Cl(3) | 2.339 (1) | Cl(2)—Cu—Cl(3) | 161.5 (1) |
| Cu—Cl(1A) | 2.699 (1) | Cl(1)—Cu—Cl(1A) | 89.8 (1) |
| Cu—Cl(3A) | 2.312 (1) | Cl(2)—Cu—Cl(1A) | 101.7 (1) |
| S—C(1) | 1.782 (4) | Cl(3)—Cu—Cl(1A) | 96.4 (1) |
| S—C(2) | 1.796 (4) | Cl(1)—Cu—Cl(3A) | 170.9 (1) |
| S—C(3) | 1.783 (4) | Cl(2)—Cu—Cl(3A) | 90.3 (1) |
| | | Cl(3)—Cu—Cl(3A) | 83.4 (1) |
| | | Cl(1A)—Cu—Cl(3A) | 97.6 (1) |
| | | C(1)—S—C(2) | 101.9 (2) |
| | | C(1)—S—C(3) | 102.1 (2) |
| | | C(2)—S—C(3) | 101.3 (2) |
| | | Cu—Cl(1)—Cu(A) | 90.2 (1) |
| | | Cu—Cl(3)—Cu(B) | 96.6 (1) |

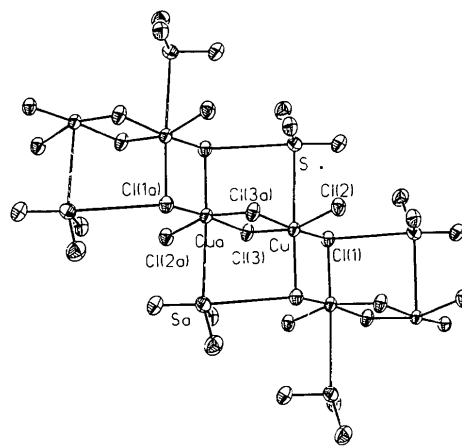
from tetrahedral geometry, when compared with the 62 previously reported isolated CuCl_4^{2-} anions (Willett, Halvorson & Patterson, 1990), while the latter falls in the most favored range found in the extent of distortion. Each of the sulfonium cations has three closest Cl neighbors (tabulated in Table 5) so as to define an octahedral geometric arrangement with an extreme trigonal distortion about each of the S atoms. The S...Cl contact distances range from 3.45 to 4.05 \AA . It is these interactions which are responsible for the existence of two crystallographically independent anions: seven of the twelve contacts are with the Cu(2) anion; only five are with Cl atoms of the Cu(1) anion. No contacts exist to Cl(1), and this Cu—Cl bond distance is the shortest.

Table 5. Bond lengths (\AA) and angles ($^\circ$) for $[(\text{CH}_3)_3\text{S}]_2\text{CuCl}_4$

| | | | |
|-------------|------------|-------------------|-----------|
| Cu(1)—Cl(1) | 2.229 (1) | Cl(1)—Cu(1)—Cl(2) | 129.4 (1) |
| Cu(1)—Cl(2) | 2.256 (2) | Cl(1)—Cu(1)—Cl(3) | 101.5 (1) |
| Cu(1)—Cl(3) | 2.239 (1) | Cl(2)—Cu(1)—Cl(3) | 100.5 (1) |
| Cu(1)—Cl(4) | 2.253 (2) | Cl(1)—Cu(1)—Cl(4) | 101.2 (1) |
| Cu(2)—Cl(5) | 2.250 (2) | Cl(2)—Cu(1)—Cl(4) | 100.0 (1) |
| Cu(2)—Cl(6) | 2.250 (2) | Cl(3)—Cu(1)—Cl(4) | 127.9 (1) |
| Cu(2)—Cl(7) | 2.260 (2) | Cl(5)—Cu(2)—Cl(6) | 97.4 (1) |
| Cu(2)—Cl(8) | 2.235 (2) | Cl(5)—Cu(2)—Cl(7) | 134.0 (1) |
| S(1)—C(1) | 1.780 (6) | Cl(6)—Cu(2)—Cl(7) | 97.9 (1) |
| S(1)—C(2) | 1.785 (8) | Cl(5)—Cu(2)—Cl(8) | 99.4 (1) |
| S(1)—C(3) | 1.773 (6) | Cl(6)—Cu(2)—Cl(8) | 136.1 (1) |
| S(2)—C(4) | 1.749 (6) | Cl(7)—Cu(2)—Cl(8) | 98.9 (1) |
| S(2)—C(5) | 1.775 (8) | C(1)—S(1)—C(2) | 101.2 (4) |
| S(2)—C(6) | 1.766 (11) | C(1)—S(1)—C(3) | 101.1 (3) |
| S(3)—C(7) | 1.781 (6) | C(2)—S(1)—C(3) | 100.8 (3) |
| S(3)—C(8) | 1.785 (6) | C(4)—S(2)—C(5) | 102.3 (3) |
| S(3)—C(9) | 1.779 (6) | C(4)—S(2)—C(6) | 102.0 (4) |
| S(4)—C(10) | 1.776 (6) | C(5)—S(2)—C(6) | 101.6 (5) |
| S(4)—C(11) | 1.789 (6) | C(7)—S(3)—C(8) | 101.3 (3) |
| S(4)—C(12) | 1.770 (6) | C(7)—S(3)—C(9) | 101.7 (3) |
| | | C(8)—S(3)—C(9) | 101.1 (3) |
| | | C(10)—S(4)—C(11) | 101.8 (4) |
| | | C(10)—S(4)—C(12) | 101.5 (3) |
| | | C(11)—S(4)—C(12) | 101.6 (3) |

Table 6. S...Cl interaction distances (\AA) and angles ($^\circ$)

| | | | |
|---|-------|------------------|-------|
| $[(\text{CH}_3)_3\text{S}]_2\text{CuCl}_4$ | | | |
| S(1)—Cl(7) | 3.585 | C(1)—S(1)—Cl(7) | 177.5 |
| S(1)—Cl(6) | 3.563 | C(3)—S(1)—Cl(6) | 176.7 |
| S(1)—Cl(4) | 3.591 | C(2)—S(1)—Cl(4) | 161.2 |
| S(2)—Cl(6) | 3.759 | C(6)—S(2)—Cl(6) | 172.4 |
| S(2)—Cl(7) | 4.070 | C(4)—S(2)—Cl(7) | 153.3 |
| S(2)—Cl(8) | 4.022 | C(5)—S(2)—Cl(8) | 166.2 |
| S(3)—Cl(2) | 3.630 | C(8)—S(3)—Cl(2) | 175.6 |
| S(3)—Cl(3) | 3.440 | C(7)—S(3)—Cl(3) | 169.2 |
| S(3)—Cl(4) | 3.657 | C(9)—S(3)—Cl(4) | 172.9 |
| S(4)—Cl(6) | 3.846 | C(12)—S(4)—Cl(6) | 125.4 |
| S(4)—Cl(5) | 3.658 | C(10)—S(4)—Cl(5) | 179.7 |
| S(4)—Cl(5) | 3.730 | C(11)—S(4)—Cl(5) | 172.4 |
| $[(\text{CH}_3)_3\text{S}]_2\text{Cu}_2\text{Cl}_6$ | | | |
| S—Cl(2) | 3.839 | C(1)—S—Cl(2) | 170.1 |
| S—Cl(1) | 3.660 | C(2)—S—Cl(1) | 171.7 |
| S—Cl(2) | 4.003 | C(3)—S—Cl(2) | 165.2 |

Fig. 1. Illustration of the dimer stacks in $[(\text{CH}_3)_3\text{S}]_2\text{Cu}_2\text{Cl}_6$.

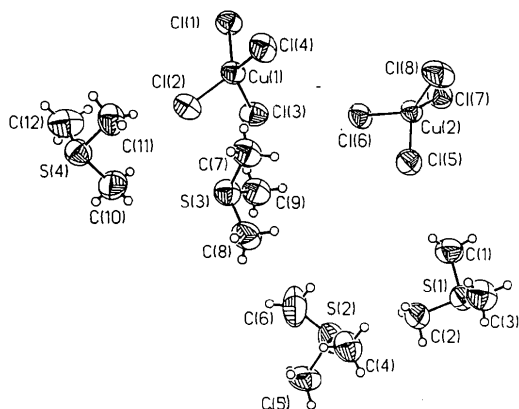


Fig. 2. Illustration of the asymmetric unit in [(CH₃)₃S]₂CuCl₄.

The repulsive S···Cu interaction between two positive species in [(CH₃)₃S]₂Cu₂Cl₆ can be rationalized, based on the structural characteristics observed for the S···Cl interactions in [(CH₃)₃S]₂CuCl₄. A search of the environs of the S atom in [(CH₃)₃S]₂Cu₂Cl₆ reveals numerous short S···Cl interactions, the three shortest of which are correctly positioned so as to define a trigonally distorted octahedral arrangement (Table 5). With two of these S···Cl interactions to Cl atoms on adjacent dimers within a stack, the S atom is located so as to effectively block the sixth octahedral coordination site for the Cu^{II} ion. The third S···Cl interaction is to a Cl atom in an adjacent stack.

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Acta Cryst. (1992). **C48**, 1192–1197

Tetraaquabis(hydrogen maleato)zinc(II) by Neutron Diffraction and Tetraaquabis(hydrogen maleato)nickel(II) by High-Order X-ray Diffraction

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Abstract. Neutron intensities ($\sin\theta/\lambda \leq 0.5 \text{ \AA}^{-1}$) of zinc bis(hydrogen maleate) tetrahydrate,

Zn(C₄H₃O₄)₂·4H₂O, and X-ray intensities ($\sin\theta/\lambda \leq 1.1 \text{ \AA}^{-1}$) of nickel bis(hydrogen maleate) tetrahydrate, Ni(C₄H₃O₄)₂·4H₂O, were collected at room temperature (298 K). Zn salt: $M_r = 367.5$, triclinic, $P\bar{1}$, $a = 7.337(5)$, $b = 9.219(7)$, $c = 5.222(3) \text{ \AA}$, $\alpha =$

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