

Table 4. Torsion angles ($^{\circ}$) for complexes (1) and (2)

	Complex (1)	Complex (2)
C(4)—O(3)—C(2)—C(1)	173.6 (6)	-166 (1)
C(2)—O(3)—C(4)—C(5)	170.0 (6)	-174 (1)
C(7)—O(6)—C(5)—C(4)	-168.2 (8)	164 (1)
C(5)—O(6)—C(7)—C(8)	162.4 (8)	-165 (1)
C(10)—O(9)—C(8)—C(7)	164.5 (6)	-174 (1)
C(8)—O(9)—C(10)—C(11)	-167.6 (7)	177 (1)
C(13)—O(12)—C(11)—C(10)	-167.6 (6)	168 (1)
C(11)—O(12)—C(13)—C(14)	167.4 (6)	-164 (1)
C(14)—O(15)—C(11)—C(2)	-149.5 (7)	141 (1)
C(1)—O(15)—C(14)—C(13)	174.4 (6)	-177 (1)
O(15)—C(1)—C(2)—O(3)	2.5 (9)	1 (1)
O(3)—C(4)—C(5)—O(6)	58.8 (9)	-62 (1)
O(6)—C(7)—C(8)—O(9)	-68.9 (8)	61 (1)
O(9)—C(10)—C(11)—O(12)	68.1 (8)	-60 (1)
O(12)—C(13)—C(14)—O(15)	-61.3 (8)	67 (1)

Torsion angles for both compounds are given Table 4: the molecular conformation is essentially the same as that observed in the complex $\text{NaI}(\text{benzo-15-crown-5})\cdot\text{H}_2\text{O}$ (Bush & Truter, 1972) and is best described as a sequence of ± 64 (4) and ± 60 (2) $^{\circ}$ for the aliphatic C—C bond [except for the C—C bond linked to the aromatic ring: 2.5 (9) and 1 (1) $^{\circ}$] and ± 166 (6) and 167 (7) $^{\circ}$ for the C—O—C—C torsion

angles, for (1) and (2) respectively. The five O atoms are not quite coplanar.

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Structure of μ -[Cystamine-*N,N,N',N'*-tetraacetato(4-)]-bis(5-methylimidazole)-dicopper(II) Dihydrate

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Abstract. μ -[(Dithiodi-2,1-ethanediy)dinitrilo-tetraacetato(4-)-*N,O,O'',S:N',O''''',O''''''',S'*]-bis-[(5-methylimidazole-*N*³)copper(II)] dihydrate,

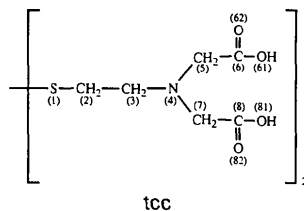
$[\text{Cu}_2(\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_8\text{S}_2)(\text{C}_4\text{H}_6\text{N}_2)_2]\cdot 2\text{H}_2\text{O}$, $M_r = 707.7$, monoclinic, $C2/c$, $a = 22.503$ (5), $b = 7.491$ (2), $c = 17.367$ (2) Å, $\beta = 110.76$ (1) $^{\circ}$, $V = 2737$ (2) Å³, $Z =$

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4, $D_m = 1.70$, $D_x = 1.72 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.7107 \text{ \AA}$, $\mu = 17.67 \text{ cm}^{-1}$, $F(000) = 1456$, $T = 295 (1) \text{ K}$, final $R = 0.030$ for 1647 observed reflections. The structure consists of dinuclear units with twofold internal symmetry. The Cu^{II} coordination geometry is slightly distorted square-pyramidal (type 4 + 1) with each Cu atom coordinated by one 5-methylimidazole N atom [Cu—N = 1.947 (3) Å], one N atom [Cu—N = 2.009 (3) Å], two O atoms [average Cu—O = 1.944 (3) Å] and one S atom [Cu—S = 2.798 (1) Å] from the same bis(carboxymethyl)cystaminato moiety. The S atom occupies the apical coordination site. Water molecules are linked together by intermolecular hydrogen bonds and each water molecule bridges three dimer entities.

Introduction. In a previous paper (Nguyen-Huy, Viossat, Busnot, Gonzalez-Pérez, Niclos-Gutiérrez, 1985), we reported the crystal structure of a Cu^{II} complex of the *N,N,N',N'*-tetrakis(carboxymethyl)cystaminato(4-) ion, [—SC₂H₄N(CH₂CO₂⁻)₂]₂ (tcc), having the formula [Cu₂(tcc)(H₂O)₂].4H₂O (I). Continuing the program on mixed-ligand Cu^{II} complexes (Gonzalez-Pérez, Nguyen-Huy, Niclos-Gutiérrez, Viossat, Busnot & Vicente Gelabert, 1989; Nguyen-Huy, Viossat, Busnot, Gonzalez-Pérez, Niclos-Gutiérrez & Gardette, 1990; Nguyen-Huy, Viossat, Busnot, Sicilia Zafra, Gonzalez-Pérez & Niclos-Gutiérrez, 1990) the compound (II) [Cu₂(tcc)(ImH)₂·(H₂O)₂].H₂O (Gonzalez-Pérez *et al.*, 1989) was studied, where ImH represents imidazole. The present work deals with an analog of (II), in which ImH is replaced by 5-methylimidazole (5-MeImH).



Experimental. The title compound [Cu₂(tcc)(5-MeImH)₂].2H₂O (III) can be obtained by the two procedures reported for (II) (Gonzalez-Pérez *et al.*, 1989) by employing 5-MeImH instead of ImH. The yield can reach 80%. Analysis calculated for C₂₀H₃₂Cu₂N₆O₁₀S₂: C 33.94; H 4.56; N 11.87; S 9.06; Cu 17.96%. Analysis found: C 33.62; H 4.53; N 11.85; S 9.01; Cu 17.40%. Both procedures yield well shaped parallelepiped crystals of (III) suitable for X-ray diffraction studies.

A crystal of size 0.25 × 0.32 × 0.38 mm was used for data collection. Density was determined by flotation in C₂H₄Br₂/CCl₄. Refined unit-cell parameters were obtained from setting angles of 25 reflections with 14.50 ≤ θ ≤ 15° on an Enraf-Nonius CAD-4

diffractometer with graphite-monochromated Mo Kα radiation. 2391 independent reflections with 2 ≤ 2θ ≤ 50° ($h = -26 \rightarrow 24$, $k = 0 \rightarrow 8$, $l = 0 \rightarrow 20$) were measured using the θ -2 θ -scan technique with scan width $s = (1.0 + 0.345 \tan \theta)^\circ$. 1647 unique reflections were observed for which $I > 3\sigma(I)$. $R_{\text{int}} = 0.024$. Two reference reflections (154 and 808) showed no significant variation in intensity. Lp correction was applied.

The structure was solved with the aid of MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refinement was by full-matrix least-squares calculations on F , minimizing $\sum w(F_o - |F_c|)^2$, initially with isotropic, and then anisotropic thermal parameters. H atoms were found from difference Fourier maps and were refined with an assigned isotropic thermal parameter U (0.054 Å²). All other atoms were refined anisotropically. Scattering factors including f' and f'' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were performed on a MicroVAX II computer using the CRYSTALS program system (Carruthers & Watkin, 1984). Final $R = 0.030$ and $wR = 0.032$, where $w = 1/\sigma^2(F_o)$. No extinction correction was applied. $S = 1.43$; maximum shift/e.s.d. = 0.10, maximum and minimum $\Delta\rho$ on the final difference Fourier map were 0.34 and -0.30 e Å⁻³. 230 parameters were refined. Fig. 1 was drawn using ORTEPII (Johnson, 1976).

Discussion. Positional parameters are given in Table 1.* Bond lengths and angles are collected in Table 2. Fig. 1 shows a view of the title complex with the atom labelling. As in compound (I), the two ligand moieties are related by the binary axis. This results in an asymmetric unit containing half a ligand, one imidazole molecule and one Cu ion.

The Cu^{II} atom is bonded to one S, two O and two N atoms in a distorted square-pyramidal coordination (type 4 + 1) (Fig. 1). The N(4), O(62) and O(82) atoms of the same chelating iminodiacetato group of the tcc ligand and the N(13) atom of 5-MeImH form the approximately square base of the pyramid and the S(1) (disulfide) atom of the same tcc half-molecule occupies the apical coordination site. The Cu^{II} atom is typically displaced from the mean plane $P(1)$ of the square base towards the fifth (S) donor atom (0.144 Å). The Cu—S bond defines an angle of

* Lists of structure factors, H-atom parameters, C—H, O—H and N—H distances, anisotropic thermal parameters and van der Waals distances as well as a stereoview packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55406 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU0311]

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Cu	0.12705 (2)	0.20268 (7)	0.36582 (3)	0.0276
S	0.02915 (5)	0.4542 (2)	0.31024 (7)	0.0367
O(1)	0.1148 (1)	0.2509 (4)	0.6060 (2)	0.0383
O(62)	0.1571 (1)	0.3263 (4)	0.4712 (2)	0.0318
O(61)	0.2391 (2)	0.4871 (5)	0.5487 (2)	0.0486
O(82)	0.1204 (1)	0.0827 (4)	0.2640 (2)	0.0348
O(81)	0.1760 (2)	0.0528 (5)	0.1814 (2)	0.0434
N(4)	0.1786 (1)	0.3834 (5)	0.3311 (2)	0.0249
N(11)	0.0083 (2)	-0.1422 (5)	0.4232 (2)	0.0343
N(13)	0.0812 (2)	0.0229 (5)	0.4037 (2)	0.0310
C(2)	0.0914 (2)	0.6066 (7)	0.3083 (3)	0.0387
C(3)	0.1387 (2)	0.5223 (6)	0.2757 (3)	0.0316
C(5)	0.2243 (2)	0.4623 (7)	0.4079 (3)	0.0309
C(6)	0.2062 (2)	0.4267 (6)	0.4825 (2)	0.0332
C(7)	0.2091 (2)	0.2731 (7)	0.2846 (3)	0.0320
C(8)	0.1650 (2)	0.1241 (6)	0.2379 (2)	0.0302
C(12)	0.0202 (2)	-0.0122 (6)	0.3779 (3)	0.0346
C(14)	0.1090 (2)	-0.0911 (6)	0.4679 (3)	0.0343
C(15)	0.0644 (2)	-0.1942 (6)	0.4815 (2)	0.0332
C(16)	0.0698 (3)	-0.3386 (8)	0.5420 (3)	0.0517

$$U_{\text{eq}} = \frac{1}{3} \text{trace } U.$$

Table 2. Bond lengths (\AA) and angles ($^\circ$)

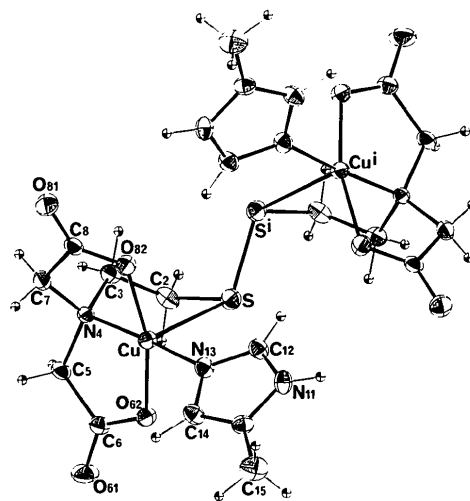
Cu—S	2.798 (1)	Cu—O(62)	1.946 (3)
Cu—O(82)	1.941 (3)	Cu—N(4)	2.009 (3)
Cu—N(13)	1.947 (3)	S—C(2)	1.816 (5)
S—S'	2.038 (2)	O(61)—C(6)	1.213 (5)
O(62)—C(6)	1.294 (5)	O(81)—C(8)	1.217 (5)
O(82)—C(8)	1.277 (5)	N(4)—C(5)	1.488 (5)
N(4)—C(3)	1.483 (5)	N(11)—C(12)	1.338 (5)
N(4)—C(7)	1.482 (5)	N(13)—C(12)	1.311 (5)
N(11)—C(15)	1.365 (5)	C(2)—C(3)	1.512 (7)
N(13)—C(14)	1.367 (5)	C(7)—C(8)	1.522 (6)
C(5)—C(6)	1.513 (6)	C(15)—C(16)	1.482 (7)
C(14)—C(15)	1.353 (6)		
O(62)—Cu—S	88.44 (9)	O(82)—Cu—S	101.6 (1)
O(82)—Cu—O(62)	165.0 (1)	N(4)—Cu—S	84.9 (1)
N(4)—Cu—O(62)	85.2 (1)	N(4)—Cu—O(82)	84.5 (1)
N(13)—Cu—S	97.8 (1)	N(13)—Cu—O(62)	93.5 (1)
N(13)—Cu—O(82)	96.2 (1)	N(13)—Cu—N(4)	177.0 (1)
S—S—Cu	119.73 (5)	C(2)—S—Cu	84.9 (2)
C(2)—S—S'	101.6 (2)	C(6)—O(62)—Cu	114.3 (2)
C(8)—O(82)—Cu	113.2 (3)	C(3)—N(4)—Cu	112.8 (2)
C(5)—N(4)—Cu	106.7 (2)	C(5)—N(4)—C(3)	111.7 (3)
C(7)—N(4)—Cu	102.4 (3)	C(7)—N(4)—C(3)	109.1 (3)
C(7)—N(4)—C(5)	113.7 (3)	C(15)—N(11)—C(12)	108.6 (4)
C(12)—N(13)—Cu	129.8 (3)	C(14)—N(13)—Cu	124.2 (3)
C(14)—N(13)—C(12)	106.0 (4)	C(3)—C(2)—S	113.3 (3)
C(2)—C(3)—N(4)	113.2 (4)	O(61)—C(6)—O(62)	124.2 (4)
C(5)—C(6)—O(62)	116.6 (3)	C(5)—C(6)—O(61)	119.2 (4)
C(8)—C(7)—N(4)	111.1 (3)	O(81)—C(8)—O(82)	126.1 (4)
C(7)—C(8)—O(82)	115.7 (4)	C(7)—C(8)—O(81)	118.1 (4)
N(13)—C(12)—N(11)	110.4 (4)	C(15)—C(14)—N(13)	110.2 (4)
C(14)—C(15)—N(11)	104.9 (4)	C(16)—C(15)—N(11)	123.7 (4)
C(6)—C(5)—N(4)	112.8 (3)	C(16)—C(15)—C(14)	131.4 (4)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

9.9° with the normal to the $P(1)$ plane. The length of this bond [2.798 (1) \AA] falls in the normal range for Cu—S (disulfide) bonds [Gonzalez-Pérez *et al.* (1989) and references herein] and the shortening of this bond from the homologous bond or interaction in the related compound (II) [2.959 (2) \AA on average] seems to result from the decrease in the coordination number from six in (II) to five in the present case (III). The Cu—N(5-MeImH) bond has a normal distance in view of the reported data for (II) and the other related compounds (Gonzalez-Pérez *et al.*,

1989; Nguyen-Hug, Viossat, Busnot, Gonzalez-Pérez *et al.*, 1990; Nguyen-Huy, Viossat, Busnot, Sicilia Zafra *et al.*, 1990; and references therein. In the new compound, the imidazole ligand plane and $P(1)$ define a large angle [67.0 (2)°] and the Cu—N(13) bond is slightly inclined *versus* the 5-MeImH plane (2.1°). Again, in spite of the near coplanarity of the 5-MeImH and of the fact that the Cu^{II} atom is displaced only 0.073 \AA , the referred ligand plays a σ -donor role without $d\pi-p\pi$ back donation.

In the studied compound (III), the tcc ligand plays the same bridging (dinucleating) and octadentate chelating roles as in its related compound (II). The internal geometry of the tcc ligand is substantially the same in both analogous compounds. Thus, in both cases, each iminodiacetato group of tcc forms two metal-glycinate rings with slightly asymmetrical envelope conformations; they are nearly coplanar [dihedral angles between mean planes of 171–172° in (II) and 162° in (III)]. The distance Cu—Cuⁱ [symmetry code: (i) $-x, y, \frac{1}{2} - z$] in the dinuclear unit of (III) [5.707 (1) \AA] is comparable to the corresponding Cu(1)—Cu(2) distance in (II) [5.970 (1) \AA], but these distances are both longer than the Cu—Cuⁱ distance in compound (I) [4.632 (2) \AA], this latter without imidazole ligands. As shown in Fig. 1, compound (III) consists of dinuclear units which are related by the binary axis. Water molecules, linked together by intermolecular hydrogen bonds, each bridge three dimer entities: *via* the two O—H bonds O(1)—H(O1)⋯O(62) [2.877 (4) \AA , 175 (5)°] and O(1)—H(O1)⋯O(81ⁱⁱ) [2.739 (5) \AA , 179 (6)°] and the N—H group of 5-MeImH, N(11)—H(N11)⋯O(1ⁱⁱⁱ) [2.757 (5) \AA , 176 (5)°] [symmetry code: (ii) $x, -y, \frac{1}{2} + z$; (iii) $-x, -y, 1 - z$]. This hydrogen-bonding

Fig. 1. ORTEPII (Johnson, 1976) view of the title complex with atom labelling [symmetry code: (i) $-x, y, \frac{1}{2} - y$].

network leads to layers which interact by van der Waals forces in the crystal.

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Acta Cryst. (1993). **C49**, 22–26

Geometry of the Iminium Moiety. III. Structures of a Chiral Form of Bis[4,5-dihydro-3,5,5-trimethyl-1-(3'-methylcyclopentylidene)pyrazolium] Hexachlorostannate and the Racemate

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Abstract. Compound (I) MPDL, bis[4,5-dihydro-3,5,5-trimethyl-1-(3'-methylcyclopentylidene)pyrazolium] hexachlorostannate, [C₁₂H₂₁N₂]₂⁺[SnCl₆]²⁻, *M_r* = 718.0, monoclinic, *P*2₁/*n*, *a* = 10.002 (2), *b* = 10.639 (1), *c* = 15.616 (4) Å, β = 102.05 (2)°, *V* = 1625.1 (2) Å³, *Z* = 2, *D_m* = 1.46 (1), *D_x* = 1.467 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 13.1 cm⁻¹, *F*(000) = 732, *T* = 294 K, *R* = 0.035 for 2291 observed reflections with *I* > 2σ(*I*). Compound (II) MPCH, bis[(*R*)-4,5-dihydro-3,5,5-trimethyl-1-(3'-methylcyclopentylidene)pyrazolium] hexachlorostannate, [C₁₂H₂₁N₂]₂⁺[SnCl₆]²⁻, *M_r* = 718.0, monoclinic, *P*2₁, *a* = 9.998 (2), *b* = 10.649 (1), *c* = 15.637 (4) Å, β = 102.03 (2)°, *V* = 1628.3 (3) Å³, *Z* = 2, *D_m* = 1.48 (1), *D_x* = 1.493 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 13.1 cm⁻¹, *F*(000) = 732, *T* = 294 K, *R* = 0.039 for 2436 observed reflections with *I* > 2σ(*I*). The compounds (I) and (II) are condensation products of a 4,5-dihydropyrazolium ion with a racemic and a chiral five-membered carbocyclic ketone respectively. Their structures are compared crystallographically, compound (II) showing evidence of disorder.

Introduction. In part II of this series (Nassimbeni, Stephen & Van Schalkwyk, 1991) the structures of four cycloalkylidene derivatives formed by condensations of several ketones and a pyrazolinium com-

pound are reported. In this paper, a comparative study is reported of the condensation products of this compound, bis(4,5-dihydro-3,5,5-trimethylpyrazolium) hexachlorostannate (PYRA), with the racemic and a chiral form of the ketone 3-methylcyclopentanone. The products are the iminium salts bis[4,5-dihydro-3,5,5-trimethyl-1-(3'-methylcyclopentylidene)pyrazolium] hexachlorostannate [(I) MPDL] and bis[(*R*)-4,5-dihydro-3,5,5-trimethyl-1-(3'-methylcyclopentylidene)pyrazolium] hexachlorostannate [(II) MPCH].

Experimental. A mixture of PYRA in dry ethanol (25% w/v) and excess (> 20 molar proportions) of either the racemic or the *R*(+) form of 3-methylcyclopentanone produced, after at least a day, stable prismatic crystals in small circular clusters. Crystal-density measurements were made by flotation in mixtures of bromoform and bromobenzene and by the use of a Paar DMA 35 density meter. The two compounds have almost identical unit-cell parameters. The space-group assignment was established photographically by taking long exposures of the zero and first layer photographs about *b* for both compounds. The photographs for compound (II) yielded a significant number of weak reflections which violated the *n*-glide condition *h* + *l* = 2*n*. This established the space group for compound (II) as *P*2₁, a subgroup of *P*2₁/*n*, which is the space group of (I). In addition, the reflections for the

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