

C19	0.3116 (2)	0.3830 (2)	-0.3385 (3)	0.065 (8)
C11†	0.357 (2)	0.670 (3)	0.433 (4)	0.59 (12)
C12†	0.301 (2)	0.623 (2)	0.375 (4)	0.49 (8)
C20	0.396 (2)	0.772 (4)	0.485 (4)	0.26 (8)

† Occupancy factor = 0.5.

Table 2. Selected geometric parameters (Å, °)

Cu1—Cu2	2.460 (2)	N1—C1	1.327 (8)
Cu1—N1	2.020 (5)	C1—C8	1.524 (6)
Cu2—N2	2.027 (4)	C1—N2	1.335 (8)
C2—N1	1.453 (6)	N2—C14	1.439 (6)
N1—Cu1—Cu2	86.43 (10)	C2—N1—C1	121.9 (4)
Cu1—Cu2—N2	86.54 (9)	N1—C1—C8	120.3 (5)
Cu2—N2—C1	119.7 (4)	C8—C1—N2	119.6 (5)
N2—C1—N1	120.1 (5)	C14—N2—C1	122.4 (4)
C1—N1—Cu1	120.5 (4)	C14—N2—Cu2	117.6 (3)
C2—N1—Cu1	117.0 (3)		

Weissenberg photographs taken using Cu *K*α radiation ( $\lambda = 1.542 \text{ \AA}$ ) indicated Laue symmetry *4/m*. Difficulty was experienced in measuring the crystal density by flotation; wide variations in the density of different crystals and density values changing with the time of immersion caused problems. The explanation was undoubtedly that solvent of crystallization, revealed later in the investigation to be present, was undergoing replacement in the immersion liquid. Intensity measurements were made with a two-circle diffractometer employing equi-inclination Weissenberg geometry. The angle  $\theta'$  is the projection of  $\theta$  onto the equatorial plane. The crystal was set to rotate about the *a* axis. A separate standard was required for each layer measured, but because of the equivalence of the *a* and *b* axes in the tetragonal system, refinement of interlayer scale factors was unnecessary. Refinement was carried out with phenyl groups constrained as rigid hexagons having individual  $U_{ij}$  parameters for the C atoms and H atoms at calculated sites with a common  $U_{iso}$ . A difference map showed a broad region of electron density around the fourfold alternating axis which was interpreted as disordered CH<sub>2</sub>Cl<sub>2</sub>.

Data collection: Stoe Stadi-2 software. Data reduction: *DATR* (Small, 1977). Program(s) used to solve structure: *SHELX76* (Sheldrick, 1976). Program(s) used to refine structure: *SHELX76*. Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1005). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Bis[(2-hydroxyethyl)dimethylammonium] Tetrakis(μ-benzoato-O:O')bis[(benzoato-O)cuprate(II)]

URHO TURPEINEN, REIJO HÄMÄLÄINEN AND ILPO MUTIKAINEN

Department of Chemistry, University of Helsinki, PO Box 55, FIN-00014, Helsinki, Finland

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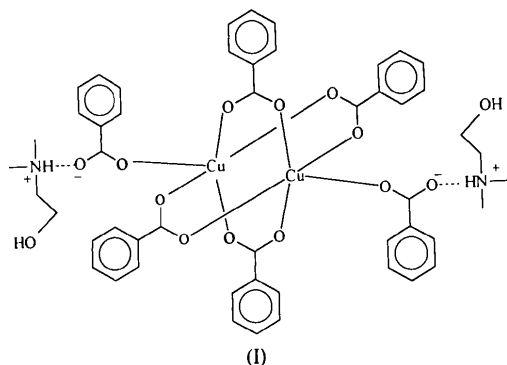
### Abstract

Copper(II) benzoate reacts with 2-dimethylaminoethanol in ethanol forming a green copper(II) complex, (C<sub>4</sub>H<sub>12</sub>NO)<sub>2</sub>[Cu<sub>2</sub>(C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>)<sub>6</sub>]. The two Cu atoms, with a Cu...Cu distance of 2.670 (2) Å in the centrosymmetric molecule, are held together by the four carboxylate groups. Each Cu atom is bound in a square-pyramidal configuration to five carboxylate O atoms. The (2-hydroxyethyl)dimethylammonium cation is hydrogen bonded to the uncoordinated O atom of the unidentate carboxylate group.

### Comment

Monomeric, tetrameric, hexameric and nonameric complexes have been obtained from reactions between 2-dimethylaminoethanol and Cu<sup>II</sup> carboxylates (Turpeinen, Hämäläinen & Ahlgrén, 1980, 1985; Turpeinen, Hämäläinen & Reedijk, 1987, 1988). Blue and green crystals precipitate simultaneously during the slow evaporation of an ethanol solution containing equimolar amounts of copper(II) benzoate and 2-dimethylaminoethanol. The structure of the blue complex is monomeric, the Cu atom being six-coordinate with two 2-dimethylaminoethanol and one benzoate group acting as bidentate ligands (Turpeinen, Hämäläinen & Ahlgrén, 1985). The structure of the green compound, (I), is a variant of the classical dimeric copper(II) acetate monohydrate compound (van Niekerk & Schoening, 1953).

The structure of (I) is composed of centrosymmetric [Cu<sub>2</sub>(C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>)<sub>6</sub>]<sup>2-</sup> anions and (2-hydroxyethyl)dimethylammonium cations joined together by



hydrogen bonds [N(1)··O(6) = 2.681(4) Å; N(1)—H(1A)··O(6) = 173°]. In the anion, the two Cu atoms are bridged by four carboxylate groups in a *syn-syn* configuration, with Cu—O bonds of 1.966(4)–1.976(3) Å. The other carboxylate groups are unidentate with Cu—O bonds of 2.117(4) Å. Each Cu atom is displaced from the basal plane of the four O atoms by 0.22(1) Å towards the apical O atoms. The Cu··Cu distance of 2.670(2) Å found in the present compound is close to the value of 2.610(1) Å observed in tetrakis( $\mu$ -benzoato-*O,O'*)bis(benzoic acid)dicopper(II), and is within the range reported for several dimeric copper(II) carboxylates (Kawata *et al.*, 1992; Porter & Doedens, 1984, and references therein).

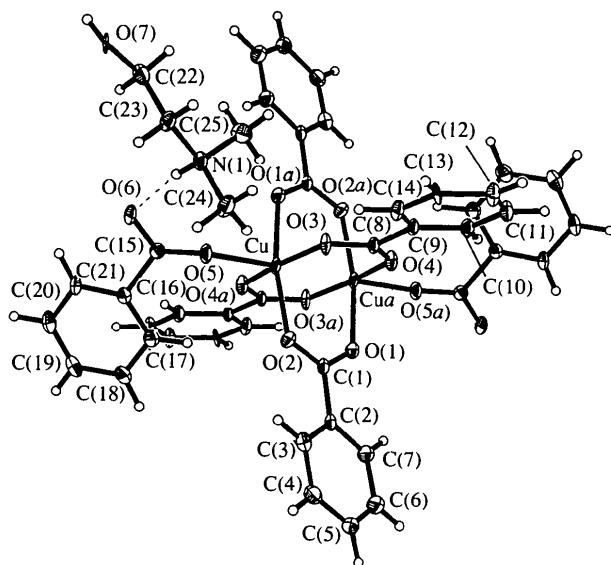


Fig. 1. View of the structure (SHELXTL/PC; Sheldrick, 1990). Displacement ellipsoids are drawn at the 20% probability level.

## Experimental

Blue and green crystals precipitated simultaneously during the slow evaporation of an ethanol solution containing equimolar amounts of copper(II) benzoate and 2-dimethylaminoethanol. The green crystals are of the title complex.

## Crystal data

(C<sub>4</sub>H<sub>12</sub>NO)<sub>2</sub>[Cu<sub>2</sub>(C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>)<sub>6</sub>]  
*M<sub>r</sub>* = 1034.0  
 Triclinic  
 P $\bar{1}$   
*a* = 9.597(4) Å  
*b* = 11.288(4) Å  
*c* = 11.984(4) Å  
 $\alpha$  = 92.28(3)°  
 $\beta$  = 109.58(3)°  
 $\gamma$  = 99.10(3)°  
*V* = 1201.7(8) Å<sup>3</sup>  
*Z* = 1  
*D<sub>x</sub>* = 1.429 Mg m<sup>-3</sup>

Mo *K*α radiation

$\lambda$  = 0.71069 Å

Cell parameters from 25 reflections

$\theta$  = 7–14°

$\mu$  = 0.95 mm<sup>-1</sup>

*T* = 293 K

Irregular

0.30 × 0.25 × 0.20 mm

Green

## Data collection

Nicolet P3 diffractometer

$\omega$  scans

Absorption correction:

$\psi$  scan (North, Phillips & Mathews, 1968)

*T<sub>min</sub>* = 0.83, *T<sub>max</sub>* = 1.00

5250 measured reflections

5250 independent reflections

3489 observed reflections

[*F* > 6σ(*F*)]

$\theta_{\max}$  = 27°

*h* = 0 → 12

*k* = -14 → 14

*l* = -15 → 14

3 standard reflections

monitored every 200

reflections

intensity decay: < 2%

## Refinement

Refinement on *F*

*R* = 0.0480

*wR* = 0.0552

*S* = 1.18

3489 reflections

307 parameters

H-atom parameters not

refined

$w = 1/\sigma^2(F)$

( $\Delta/\sigma$ )<sub>max</sub> = 0.008

$\Delta\rho_{\max}$  = 0.51 e Å<sup>-3</sup>

$\Delta\rho_{\min}$  = -0.76 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from SHELXTL/PC

(Sheldrick, 1990)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
Cu	0.0663 (1)	0.0464 (1)	-0.0743 (1)	0.033 (1)
O(1)	0.0842 (4)	0.0787 (3)	0.1930 (3)	0.049 (1)
O(2)	0.1992 (4)	0.1519 (3)	0.0697 (3)	0.049 (1)
O(3)	0.1985 (4)	-0.0736 (3)	-0.0325 (3)	0.045 (1)
O(4)	0.0930 (4)	-0.1441 (3)	0.0973 (3)	0.044 (1)
O(5)	0.1553 (4)	0.1264 (3)	-0.1985 (3)	0.046 (1)
O(6)	0.1674 (5)	0.1701 (3)	-0.3729 (3)	0.053 (2)
O(7)	0.2908 (5)	-0.0853 (4)	-0.6485 (3)	0.069 (2)
N(1)	0.2848 (5)	-0.0317 (3)	-0.3449 (3)	0.043 (2)
C(1)	0.1875 (6)	0.1463 (4)	0.1709 (4)	0.037 (2)
C(2)	0.3048 (6)	0.2269 (4)	0.2719 (4)	0.040 (2)
C(3)	0.4316 (7)	0.2909 (4)	0.2585 (4)	0.049 (2)
C(4)	0.5452 (7)	0.3589 (5)	0.3540 (5)	0.055 (2)
C(5)	0.5322 (8)	0.3614 (5)	0.4650 (5)	0.060 (2)
C(6)	0.4047 (8)	0.2993 (5)	0.4800 (5)	0.065 (3)
C(7)	0.2913 (7)	0.2314 (5)	0.3850 (4)	0.052 (2)
C(8)	0.1857 (5)	-0.1444 (4)	0.0434 (4)	0.036 (2)
C(9)	0.2862 (5)	-0.2357 (4)	0.0716 (4)	0.035 (2)
C(10)	0.2736 (6)	-0.3166 (4)	0.1548 (4)	0.040 (2)
C(11)	0.3688 (7)	-0.3997 (4)	0.1826 (5)	0.051 (2)
C(12)	0.4784 (7)	-0.4013 (5)	0.1317 (5)	0.054 (2)

C(13)	0.4901 (7)	-0.3210 (5)	0.0492 (5)	0.055 (2)
C(14)	0.3953 (6)	-0.2385 (5)	0.0196 (4)	0.046 (2)
C(15)	0.1473 (5)	0.1964 (4)	-0.2767 (4)	0.037 (2)
C(16)	0.1149 (6)	0.3205 (4)	-0.2568 (4)	0.041 (2)
C(17)	0.1262 (6)	0.3674 (4)	-0.1445 (4)	0.043 (2)
C(18)	0.0972 (7)	0.4818 (5)	-0.1283 (5)	0.058 (2)
C(19)	0.0560 (9)	0.5486 (5)	-0.2218 (6)	0.076 (3)
C(20)	0.0474 (11)	0.5036 (6)	-0.3324 (6)	0.099 (4)
C(21)	0.0769 (9)	0.3902 (5)	-0.3502 (5)	0.071 (3)
C(22)	0.2192 (7)	-0.0853 (5)	-0.5629 (4)	0.054 (2)
C(23)	0.3415 (6)	-0.0512 (5)	-0.4451 (4)	0.044 (2)
C(24)	0.4138 (7)	0.0014 (5)	-0.2324 (4)	0.060 (2)
C(25)	0.1736 (8)	-0.1357 (5)	-0.3353 (5)	0.063 (3)

Table 2. Selected geometric parameters (Å, °)

Cu—O(2)	1.976 (3)	C(3)—C(4)	1.384 (7)
Cu—O(3)	1.966 (4)	C(4)—C(5)	1.377 (9)
Cu—O(5)	2.117 (4)	C(5)—C(6)	1.381 (10)
Cu—Cu <sup>1</sup>	2.670 (2)	C(6)—C(7)	1.380 (7)
Cu—O(1 <sup>1</sup> )	1.980 (3)	C(8)—C(9)	1.494 (7)
Cu—O(4 <sup>1</sup> )	1.976 (4)	C(9)—C(10)	1.399 (7)
O(1)—C(1)	1.260 (6)	C(9)—C(14)	1.390 (9)
O(2)—C(1)	1.259 (6)	C(10)—C(11)	1.385 (8)
O(3)—C(8)	1.258 (6)	C(11)—C(12)	1.383 (10)
O(4)—C(8)	1.263 (7)	C(12)—C(13)	1.388 (8)
O(5)—C(15)	1.240 (6)	C(13)—C(14)	1.378 (9)
O(6)—C(15)	1.263 (7)	C(15)—C(16)	1.508 (7)
O(7)—C(22)	1.413 (8)	C(16)—C(17)	1.390 (7)
N(1)—C(23)	1.497 (8)	C(16)—C(21)	1.376 (7)
N(1)—C(24)	1.477 (6)	C(17)—C(18)	1.383 (8)
N(1)—C(25)	1.492 (8)	C(18)—C(19)	1.359 (9)
C(1)—C(2)	1.495 (5)	C(19)—C(20)	1.371 (11)
C(2)—C(3)	1.375 (8)	C(20)—C(21)	1.377 (10)
C(2)—C(7)	1.404 (8)	C(22)—C(23)	1.491 (6)
O(2)—Cu—O(3)	89.6 (1)	C(4)—C(5)—C(6)	120.1 (5)
O(2)—Cu—O(5)	98.2 (1)	C(5)—C(6)—C(7)	120.5 (6)
O(3)—Cu—O(5)	96.8 (2)	C(2)—C(7)—C(6)	119.7 (6)
O(2)—Cu—Cu <sup>1</sup>	83.3 (1)	O(3)—C(8)—O(4)	124.5 (5)
O(3)—Cu—Cu <sup>1</sup>	87.8 (1)	O(3)—C(8)—C(9)	118.0 (5)
O(5)—Cu—Cu <sup>1</sup>	175.2 (1)	O(4)—C(8)—C(9)	117.5 (4)
O(2)—Cu—O(1 <sup>1</sup> )	167.2 (2)	C(8)—C(9)—C(10)	119.4 (5)
O(3)—Cu—O(1 <sup>1</sup> )	88.0 (1)	C(8)—C(9)—C(14)	120.8 (4)
O(5)—Cu—O(1 <sup>1</sup> )	94.6 (1)	C(10)—C(9)—C(14)	119.7 (5)
O(2)—Cu—O(4 <sup>1</sup> )	91.6 (2)	C(9)—C(10)—C(11)	119.3 (5)
O(3)—Cu—O(4 <sup>1</sup> )	166.8 (2)	C(10)—C(11)—C(12)	120.8 (5)
O(5)—Cu—O(4 <sup>1</sup> )	96.0 (2)	C(11)—C(12)—C(13)	119.5 (6)
C(1)—O(1)—Cu <sup>1</sup>	123.1 (3)	C(12)—C(13)—C(14)	120.4 (6)
Cu—O(2)—C(1)	124.3 (3)	C(9)—C(14)—C(13)	120.2 (5)
Cu—O(3)—C(8)	119.4 (4)	O(5)—C(15)—O(6)	123.2 (5)
C(8)—O(4)—Cu <sup>1</sup>	128.8 (3)	O(5)—C(15)—C(16)	119.3 (5)
Cu—O(5)—C(15)	149.4 (4)	O(6)—C(15)—C(16)	117.4 (4)
C(23)—N(1)—C(24)	109.4 (4)	C(15)—C(16)—C(17)	121.0 (4)
C(23)—N(1)—C(25)	113.8 (4)	C(15)—C(16)—C(21)	120.0 (5)
C(24)—N(1)—C(25)	111.8 (4)	C(17)—C(16)—C(21)	119.0 (5)
O(1)—C(1)—O(2)	124.9 (4)	C(16)—C(17)—C(18)	120.0 (5)
O(1)—C(1)—C(2)	117.9 (4)	C(17)—C(18)—C(19)	120.5 (6)
O(2)—C(1)—C(2)	117.1 (4)	C(18)—C(19)—C(20)	119.7 (6)
C(1)—C(2)—C(3)	121.3 (5)	C(19)—C(20)—C(21)	120.7 (6)
C(1)—C(2)—C(7)	119.7 (5)	C(16)—C(21)—C(20)	120.1 (6)
C(3)—C(2)—C(7)	118.8 (4)	O(7)—C(22)—C(23)	106.2 (5)
C(2)—C(3)—C(4)	121.4 (6)	N(1)—C(23)—C(22)	113.5 (5)
C(3)—C(4)—C(5)	119.4 (6)		

Symmetry code: (i)  $-x, -y, -z$ .

All non-H atoms were refined anisotropically. H atoms attached to C and N atoms were introduced in calculated positions and refined with fixed geometry with respect to their carrier atoms. The H atom of the alcoholic group was located from a  $\Delta F$  map.

Data collection and cell refinement: Nicolet P3 diffractometer system. Structure solution, refinement, other calculations and graphics: *SHELXTL/PC* (Sheldrick, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1186). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Tribenzyltin Acetate, a Redetermination

GEORGE FERGUSON

*Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1*

TREVOR R. SPALDING, ANNA T. O'DOWD AND  
KAREN C. O'SHEA

*Department of Chemistry, University College, Cork, Ireland*

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## Abstract

This redetermination of the structure of tribenzyltin acetate, [Sn(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)(C<sub>7</sub>H<sub>7</sub>)<sub>3</sub>], has confirmed the structure determined previously from photographic data [Alcock & Timms (1968). *J. Chem. Soc. A*, pp. 1873–1876], but the molecular dimensions reported here are an order of magnitude more precise. The structure is polymeric with bridging unsymmetric acetate groups [Sn—O 2.1309 (15) and 2.5592 (16) Å, O—Sn—O 169.54 (5)°, and C—O 1.281 (3) and 1.239 (2) Å]. Distorted trigonal bipyramidal coordination at Sn is completed by three benzyl groups [Sn—C 2.139–2.161 (2) Å].