Acta Cryst E STRUCTURE REPORTS

OPEN ACCESS

ISSN 1600-5368

Crystal structure of bis{2-[(2-hydroxyethyl)amino]ethanol-κ^3O,N,O'}copper(II) terephthalate

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Received 29 September 2014; accepted 9 October 2014

Edited by M. Weil, Vienna University of Technology, Austria

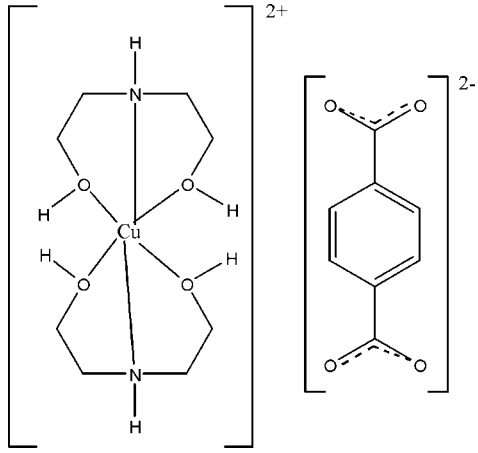
The molecular components of the title salt, $[Cu(C_4H_{11}NO_2)_2](C_8H_4O_4)$, are one Cu^{II} cation O,N,O'-chelated by two tridentate 2-[(2-hydroxyethyl)amino]ethanol ligands, and a terephthalate counter-dianion, located about a centre of inversion. The complex Cu^{II} cation is located about a centre of inversion and shows typical Jahn–Teller distortion, with two short Cu—O and two short Cu—N bonds in the equatorial plane and two long Cu—O bonds to the axial atoms. The cations are arranged in sheets parallel to (100), with the centrosymmetric terephthalate anions located between the sheets. Each anion is the acceptor of four O—H···O and two N—H···O hydrogen bonds, forming a three-dimensional network structure.

Keywords: crystal structure; copper(II) chelate complex; terephthalate.

CCDC reference: 1028231

1. Related literature

For related copper(II) compounds with terephthalate anions, see: Abbaszadeh *et al.* (2012); Al-Hashemi *et al.* (2010).



2. Experimental

2.1. Crystal data

$[Cu(C_4H_{11}NO_2)_2](C_8H_4O_4)$
M_r = 437.93
Monoclinic, $P2_1/c$
a = 8.6013 (9) Å
b = 9.0398 (9) Å
c = 11.5732 (12) Å
β = 91.695 (2)°
V = 899.47 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.26 mm^{-1}
T = 293 K
0.29 × 0.27 × 0.26 mm

2.2. Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.728, T_{max} = 0.812
4784 measured reflections
1780 independent reflections
1611 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.080
S = 1.08
1780 reflections
133 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.85 (2)	2.08 (2)	2.9196 (19)	168 (2)
O4—H4A···O2^{ii}	0.84 (2)	1.66 (2)	2.496 (2)	180 (3)
O3—H3A···O1^{iii}	0.81 (2)	1.88 (2)	2.6803 (19)	167 (2)

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+2, -y+1, -z+2$.

Data collection: *APEX2* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

Acknowledgements

This project was supported by the International Scientific and Technological Cooperation Foundation of Jilin Province (grant No. 20120722).

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5069).

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supporting information

Acta Cryst. (2014). E**70**, m372–m373 [doi:10.1107/S1600536814022272]

Crystal structure of bis{2-[(2-hydroxyethyl)amino]ethanol-κ^3O,N,O'}copper(II) terephthalate

Ya-Ping Li, Dajun Sun, Julia Ming, Liying Han and Guan-Fang Su

S1. Preparation

The synthesis was performed under hydrothermal conditions. A mixture of $Cu(CH_3COO)_2{\cdot}2H_2O$, (0.2 mmol, 0.046 g), 2-(2-hydroxy-ethylamine) (0.4 mmol, 0.043 g), sodium terephthalate (0.2 mmol, 0.042 g) and water (20 ml) in a 30 ml stainless steel reactor with a Teflon liner were heated from 293 to 433 K in 2 h, and a constant temperature was maintained at 433 K for 72 h, after which the mixture was cooled to 298 K. Blue crystals of the title compound were recovered from the reaction.

S2. Refinement

All C—H H atoms were positioned with idealized geometry and refined with U_{iso}(H) = 1.2U_{eq}(C) using a riding model. The hydroxy H-atoms and amine H atoms were located in a difference Fourier map and were refined with O—H or N—H distances restrained to 0.85 (3) Å and with U_{iso}(H) = 1.5U_{eq}(N,O).

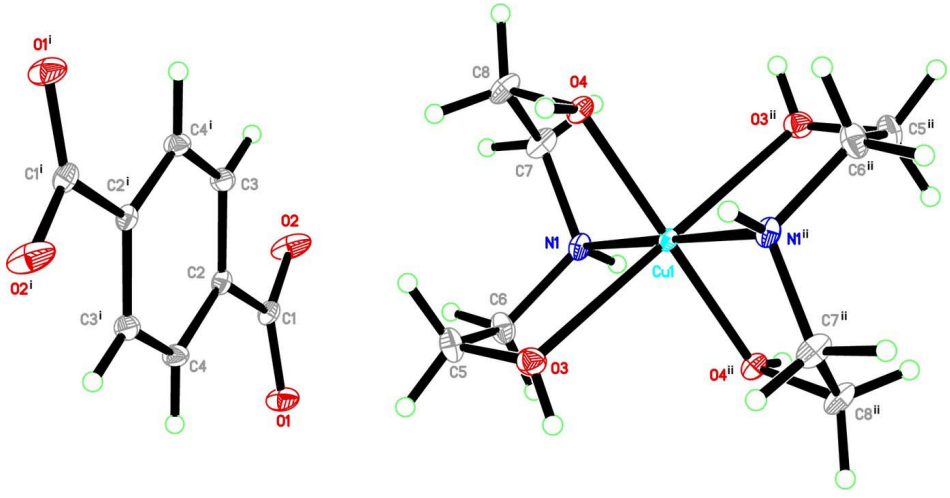

Figure 1

The molecular components of the title compound. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) 3-x, 1-y, 2-z; (ii) 2-x, -y, 2-z.]

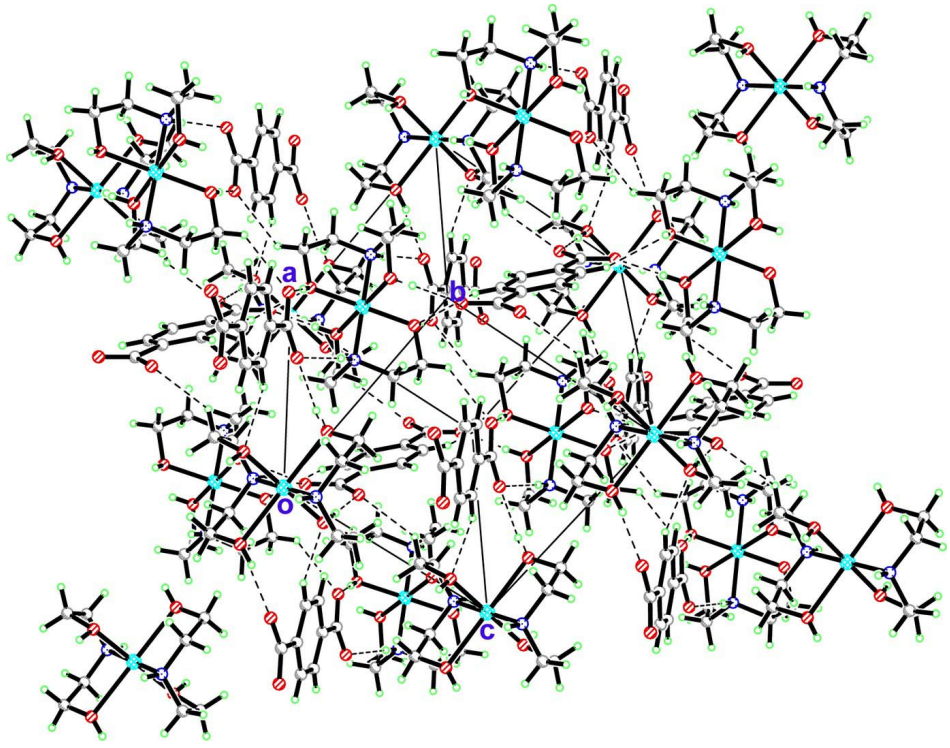

Figure 2

The packing of the molecular components in the title compound. N—H···O and O—H···O hydrogen bonds are shown by dashed lines.

Bis{2-[(2-hydroxyethyl)amino]ethanol-κ^3O,N,O'}copper(II) terephthalate

Crystal data

$[Cu(C_4H_{11}NO_2)_2](C_8H_4O_4)$
$M_r = 437.93$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.6013$ (9) Å
$b = 9.0398$ (9) Å
$c = 11.5732$ (12) Å
$\beta = 91.695$ (2)°
$V = 899.47$ (16) Å^3
$Z = 2$
$F(000) = 458$
$D_x = 1.617$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1780 reflections
$\theta = 1.7$–22.8°
$\mu = 1.26$ mm^{-1}
$T = 293$ K
Block, blue
0.29 × 0.27 × 0.26 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.728$, $T_{max} = 0.812$
4784 measured reflections
1780 independent reflections
1611 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 26.1°$, $\theta_{min} = 2.4°$
$h = -10 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.080$
$S = 1.08$
1780 reflections
133 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.3198P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.83$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.3193 (2)	0.5638 (2)	0.79022 (15)	0.0172 (4)
C2	1.4142 (2)	0.5319 (2)	0.89901 (16)	0.0152 (4)
C3	1.5213 (2)	0.4163 (2)	0.90128 (16)	0.0182 (4)
H3	1.5359	0.3603	0.8351	0.022*
C4	1.3941 (2)	0.6154 (2)	0.99805 (15)	0.0181 (4)
H4	1.3232	0.6932	0.9968	0.022*
C5	1.1185 (2)	0.3121 (2)	1.02670 (17)	0.0256 (4)
H5A	1.2286	0.2892	1.0319	0.031*
H5B	1.1057	0.4155	1.0468	0.031*
C6	1.0583 (2)	0.2870 (2)	0.90354 (17)	0.0235 (4)
H6A	0.9534	0.3259	0.8956	0.028*
H6B	1.1229	0.3417	0.8511	0.028*
C7	1.2043 (2)	0.0697 (2)	0.82537 (15)	0.0233 (4)
H7A	1.2537	0.1440	0.7784	0.028*
H7B	1.1821	−0.0158	0.7769	0.028*
C8	1.3139 (2)	0.0254 (2)	0.92433 (18)	0.0229 (4)
H8A	1.3931	−0.0412	0.8968	0.027*
H8B	1.3649	0.1123	0.9568	0.027*
N1	1.05698 (17)	0.12937 (17)	0.86951 (12)	0.0159 (3)
H1A	0.985 (2)	0.123 (2)	0.8179 (16)	0.024*
O1	1.20598 (16)	0.64876 (16)	0.79517 (11)	0.0258 (3)
O2	1.3607 (2)	0.49850 (17)	0.70051 (13)	0.0334 (4)
O3	1.03789 (17)	0.22244 (15)	1.10678 (11)	0.0211 (3)
H3A	0.956 (2)	0.259 (3)	1.127 (2)	0.032*

O4	1.22477 (15)	−0.04666 (15)	1.01071 (11)	0.0163 (3)
H4A	1.270 (3)	−0.031 (3)	1.0742 (16)	0.025*
Cu1	1.0000	0.0000	1.0000	0.01196 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0139 (9)	0.0228 (10)	0.0149 (9)	−0.0016 (8)	−0.0017 (7)	0.0012 (7)
C2	0.0130 (9)	0.0170 (8)	0.0156 (8)	−0.0019 (8)	−0.0020 (7)	0.0018 (7)
C3	0.0173 (9)	0.0203 (9)	0.0168 (8)	0.0013 (7)	−0.0014 (7)	−0.0034 (7)
C4	0.0162 (9)	0.0189 (9)	0.0192 (9)	0.0040 (8)	−0.0023 (7)	−0.0008 (7)
C5	0.0279 (11)	0.0218 (10)	0.0268 (10)	−0.0081 (8)	−0.0029 (8)	−0.0039 (8)
C6	0.0283 (11)	0.0185 (9)	0.0235 (10)	−0.0043 (8)	−0.0035 (8)	0.0038 (8)
C7	0.0184 (10)	0.0378 (12)	0.0137 (8)	0.0013 (9)	0.0006 (7)	0.0045 (8)
C8	0.0140 (10)	0.0371 (11)	0.0176 (9)	0.0004 (8)	0.0027 (8)	0.0047 (8)
N1	0.0135 (8)	0.0210 (8)	0.0128 (7)	−0.0019 (6)	−0.0052 (6)	0.0016 (6)
O1	0.0223 (8)	0.0351 (8)	0.0195 (7)	0.0124 (6)	−0.0071 (5)	−0.0038 (6)
O2	0.0252 (9)	0.0597 (13)	0.0149 (7)	0.0192 (7)	−0.0050 (6)	−0.0049 (6)
O3	0.0216 (7)	0.0223 (7)	0.0194 (6)	0.0015 (6)	−0.0005 (5)	−0.0037 (5)
O4	0.0126 (6)	0.0238 (7)	0.0125 (6)	0.0002 (6)	−0.0023 (5)	0.0010 (5)
Cu1	0.01017 (19)	0.0143 (2)	0.01133 (18)	−0.00020 (10)	−0.00122 (12)	0.00137 (10)

Geometric parameters (Å, º)

C1—O1	1.243 (2)	C7—N1	1.482 (2)
C1—O2	1.255 (2)	C7—C8	1.515 (3)
C1—C2	1.508 (2)	C7—H7A	0.9700
C2—C4	1.387 (3)	C7—H7B	0.9700
C2—C3	1.392 (3)	C8—O4	1.434 (2)
C3—C4^{i}	1.386 (2)	C8—H8A	0.9700
C3—H3	0.9300	C8—H8B	0.9700
C4—C3^{i}	1.386 (2)	N1—Cu1	1.9830 (15)
C4—H4	0.9300	N1—H1A	0.847 (16)
C5—O3	1.427 (2)	O3—Cu1	2.3776 (13)
C5—C6	1.519 (3)	O3—H3A	0.814 (16)
C5—H5A	0.9700	O4—Cu1	1.9791 (13)
C5—H5B	0.9700	O4—H4A	0.835 (17)
C6—N1	1.478 (2)	Cu1—O4^{ii}	1.9791 (13)
C6—H6A	0.9700	Cu1—N1^{ii}	1.9830 (15)
C6—H6B	0.9700	Cu1—O3^{ii}	2.3776 (13)
O1—C1—O2	124.81 (17)	C7—C8—H8A	110.0
O1—C1—C2	119.04 (16)	O4—C8—H8B	110.0
O2—C1—C2	116.13 (16)	C7—C8—H8B	110.0
C4—C2—C3	119.43 (17)	H8A—C8—H8B	108.4
C4—C2—C1	120.51 (17)	C6—N1—C7	116.30 (16)
C3—C2—C1	120.05 (17)	C6—N1—Cu1	111.45 (11)
C4^{i}—C3—C2	120.13 (17)	C7—N1—Cu1	106.31 (11)

C4^{i}—C3—H3	119.9	C6—N1—H1A	104.5 (15)
C2—C3—H3	119.9	C7—N1—H1A	110.2 (15)
C3^{i}—C4—C2	120.43 (17)	Cu1—N1—H1A	107.9 (15)
C3^{i}—C4—H4	119.8	C5—O3—Cu1	101.82 (10)
C2—C4—H4	119.8	C5—O3—H3A	113.5 (18)
O3—C5—C6	111.45 (16)	Cu1—O3—H3A	112.5 (18)
O3—C5—H5A	109.3	C8—O4—Cu1	113.63 (11)
C6—C5—H5A	109.3	C8—O4—H4A	106.7 (18)
O3—C5—H5B	109.3	Cu1—O4—H4A	116.8 (18)
C6—C5—H5B	109.3	O4—Cu1—O4^{ii}	180.0
H5A—C5—H5B	108.0	O4—Cu1—N1^{ii}	95.14 (6)
N1—C6—C5	113.19 (15)	O4^{ii}—Cu1—N1^{ii}	84.86 (6)
N1—C6—H6A	108.9	O4—Cu1—N1	84.86 (6)
C5—C6—H6A	108.9	O4^{ii}—Cu1—N1	95.14 (6)
N1—C6—H6B	108.9	N1^{ii}—Cu1—N1	180.0
C5—C6—H6B	108.9	O4—Cu1—O3^{ii}	88.34 (5)
H6A—C6—H6B	107.8	O4^{ii}—Cu1—O3^{ii}	91.66 (5)
N1—C7—C8	110.78 (15)	N1^{ii}—Cu1—O3^{ii}	82.18 (5)
N1—C7—H7A	109.5	N1—Cu1—O3^{ii}	97.82 (5)
C8—C7—H7A	109.5	O4—Cu1—O3	91.66 (5)
N1—C7—H7B	109.5	O4^{ii}—Cu1—O3	88.34 (5)
C8—C7—H7B	109.5	N1^{ii}—Cu1—O3	97.82 (5)
H7A—C7—H7B	108.1	N1—Cu1—O3	82.18 (5)
O4—C8—C7	108.27 (16)	O3^{ii}—Cu1—O3	180.0
O4—C8—H8A	110.0		

O1—C1—C2—C4	−11.6 (3)	C8—O4—Cu1—N1	−1.66 (13)
O2—C1—C2—C4	169.71 (18)	C8—O4—Cu1—O3^{ii}	96.35 (13)
O1—C1—C2—C3	167.30 (18)	C8—O4—Cu1—O3	−83.65 (13)
O2—C1—C2—C3	−11.4 (3)	C6—N1—Cu1—O4	−103.85 (13)
C4—C2—C3—C4^{i}	0.4 (3)	C7—N1—Cu1—O4	23.81 (11)
C1—C2—C3—C4^{i}	−178.47 (17)	C6—N1—Cu1—O4^{ii}	76.15 (13)
C3—C2—C4—C3^{i}	−0.4 (3)	C7—N1—Cu1—O4^{ii}	−156.19 (11)
C1—C2—C4—C3^{i}	178.46 (17)	C6—N1—Cu1—N1^{ii}	5 (100)
O3—C5—C6—N1	−52.7 (2)	C7—N1—Cu1—N1^{ii}	133 (100)
N1—C7—C8—O4	41.4 (2)	C6—N1—Cu1—O3^{ii}	168.55 (12)
C5—C6—N1—C7	−85.3 (2)	C7—N1—Cu1—O3^{ii}	−63.79 (12)
C5—C6—N1—Cu1	36.8 (2)	C6—N1—Cu1—O3	−11.45 (12)
C8—C7—N1—C6	82.9 (2)	C7—N1—Cu1—O3	116.21 (12)
C8—C7—N1—Cu1	−41.82 (18)	C5—O3—Cu1—O4	69.87 (12)
C6—C5—O3—Cu1	37.08 (18)	C5—O3—Cu1—O4^{ii}	−110.13 (12)
C7—C8—O4—Cu1	−20.7 (2)	C5—O3—Cu1—N1^{ii}	165.29 (12)
C8—O4—Cu1—O4^{ii}	−53 (21)	C5—O3—Cu1—N1	−14.71 (12)
C8—O4—Cu1—N1^{ii}	178.34 (13)	C5—O3—Cu1—O3^{ii}	−40.8 (3)

Symmetry codes: (i) −*x*+3, −*y*+1, −*z*+2; (ii) −*x*+2, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{iii}	0.85 (2)	2.08 (2)	2.9196 (19)	168 (2)
O4—H4*A*···O2^{iv}	0.84 (2)	1.66 (2)	2.496 (2)	180 (3)
O3—H3*A*···O1^{v}	0.81 (2)	1.88 (2)	2.6803 (19)	167 (2)

Symmetry codes: (iii) −*x*+2, *y*−1/2, −*z*+3/2; (iv) *x*, −*y*+1/2, *z*+1/2; (v) −*x*+2, −*y*+1, −*z*+2.