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catena-Poly[[[aquacopper(II)]-μ-[(*E*)-2-(4-oxopentan-2-ylideneamino)benzoato-$\kappa^4O,N,O':O''$]] monohydrate]

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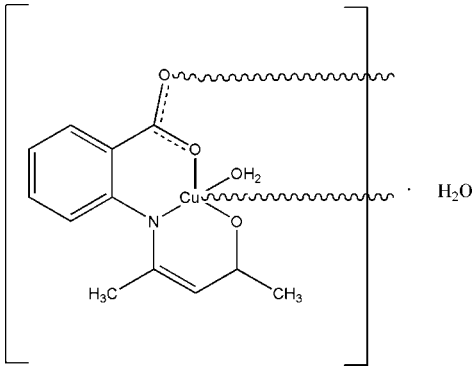
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; R factor = 0.047; wR factor = 0.124; data-to-parameter ratio = 17.4.

The ligand of the title compound, $\{[Cu(C_{12}H_{11}NO_2)(H_2O)]\cdot H_2O\}_n$, has been synthesized from *o*-aminobenzoic acid and acetyl acetone. Bond lengths and angles show normal values. The crystal structure is stabilized by several hydrogen bonds.

Related literature

For related literature, see: Plesch *et al.* (1991); Shi & Hu (2007).



Experimental

Crystal data

$[Cu(C_{12}H_{11}NO_2)(H_2O)]\cdot H_2O$
M_r = 316.79
Orthorhombic, *Pbca*
a = 8.1933 (7) Å
b = 8.8144 (7) Å
c = 35.209 (3) Å
V = 2542.7 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.73 mm^{-1}
T = 292 (2) K
0.20 × 0.10 × 0.02 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.723, T_{max} = 0.966
17406 measured reflections
3029 independent reflections
2209 reflections with $I > 2\sigma(I)$
R_{int} = 0.110

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.124
S = 0.99
3029 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5B···O4^{i}	0.87	2.45	3.264 (3)	156
O5—H5B···O1^{i}	0.87	2.51	3.227 (3)	140
O5—H5A···O1	0.93	1.91	2.815 (3)	162
O4—H4B···O5	0.79	2.49	3.208 (4)	150
O4—H4A···O5^{ii}	0.81	1.86	2.663 (3)	176

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2346).

References

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supplementary materials

Acta Cryst. (2007). E**63**, m1494 [**doi:10.1107/S1600536807019514**]

catena-Poly[[[aquacopper(II)]-μ-[(*E*)-2-(4-oxopentan-2-ylideneamino)benzoato-κ^4*O*,*N*,*O*':*O*'']] monohydrate]

M.-N. Cao, S.-M. Shi, F.-H. Luo, C.-X. Cheng and Z.-Q. Hu

Comment

The crystal structure and some properties of a substituted benzoate-copper(II) complex were previously reported by Plesch *et al.*, (1991) and Shi *et al.*, (2006).

In the title compound, each Cu atom is five-coordinated by one oxygen atom of a water molecule, one carboxylate O atom and one amino N atom of the ligand ((*E*)-2-(4-oxopentan-2-ylideneamino)-benzoato) and one μ_2-O atom from another ligand, forming a slightly distorted trigonal bipyramid (Fig. 1). The crystal structure is stabilized by several hydrogen bonds (Table 2).

Experimental

To an ethanol solution of *o*-aminobenzic acid (6.85 g, 0.1 mol), acetylacetone (5 g, 0.05 mol) was slowly added with continuous stirring at reflux for 5 h. The reaction mixture was cooled to room temperature, and the Schiff base ligand, *viz*. (*E*)-2-(4-oxopentan-2-ylideneamino)benzoic acid, precipitated after one night as a yellow solid. White crystals of the ligand were recrystallized from ethanol. 1 mmol of the Schiff base ligand, 0.5 mmol $Cu(OAc)_2$, dimethylformamide (15 ml) and 2 drops of triethylamine were stirred for 5 h at room temperature. The solution was filtered and allowed to stand at room temperature without disturbing, crystals of of the title compound were obtained after about 3 weeks.

Refinement

After having located them in a difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on their parent atoms with C—H = 0.93 Å, or C_{methyl}—H = 0.96 Å. The O—H distances were kept as initially found in the difference map U_{iso}(H) was set to 1.2U_{eq}(C,*O*) or U_{iso}(H) = 1.5U_{eq}(C_{methyl}). The methyl groups were allowed to rotate but not to tip.

Figures

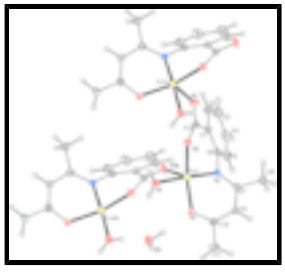


Fig. 1. Molecular structure of (I) showing 30% probability displacement ellipsoids. [Symmetry codes: (*a*) 1/2 − *x*, −1/2 + *y*, *z*; (*b*) 1/2 − *x*, 1/2 + *y*, *z*].

catena-Poly[[[aquacopper(II)]-μ-[(_E_)-2-(4-oxopentan-2-ylideneamino)benzoato- $\kappa^4O,N,O':O''$] monohydrate]

Crystal data

$[Cu(C_{12}H_{11}NO_2)(H_2O)]\cdot H_2O$	F_{000} = 1304
M_r = 316.79	D_x = 1.655 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 3438 reflections
a = 8.1933 (7) Å	θ = 2.7–25.1º
b = 8.8144 (7) Å	μ = 1.73 mm^{-1}
c = 35.209 (3) Å	*T* = 292 (2) K
V = 2542.7 (4) Å^3	Block, green
Z = 8	0.20 × 0.10 × 0.02 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3029 independent reflections
Radiation source: fine-focus sealed tube	2209 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.110
T = 292(2) K	θ_{max} = 28.0º
φ and ω scans	θ_{min} = 1.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	*h* = −10→10
T_{min} = 0.723, T_{max} = 0.966	*k* = −11→9
17406 measured reflections	*l* = −41→46

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H-atom parameters constrained
$wR(F^2)$ = 0.124	$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max} < 0.001$
3029 reflections	$\Delta\rho_{max}$ = 0.55 e Å^{-3}
174 parameters	$\Delta\rho_{min}$ = −0.62 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.06114 (4)	0.28463 (4)	0.410032 (10)	0.02286 (15)
C1	0.1120 (4)	0.0753 (4)	0.33769 (9)	0.0251 (7)
C2	0.1813 (4)	0.0274 (4)	0.30336 (10)	0.0377 (8)
H2	0.2658	−0.0430	0.3039	0.045*
C3	0.1284 (5)	0.0812 (5)	0.26910 (10)	0.0465 (10)
H3	0.1747	0.0459	0.2467	0.056*
C4	0.0047 (5)	0.1891 (4)	0.26804 (10)	0.0408 (9)
H4	−0.0288	0.2299	0.2450	0.049*
C5	−0.0676 (4)	0.2348 (4)	0.30134 (11)	0.0361 (8)
H5	−0.1515	0.3058	0.3005	0.043*
C6	−0.0183 (4)	0.1776 (4)	0.33641 (8)	0.0246 (7)
C7	−0.2511 (4)	0.2408 (3)	0.37425 (9)	0.0249 (7)
C8	−0.3701 (4)	0.1592 (4)	0.34817 (10)	0.0398 (9)
H8A	−0.4201	0.2312	0.3313	0.060*
H8B	−0.4528	0.1104	0.3631	0.060*
H8C	−0.3125	0.0843	0.3336	0.060*
C9	−0.3244 (4)	0.3229 (4)	0.40459 (9)	0.0276 (7)
H9	−0.4373	0.3162	0.4067	0.033*
C10	−0.2457 (4)	0.4110 (4)	0.43107 (9)	0.0257 (7)
C11	−0.3420 (4)	0.4982 (4)	0.45991 (9)	0.0320 (8)
H11A	−0.3248	0.4550	0.4846	0.048*
H11B	−0.4559	0.4933	0.4536	0.048*
H11C	−0.3070	0.6021	0.4599	0.048*
C12	0.1892 (4)	0.0166 (4)	0.37355 (9)	0.0257 (7)
N1	−0.0924 (3)	0.2318 (3)	0.37032 (7)	0.0230 (6)
O1	−0.0887 (2)	0.4241 (3)	0.43399 (7)	0.0326 (6)
O2	0.1718 (3)	0.0870 (2)	0.40485 (6)	0.0266 (5)
O3	0.2711 (3)	−0.1019 (3)	0.37077 (7)	0.0399 (6)
O4	0.2098 (3)	0.3404 (3)	0.45295 (7)	0.0393 (6)
H4A	0.2786	0.2819	0.4603	0.059*
H4B	0.2019	0.4185	0.4640	0.059*
O5	0.0519 (3)	0.6591 (3)	0.47715 (7)	0.0411 (6)
H5A	0.0032	0.5965	0.4590	0.062*
H5B	0.0082	0.6477	0.4995	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0211 (2)	0.0222 (2)	0.0253 (2)	0.00220 (15)	−0.00185 (15)	−0.00432 (15)
C1	0.0225 (15)	0.0237 (16)	0.0292 (18)	−0.0017 (13)	0.0015 (13)	−0.0027 (13)
C2	0.0340 (19)	0.043 (2)	0.036 (2)	0.0065 (16)	0.0054 (16)	−0.0098 (17)
C3	0.051 (2)	0.063 (3)	0.026 (2)	−0.005 (2)	0.0076 (17)	−0.0108 (19)
C4	0.052 (2)	0.049 (2)	0.0209 (18)	−0.0052 (19)	−0.0057 (16)	0.0011 (16)
C5	0.0362 (19)	0.038 (2)	0.034 (2)	−0.0001 (16)	−0.0036 (15)	0.0015 (16)
C6	0.0258 (16)	0.0248 (17)	0.0233 (17)	−0.0047 (13)	−0.0016 (12)	−0.0031 (13)
C7	0.0253 (15)	0.0214 (16)	0.0280 (17)	0.0008 (13)	−0.0049 (13)	0.0019 (13)
C8	0.0289 (18)	0.046 (2)	0.044 (2)	−0.0029 (17)	−0.0074 (16)	−0.0110 (19)
C9	0.0191 (15)	0.0300 (18)	0.0337 (18)	0.0006 (13)	0.0008 (13)	−0.0016 (14)
C10	0.0235 (16)	0.0229 (16)	0.0306 (17)	0.0049 (13)	0.0002 (13)	0.0031 (14)
C11	0.0261 (17)	0.035 (2)	0.0349 (19)	0.0080 (14)	0.0010 (14)	−0.0064 (15)
C12	0.0194 (14)	0.0257 (17)	0.0320 (18)	−0.0040 (13)	0.0005 (13)	−0.0006 (14)
N1	0.0238 (13)	0.0196 (13)	0.0257 (14)	0.0016 (10)	−0.0024 (10)	−0.0007 (11)
O1	0.0221 (11)	0.0309 (13)	0.0449 (15)	0.0026 (9)	−0.0021 (10)	−0.0164 (11)
O2	0.0324 (13)	0.0205 (11)	0.0268 (12)	0.0041 (10)	−0.0022 (9)	−0.0027 (9)
O3	0.0448 (15)	0.0365 (14)	0.0385 (15)	0.0230 (12)	−0.0048 (12)	−0.0094 (11)
O4	0.0385 (14)	0.0382 (14)	0.0411 (14)	0.0152 (12)	−0.0173 (11)	−0.0181 (12)
O5	0.0456 (16)	0.0429 (15)	0.0349 (14)	−0.0157 (12)	0.0022 (11)	0.0004 (12)

Geometric parameters (Å, °)

Cu1—O1	1.931 (2)	C7—C8	1.521 (4)
Cu1—N1	1.938 (3)	C8—H8A	0.9600
Cu1—O2	1.972 (2)	C8—H8B	0.9600
Cu1—O4	2.002 (2)	C8—H8C	0.9600
Cu1—O3^{i}	2.191 (2)	C9—C10	1.374 (4)
C1—C6	1.399 (4)	C9—H9	0.9300
C1—C2	1.400 (4)	C10—O1	1.296 (4)
C1—C12	1.504 (4)	C10—C11	1.498 (4)
C2—C3	1.367 (5)	C11—H11A	0.9600
C2—H2	0.9300	C11—H11B	0.9600
C3—C4	1.390 (6)	C11—H11C	0.9600
C3—H3	0.9300	C12—O3	1.246 (4)
C4—C5	1.374 (5)	C12—O2	1.273 (4)
C4—H4	0.9300	O3—Cu1^{ii}	2.191 (2)
C5—C6	1.394 (5)	O4—H4A	0.8066
C5—H5	0.9300	O4—H4B	0.7933
C6—N1	1.422 (4)	O5—H5A	0.9344
C7—N1	1.310 (4)	O5—H5B	0.8690
C7—C9	1.423 (4)		
O1—Cu1—N1	93.19 (10)	C7—C8—H8A	109.5
O1—Cu1—O2	153.49 (10)	C7—C8—H8B	109.5
N1—Cu1—O2	91.10 (10)	H8A—C8—H8B	109.5

O1—Cu1—O4	84.30 (9)	C7—C8—H8C	109.5
N1—Cu1—O4	176.97 (10)	H8A—C8—H8C	109.5
O2—Cu1—O4	90.40 (9)	H8B—C8—H8C	109.5
O1—Cu1—O3^{i}	112.57 (10)	C10—C9—C7	126.8 (3)
N1—Cu1—O3^{i}	93.54 (10)	C10—C9—H9	116.6
O2—Cu1—O3^{i}	93.23 (9)	C7—C9—H9	116.6
O4—Cu1—O3^{i}	89.00 (10)	O1—C10—C9	124.8 (3)
C6—C1—C2	118.4 (3)	O1—C10—C11	115.0 (3)
C6—C1—C12	124.7 (3)	C9—C10—C11	120.2 (3)
C2—C1—C12	116.8 (3)	C10—C11—H11A	109.5
C3—C2—C1	121.9 (3)	C10—C11—H11B	109.5
C3—C2—H2	119.0	H11A—C11—H11B	109.5
C1—C2—H2	119.0	C10—C11—H11C	109.5
C2—C3—C4	119.5 (3)	H11A—C11—H11C	109.5
C2—C3—H3	120.3	H11B—C11—H11C	109.5
C4—C3—H3	120.3	O3—C12—O2	122.5 (3)
C5—C4—C3	119.5 (4)	O3—C12—C1	116.7 (3)
C5—C4—H4	120.3	O2—C12—C1	120.8 (3)
C3—C4—H4	120.3	C7—N1—C6	122.2 (3)
C4—C5—C6	121.6 (3)	C7—N1—Cu1	123.6 (2)
C4—C5—H5	119.2	C6—N1—Cu1	114.16 (19)
C6—C5—H5	119.2	C10—O1—Cu1	122.6 (2)
C5—C6—C1	118.9 (3)	C12—O2—Cu1	124.2 (2)
C5—C6—N1	119.9 (3)	C12—O3—Cu1^{ii}	132.2 (2)
C1—C6—N1	121.0 (3)	Cu1—O4—H4A	120.7
N1—C7—C9	121.9 (3)	Cu1—O4—H4B	122.2
N1—C7—C8	123.0 (3)	H4A—O4—H4B	117.0
C9—C7—C8	115.0 (3)	H5A—O5—H5B	112.1
C6—C1—C2—C3	2.0 (5)	C1—C6—N1—C7	−135.7 (3)
C12—C1—C2—C3	−175.6 (3)	C5—C6—N1—Cu1	−132.7 (3)
C1—C2—C3—C4	1.5 (6)	C1—C6—N1—Cu1	42.7 (4)
C2—C3—C4—C5	−3.0 (6)	O1—Cu1—N1—C7	−26.1 (3)
C3—C4—C5—C6	1.1 (6)	O2—Cu1—N1—C7	127.7 (3)
C4—C5—C6—C1	2.4 (5)	O3^{i}—Cu1—N1—C7	−139.0 (3)
C4—C5—C6—N1	177.8 (3)	O1—Cu1—N1—C6	155.5 (2)
C2—C1—C6—C5	−3.8 (5)	O2—Cu1—N1—C6	−50.7 (2)
C12—C1—C6—C5	173.5 (3)	O3^{i}—Cu1—N1—C6	42.6 (2)
C2—C1—C6—N1	−179.2 (3)	C9—C10—O1—Cu1	−15.8 (5)
C12—C1—C6—N1	−1.9 (5)	C11—C10—O1—Cu1	162.9 (2)
N1—C7—C9—C10	5.7 (5)	N1—Cu1—O1—C10	26.3 (3)
C8—C7—C9—C10	−177.1 (3)	O2—Cu1—O1—C10	−72.6 (3)
C7—C9—C10—O1	−5.2 (6)	O4—Cu1—O1—C10	−151.9 (3)
C7—C9—C10—C11	176.2 (3)	O3^{i}—Cu1—O1—C10	121.5 (3)
C6—C1—C12—O3	160.9 (3)	O3—C12—O2—Cu1	174.0 (2)
C2—C1—C12—O3	−21.7 (4)	C1—C12—O2—Cu1	−5.2 (4)
C6—C1—C12—O2	−19.8 (5)	O1—Cu1—O2—C12	134.0 (2)
C2—C1—C12—O2	157.6 (3)	N1—Cu1—O2—C12	34.6 (2)

C9—C7—N1—C6	−166.8 (3)	O4—Cu1—O2—C12	−148.0 (2)
C8—C7—N1—C6	16.2 (5)	O3^{i}—Cu1—O2—C12	−59.0 (2)
C9—C7—N1—Cu1	14.9 (4)	O2—C12—O3—Cu1^{ii}	−10.7 (5)
C8—C7—N1—Cu1	−162.1 (2)	C1—C12—O3—Cu1^{ii}	168.5 (2)
C5—C6—N1—C7	48.9 (4)		

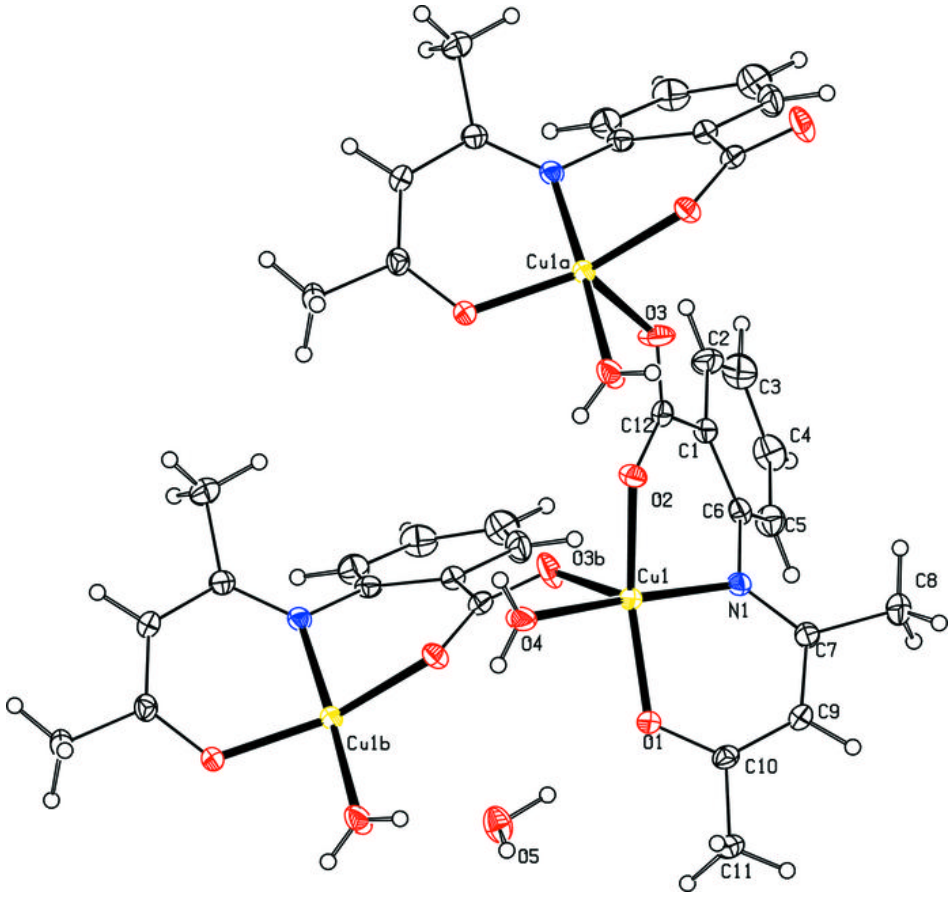
Symmetry codes: (i) −*x*+1/2, *y*+1/2, *z*; (ii) −*x*+1/2, *y*−1/2, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5B···O4^{iii}	0.87	2.45	3.264 (3)	156
O5—H5B···O1^{iii}	0.87	2.51	3.227 (3)	140
O5—H5A···O1	0.93	1.91	2.815 (3)	162
O4—H4B···O5	0.79	2.49	3.208 (4)	150
O4—H4A···O5^{ii}	0.81	1.86	2.663 (3)	176

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

Fig. 1


Cu1a
O3
C2
C3
C12
C1
C4
O2
C6
C5
O3b
Cu1
N1
C8
O4
C7
C9
Cu1b
O1
C10
O5
C11