STRUCTURAL CHEMISTRY

ISSN 2053-2296

Supramolecular hydrogen-bonded networks formed from copper(II) carboxylate dimers

Brendan F. Abrahams,* Christopher J. Commons,* Timothy A. Hudson and Robin Sanchez-Arlt

University of Melbourne, School of Chemistry, Grattan St, Parkville, 3052, Australia. *Correspondence e-mail: bfa@unimelb.edu.au, christopher.commons@unimelb.edu.au

Received 12 April 2024
Accepted 15 May 2024

Edited by D. R. Turner, University of Monash, Australia

Keywords: crystal structure; hydrogen-bonded network; 4-hydroxybenzoic acid; short hydrogen bond; crystal engineering; topology; paddle wheel complex; carboxylate dimer.

CCDC references: 2355537; 2355536; 2355535; 2355534; 2355533; 2355532; 2355531; 2355530; 2355529; 2355528; 2355527; 2355526

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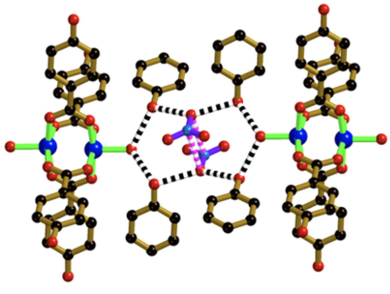


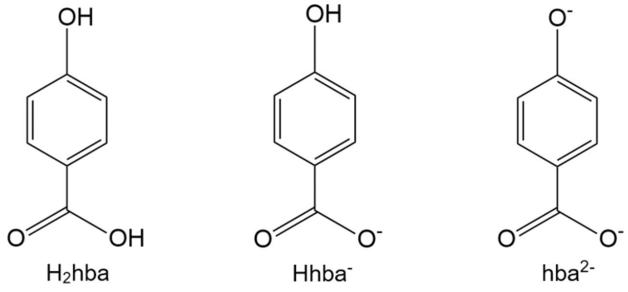
The well-known copper carboxylate dimer, with four carboxylate ligands extending outwards towards the corners of a square, has been employed to generate a series of crystalline compounds. In particular, this work centres on the use of the 4-hydroxybenzoate anion ($Hhba^-$) and its deprotonated phenolate form 4-oxidobenzoate (hba^{2-}) to obtain complexes with the general formula $[Cu_2(Hhba)_{4-x}(hba)_xL_{2-y}]^{x-}$, where L is an axial coligand (including solvent molecules), x = 0, 1 or 2, and y = 0 or 1. In some cases, short hydrogen bonds result in complexes which may be represented as $[Cu_2(Hhba)_2(H_{0.5}hba)_2L_2]^-$. The main focus of the investigation is on the formation of a variety of extended networks through hydrogen bonding and, in some crystals, coordinate bonds when bridging coligands (L) are employed. Crystals of $[Cu_2(Hhba)_4(dioxane)_2]\cdot4(dioxane)$ consist of the expected Cu dimer with the $Hhba^-$ anions forming hydrogen bonds to 1,4-dioxane molecules which block network formation. In the case of crystals of composition $[Et_4N][Cu_2(Hhba)_2(H_{0.5}hba)_2(CH_3OH)(H_2O)]\cdot2(dioxane)$, $Li[Cu_2(Hhba)_2(H_{0.5}hba)_2(H_2O)_2]\cdot3(dioxane)\cdot4H_2O$ and $[Cu_2(Hhba)_2(H_{0.5}hba)_2(H_{0.5}DABCO)_2]\cdot3CH_3OH$ (DABCO is 1,4-diazabicyclo[2.2.2]octane), square-grid hydrogen-bonded networks are generated in which the complex serves as one type of 4-connecting node, whilst a second 4-connecting node is a hydrogen-bonding motif assembled from four phenol/phenolate groups. Another two-dimensional (2D) network based upon a related square-grid structure is formed in the case of $[Et_4N]_2[Cu_2(Hhba)_2(hba)_2(dioxane)_2][Cu_2(Hhba)_4(dioxane)(H_2O)]\cdot CH_3OH$. In $[Cu_2(Hhba)_4(H_2O)_2]\cdot2(Et_4NNO_3)$, a square-grid structure is again apparent, but, in this case, a pair of nitrate anions, along with four phenolic groups and a pair of water molecules, combine to form a second type of 4-connecting node. When 1,8-bis(dimethylamino)-naphthalene (bdn, 'proton sponge') is used as a base, another square-grid network is generated, *i.e.* $[Hbdn]_2[Cu_2(Hhba)_2(hba)_2(H_2O)_2]\cdot3(dioxane)\cdot H_2O$, but with only the copper dimer complex serving as a 4-connecting node. Complex three-dimensional networks are formed in $[Cu_2(Hhba)_4(O\text{-bipy})]\cdot H_2O$ and $[Cu_2(Hhba)_4(O\text{-bipy})_2]\cdot2(dioxane)$, where the potentially bridging 4,4′-bipyridine *N*,*N*′-dioxide (O-bipy) ligand is employed. Rare cases of mixed carboxylate copper dimer complexes were obtained in the cases of $[Cu_2(Hhba)_3(OAc)(dioxane)]\cdot3.5(dioxane)$ and $[Cu_2(Hhba)_2(OAc)_2(DABCO)_2]\cdot10(dioxane)$, with each structure possessing a 2D network structure. The final compound reported is a simple hydrogen-bonded chain of composition $(H_{0.5}DABCO)(H_{1.5}hba)$, formed from the reaction of H_2hba and DABCO.

1. Introduction

In 1953, the structure of the copper(II) acetate tetra-μ-acetato-bis[aquacopper(II)], $[Cu_2(OAc)_4(H_2O)_2]$, was reported and shown to consist of a pair of Cu^{II} centres bridged by four carboxylate groups (van Niekerk & Schoening, 1953). This remarkable compound, possessing a beautiful blue colour and an elegant structure, is one of the classic coordination complexes and, as a result, is a common synthetic target in

undergraduate chemistry laboratory programs. The four carboxylate ligands extend outwards towards the vertices of an approximate square, and thus the binuclear unit has been employed as a square-planar connector in both discrete and polymeric supramolecular systems formed from bridging ligands possessing two or more carboxylate groups (Eddaoudi *et al.*, 2001; Chui *et al.*, 1999; Lee *et al.*, 2021). The binuclear tetracarboxylate moiety has been commonly referred to as a 'paddle wheel unit', with the Cu···Cu direction colinear with the 'wheel axle' and the carboxylate groups serving as 'paddles'.

Over the last decade our group has been interested in coordination polymers formed from the combination of metal ions with the anions of 4-hydroxybenzoic acid (H_2hba; Scheme 1), resulting in a variety of polymeric networks with compositions such as Zn(hba) and Co(hba) (White *et al.*, 2015), and $Cu_3(hba)_2(OH)_2$ (Abrahams *et al.*, 2022), which formed as solvates. Whilst these networks contain the hba^{2-} ligand in the presence of transition-metal ions, the reactions with Group 1 and 2 metal ions often failed to remove the phenolic proton of H_2hba, even when the metal hydroxide was employed. These reactions commonly resulted in only the $Hhba^-$ monoanion being formed (Abrahams *et al.*, 2021). In some instances, the ligand formed a strong hydrogen bond with an adjacent ligand to create a dimeric unit with a 1− charge, *i.e.* $(H_{1.5}hba)_2$. In this unit, the O atoms involved are closely separated (approximately 2.45 Å), with the H atom between the two halves of the dimer either at the mid-point of the two O atoms or disordered over two closely separated positions.



Scheme 1

The observation that the phenolic protons were retained in the generation of hydrogen-bonded networks prompted an investigation into whether the binuclear Cu^{II} dimer unit, combined with 4-hydroxybenzoic acid in either its monoanionic or dianionic form, was able to serve as a square-planar hydrogen-bonding unit in extended structures. Within such structures, negatively charged phenolate groups could serve as hydrogen-bond acceptors, whilst phenolic groups could serve as both acceptors and donors. This current work describes a series of 11 compounds (**1**–**11**) involving $Hhba^-$/hba^{2-} ligands in which the geometry of the Cu dimer leads to the generation of a variety of hydrogen-bonded complexes and networks. The structure of a 1:1 cocrystal (**12**) formed from the combination of H_2hba and DABCO (DABCO is 1,4-diazabicyclo[2.2.2]octane) is also reported. Compound **12** serves as a convenient starting material for the generation of compound **4**.

2. Experimental

2.1. Synthesis and crystallization

1–**11** are compounds of copper. Compound **12** is a cocrystal of H_2hba and DABCO, denoted $(H_{0.5}DABCO)(H_{1.5}hba)$, which was used in the synthesis of **4**.

2.1.1. $[Cu_2(Hhba)_4(dioxane)_2]\cdot 4(dioxane)$, 1. $Cu(OAc)_2\cdot H_2O$ (0.40 g, 2.0 mmol) and H_2hba (0.55 g, 4.0 mmol) were dissolved in 1,4-dioxane (40 ml). The solution was heated, with stirring, for 2.5 h at approximately 80 °C. The solvent was allowed to evaporate slowly and blue crystals separated from the solution after several days.

2.1.2. $[NEt_4][Cu_2(Hhba)_2(H_{0.5}hba)_2(CH_3OH)(H_2O)]\cdot 2(dioxane)$, 2. $Cu(OAc)_2\cdot H_2O$ (0.10 g, 0.50 mmol) and H_2hba (0.28 g, 2.0 mmol) were dissolved in 1,4-dioxane (10 ml). The solution was heated, with stirring, for 1.5 h at approximately 80 °C. Methanol (10 ml) and water (1 ml) were added, followed by the dropwise addition, with stirring, of a 25 wt% solution of Et_4NOH in water (1.3 ml). The solvent was allowed to evaporate slowly and blue crystals separated from solution after several days.

2.1.3. $Li[Cu_2(Hhba)_2(H_{0.5}hba)_2(H_2O)_2]\cdot 3(dioxane)\cdot 4H_2O$, 3. $Cu(OAc)_2\cdot H_2O$ (0.40 g, 2.0 mmol) and H_2hba (0.55 g, 4.0 mmol) were dissolved in 1,4-dioxane (30 ml). The solution was heated, with stirring, for 2.5 h at approximately 80 °C.

To a 2 ml sample of this solution was added $LiOH\cdot H_2O$ (0.011 g, 0.27 mmol) and methanol (1 ml). The solvent was allowed to evaporate slowly and a mixture of dark-green crystals of **3** and pale-green crystals separated from the solution after several days. The pale-green crystals were shown by X-ray diffraction to be the mononuclear molecular complex $[Cu(Hhba)_2(H_2O)_3]\cdot H_2O$. The structure of this complex has been reported previously [Cambridge Structural Database (CSD) refcodes BAPHEF (Shnulin *et al.*, 1981) and ICUJUN (Liu & Li, 2007); CSD Version 5.45, March 2024 release; Groom *et al.*, 2016].

2.1.4. $[Cu_2(Hhba)_2(H_{0.5}hba)_2(H_{0.5}DABCO)_2]\cdot 3CH_3OH$, 4. $Cu(OAc)_2\cdot H_2O$ (0.016 g, 0.080 mmol) and $(H_{0.5}DABCO)(H_{1.5}hba)$ (0.46 g, 1.3 mmol) were dissolved in CH_3OH (12 ml). The solvent was allowed to evaporate slowly and dark-green crystals formed after one day. [The synthesis of $(H_{0.5}DABCO)(H_{1.5}hba)$, **12**, is described below.]

2.1.5. $[Et_4N]_2[Cu_2(Hhba)_2(hba)_2(dioxane)_2][Cu_2(Hhba)_4(dioxane)(H_2O)]\cdot CH_3OH$, 5. $Cu(OAc)_2\cdot H_2O$ (0.040 g, 0.20 mmol) and H_2hba (0.055 g, 0.040 mmol) were dissolved in 1,4-dioxane (10 ml). The solution was heated, with stirring, for 4 h at approximately 80 °C and methanol (10 ml) was added. A few drops of a 25 wt% solution of Et_4NOH in water were added slowly with stirring until the precipitate that formed upon addition of each drop only just dissolved. Solvent was allowed to evaporate slowly and dark-blue crystals separated from solution after several days.

Table 1
Experimental details.

Data for compounds **4** and **5** were collected at the Australian Synchrotron (beamlines MX1 and MX2, respectively). An Oxford Diffraction Supernova diffractometer was used for compounds **8** and **12**. Other diffraction data were measured using a Rigaku XtalLAB Synergy S (Dualflex, HyPix) diffractometer. Cu $K\alpha^1$ radiation was employed on the laboratory-based diffractometers. Absorption corrections on the synchrotron data employed the multi-scan method using *XDS* (Kabsch, 2010). The multi-scan method was also used for diffractometer data employing *CrysAlis PRO* (Rigaku OD, 2018). Data were collected at 100 K, except for **8** and **12** (130 K). H atoms were treated by a mixture of independent and constrained refinement.

	1	**2**	**3**	**4**
Crystal data				
Chemical formula	$[Cu_2(C_7H_5O_3)_4(C_4H_8O_2)_2]\cdot 4C_4H_8O_2$	$(C_8H_{20}N)[Cu_2(C_7H_5O_3)_2(C_7H_{4.5}O_3)_2(CH_4O)(H_2O)]\cdot 2C_4H_8O_2$	$[Li(C_4H_8O_2)(H_2O)_3][Cu_2(C_7H_5O_3)_2(C_7H_{4.5}O_3)_2(H_2O)_2]\cdot 2C_4H_8O_2\cdot H_2O$	$[Cu_2(C_7H_5O_3)_2(C_7H_{4.5}O_3)_2(C_6H_{12.5}N_2)_2]\cdot 3CH_4O$
M_r	1204.14	1031.02	1047.81	995.99
Crystal system, space group	Orthorhombic, *Pccn*	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
a, b, c (Å)	27.7701 (8), 21.2278 (4), 9.3888 (1)	10.1659 (4), 10.7383 (6), 12.9878 (4)	9.7711 (8), 10.1924 (6), 12.5863 (7)	8.8860 (18), 10.702 (2), 12.360 (3)
α, β, γ (°)	90, 90, 90	76.132 (4), 69.646 (3), 75.304 (4)	73.615 (5), 79.528 (6), 83.412 (6)	73.13 (3), 73.07 (3), 81.68 (3)
V (Å^3)	5534.7 (2)	1267.74 (10)	1179.86 (15)	1073.8 (4)
Z	4	1	1	1
μ (mm^{-1})	1.66	1.63	1.85	1.07
Crystal size (mm)	0.14 × 0.13 × 0.05	0.17 × 0.12 × 0.05	0.14 × 0.07 × 0.03	0.18 × 0.11 × 0.07
Data collection				
T_{min}, T_{max}	0.567, 1.000	0.885, 1.000	0.610, 1.000	0.321, 0.432
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18728, 5586, 4112	17988, 5262, 4672	10093, 4145, 3048	15021, 3849, 2994
R_{int}	0.049	0.047	0.070	0.061
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.634	0.634	0.602	0.617
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.187, 1.04	0.068, 0.200, 1.06	0.089, 0.261, 1.09	0.089, 0.263, 1.05
No. of reflections	5586	5262	4145	3849
No. of parameters	354	459	338	325
No. of restraints	2	419	42	364
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.47, −0.48	0.73, −1.00	1.21, −1.32	1.14, −0.76

	5	**6**	**7**	**8**
Crystal data				
Chemical formula	$(C_8H_{20}N)[Cu_2(C_7H_4O_3)_2(C_7H_5O_3)_2(C_4H_8O_2)_2][Cu_2(C_7H_5O_3)_4(C_4H_8O_2)(H_2O)]\cdot CH_4O$	$(C_8H_{20}N)_2[Cu_2(C_7H_5O_3)_4(H_2O)_2](NO_3)_2$	$(C_{14}H_{19}N_2)_2[Cu_2(C_7H_5O_3)_2(C_7H_4O_3)_2(H_2O)_2]\cdot 3(C_4H_8O_2)\cdot H_2O$	$[Cu_2(C_7H_5O_3)_4(C_{10}H_8N_2O_2)]\cdot H_2O$
M_r	1923.89	1096.07	1420.47	881.72
Crystal system, space group	Orthorhombic, *Pnma*	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Monoclinic, *C*2/*c*
a, b, c (Å)	12.833 (3), 54.548 (11), 12.119 (2)	10.4964 (3), 10.6595 (2), 12.3226 (3)	10.2155 (2), 12.0897 (3), 15.7111 (4)	11.9851 (8), 17.7472 (11), 17.1294 (10)
α, β, γ (°)	90, 90, 90	97.271 (2), 101.042 (2), 108.130 (2)	69.581 (3), 75.988 (2), 79.651 (2)	90, 92.637 (5), 90
V (Å^3)	8483 (3)	1260.15 (6)	1754.64 (8)	3639.6 (4)
Z	4	1	1	4
μ (mm^{-1})	1.08	1.72	1.38	2.12
Crystal size (mm)	0.13 × 0.09 × 0.06	0.16 × 0.13 × 0.09	0.19 × 0.16 × 0.12	0.32 × 0.06 × 0.05
Data collection				
T_{min}, T_{max}	0.321, 0.432	0.720, 1.000	0.890, 1.000	0.898, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	151309, 13753, 11493	14549, 5136, 4648	21601, 6618, 5772	6868, 3663, 3386
R_{int}	0.054	0.048	0.036	0.024
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.753	0.634	0.610	0.629
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.148, 1.02	0.038, 0.101, 1.08	0.064, 0.189, 1.05	0.034, 0.098, 1.05
No. of reflections	13753	5136	6618	3663
No. of parameters	585	408	361	263
No. of restraints	67	263	3	2
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.42, −0.84	0.47, −0.54	1.05, −0.62	0.36, −0.57

Table 1 (continued)

	9	10	11	12
Crystal data				
Chemical formula	$[Cu_2(C_7H_5O_3)_4(C_{10}H_8N_2O_2)_2]\cdot 2C_4H_8O_2$	$[Cu_2(C_7H_5O_3)_3(C_2H_3O_2)(C_4H_8O_2)]\cdot 3.5C_4H_8O_2$	$[Cu_2(C_7H_5O_3)_2(C_2H_3O_2)_2(C_6H_{12}N_2)_2]\cdot 10C_4H_8O_2$	$C_6H_{13}N_2^+\cdot C_7H_5O_3^-$
M_r	1228.09	993.92	1624.77	250.29
Crystal system, space group	Orthorhombic, *Pbca*	Monoclinic, *I*2/*a*	Monoclinic, *Pc*	Monoclinic, $P2_1/n$
a, *b*, *c* (Å)	17.5993 (3), 16.4583 (2), 19.7389 (2)	19.4801 (2), 13.0774 (2), 34.5443 (5)	9.7404 (3), 20.3001 (4), 22.9088 (7)	15.3994 (5), 10.6592 (3), 16.9261 (6)
α, β, γ (°)	90, 90, 90	90, 97.702 (1), 90	90, 118.494 (4), 90	90, 110.424 (4), 90
V (Å^3)	5717.46 (13)	8720.7 (2)	3981.1 (2)	2603.67 (15)
Z	4	8	2	8
μ (mm^{-1})	1.59	1.90	1.36	0.75
Crystal size (mm)	0.09 × 0.07 × 0.05	0.26 × 0.09 × 0.06	0.34 × 0.25 × 0.14	0.22 × 0.16 × 0.13
Data collection				
T_{min}, T_{max}	0.344, 1.000	0.764, 1.000	0.533, 1.000	0.952, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23256, 5876, 4731	37963, 9032, 6906	27595, 12037, 9752	11079, 5386, 3805
R_{int}	0.056	0.071	0.040	0.039
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.634	0.635	0.635	0.632
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.044, 0.129, 1.06	0.065, 0.191, 1.09	0.098, 0.287, 1.21	0.044, 0.120, 1.01
No. of reflections	5876	9032	12037	5386
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.59, −0.68	1.12, −1.24	1.38, −0.70	0.19, −0.24
Absolute structure	–	–	Refined as an inversion twin	–
Absolute structure parameter	–	–	0.46 (6)	–

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015, 2018, 2021, 2023), *XDS* (Kabsch, 2010), *SHELXT* (Sheldrick, 2015*a*), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL2018* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

2.1.6. $[Cu_2(Hhba)_4(H_2O)_2]\cdot 2Et_4N(NO_3)$, 6. H_2hba (0.55 g, 4.0 mmol) was added to a 25 wt% solution of Et_4NOH in water (8.0 ml) and heated, with stirring, at approximately 80 °C for 30 min. Water was evaporated from the solution and the resulting white solid dried by vacuum sublimation. A solution of the white solid (0.10 g) in methanol (2 ml) was added to a solution of $Cu(NO_3)_2\cdot 3H_2O$ (0.10 g, 0.41 mmol) in methanol (2 ml). A yellow–green precipitate formed immediately which redissolved with stirring. The solvent was allowed to evaporate slowly and blue crystals separated from the solution after several days.

2.1.7. $[Hbdn]_2[Cu_2(Hhba)_2(hba)_2(H_2O)_2]\cdot 3(dioxane)\cdot H_2O$, 7. $Cu(OAc)_2\cdot H_2O$ (0.10 g, 0.50 mmol) and H_2hba (0.14 g, 1.0 mmol) were added to 1,4-dioxane (10 ml). The solution was heated, with stirring, at approximately 70 °C for 10 min. 1,8-Bis(dimethylamino)naphthalene (bdn, 'proton sponge'; 0.070 g, 0.33 mmol) and acetonitrile (2 ml) were added, and the mixture heated for a further 30 min. The solvent was allowed to evaporate slowly and green crystals separated from solution after several days.

2.1.8. $[Cu_2(Hhba)_4(O\text{-}bipy)]\cdot H_2O$, 8. $Cu(OAc)_2\cdot H_2O$ (0.20 g, 1.0 mmol), 4,4′-bipyridine *N*,*N*′-dioxide (O-bipy; 0.10 g, 0.53 mmol) and H_2hba (0.20 g, 1.4 mmol) were added to water (10 ml) and xylene (1 ml). The mixture was heated at 110 °C in a Teflon-lined autoclave for 5 h to yield dark-green crystals.

2.1.9. $[Cu_2(Hhba)_4(O\text{-}bipy)_2]\cdot 2(dioxane)$, 9. $Cu(OAc)_2\cdot H_2O$ (0.10 g, 0.50 mmol), O-bipy (0.050 g, 0.27 mmol) and H_2hba (0.050 g, 0.36 mmol) were added to 1,4-dioxane (10 ml). The mixture was heated at 110 °C in a Teflon-lined autoclave for 1 d to yield dark-green crystals.

2.1.10. $[Cu_2(Hhba)_3(OAc)(dioxane)]\cdot 3.5dioxane$, 10. $Cu(OAc)_2\cdot H_2O$ (0.40 g, 2.0 mmol) and H_2hba (0.55 g, 4.0 mmol) were dissolved in 1,4-dioxane (40 ml). The solution was heated, with stirring, for 4 h at approximately 80 °C. The solvent was allowed to evaporate slowly and dark-blue crystals separated from solution after several days.

2.1.11. $[Cu_2(Hhba)_2(OAc)_2(DABCO)_2]\cdot 10(dioxane)$, 11. $Cu(OAc)_2\cdot H_2O$ (0.10 g, 0.50 mmol), H_2hba (0.069 g, 0.50 mmol) and 1,4-diazabicyclo[2.2.2]octane (DABCO; 0.061 g, 0.54 mmol) were added to 1,4-dioxane (50 ml). A 0.01 *M* NaOH solution (50 µl) was then added. The mixture was heated at 115 °C in a Teflon-lined autoclave for 2 d to yield small green crystals.

2.1.12. $(H_{0.5}DABCO)(H_{1.5}hba)$, 12. Separate 5 ml methanol solutions of H_2hba (1.34 g, 10 mmol) and DABCO (2.75 g, 25 mmol) were prepared. The two solutions were heated to approximately 40 °C, mixed and then heated for a further 15 min. Acetone (20 ml) was added and the solution allowed to evaporate. Colourless crystals formed after approximately 1 h.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms of water molecules, phenolic groups and carboxylic acid groups were commonly located in difference Fourier maps and were generally refined with O—H distances restrained to 0.85 Å and U_{iso}(H) values of $1.5U_{eq}$(O).

In **2**, H atoms were involved in short hydrogen bonds (O···O distances less than 2.45 Å) between the symmetry-

related O atoms of phenolate groups, and single peaks were observed in difference Fourier maps midway between the atoms. These peaks were assigned as H atoms and refined independently. Short hydrogen bonds were also present in **3** and **4** between the symmetry-related O atoms of phenol groups; however, single peaks midway between the O atoms were not observed. In these cases, H atoms were located on both O atoms with 0.5 occupancies and O—H distances restrained to 0.85 Å.

The H_2hba and DABCO molecules are closely aligned in **12** (N···O distances of approximately 2.52 Å) and single peaks were observed in difference Fourier maps almost midway

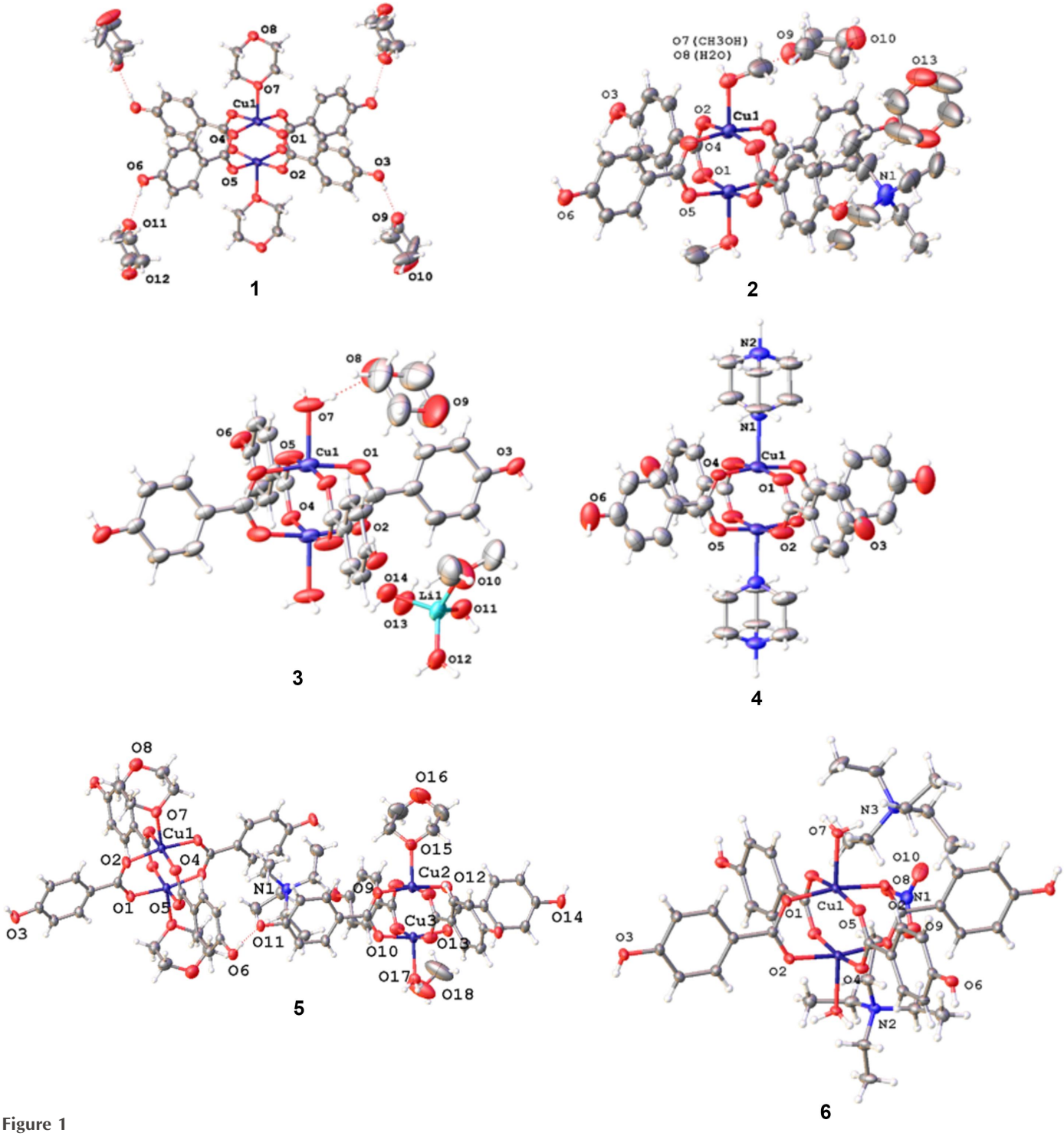


Figure 1
The structures of compounds **1**–**6**, showing the copper dimer units and the atom-labelling schemes. For clarity, the labels of the C and H atoms are not shown. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size. The red dotted lines represent hydrogen-bonding interactions. In **2**, only one configuration of the disordered dioxane molecules is shown, and in **4**, only one configuration of the DABCO molecules is shown.

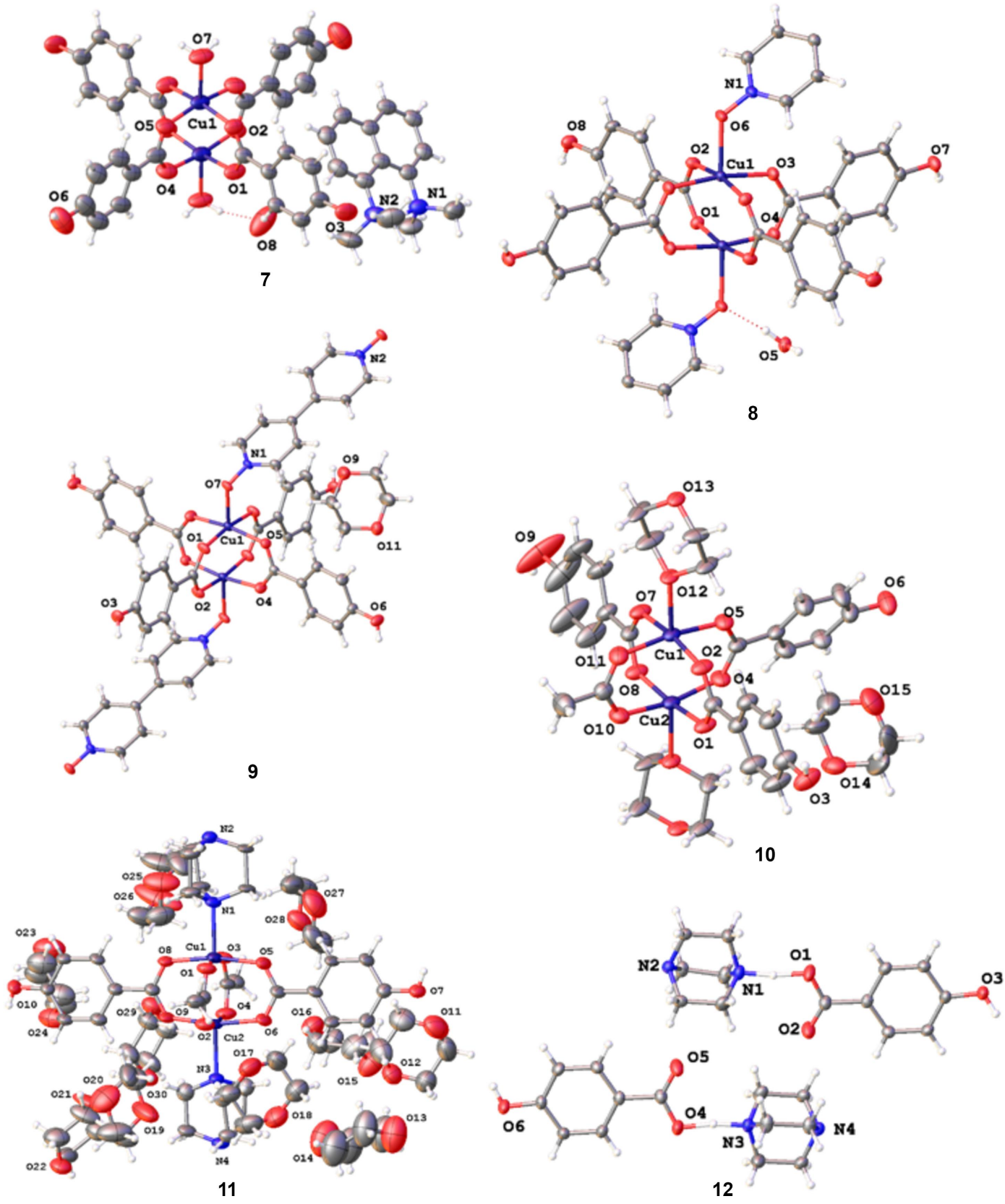


Figure 2
The structures of compounds **7**–**11**, showing the copper dimer units and the atom-labelling schemes. The structure of the cocrystal formed from the combination of H_2hba and DABCO (**12**) is also shown. For clarity, the labels of the C and H atoms are not shown. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size. The red dotted lines represent hydrogen-bonding interactions. In **9**, only one configuration of the disordered dioxane molecules is shown, and in **11**, only one configuration of the DABCO molecules is shown.

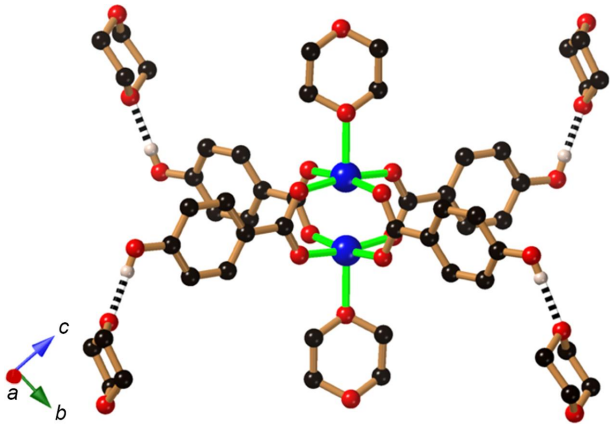


Figure 3
The structure of $[Cu_2(Hhba)_4(dioxane)_2]\cdot 4(dioxane)$ (**1**), showing the links to two coordinated dioxane molecules and hydrogen-bond interactions with four non-coordinated dioxane molecules. Colour code for this and later figures, as applicable: Cu dark blue, C black, O red, N and Li pale blue, and H pale pink. Hydrogen bonds are indicated by black and white striped connections. For clarity, H atoms not involved in hydrogen bonding have been omitted in this and most of the following figures.

between the N and O atoms. These peaks were assigned as H atoms and refined independently.

Other H atoms were placed in calculated positions and refined as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene and aromatic C atoms, $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl C atoms, and with C—H = 0.95 Å for aromatic groups, 0.99 Å for methylene group and 0.98 Å for methyl groups. The H atoms of two disordered water molecules (containing O11 and O13) were not modelled in **3**. Details of the refinements can be found in the embedded CIF files.

The mask process within *OLEX2* (Dolomanov *et al.*, 2009) was employed in the structure refinements for **4**, **7** and **10**, which represent structures for which not all solvent molecules could be satisfactorily modelled.

3. Results and discussion

Labelled structures of compounds **1**–**12** showing displacement ellipsoids for non-H atoms are presented in Figs. 1 and 2.

Our general strategy towards the synthesis of hydrogen-bonded copper(II) complexes incorporating $Hhba^-$ anions involves the addition of H_2hba to $Cu_2(OAc)_4\cdot 2H_2O$. In this reaction, the acetate anion can serve as the base for H_2hba. The combination of $Cu_2(OAc)_4\cdot 2H_2O$ with H_2hba in a 1:4 ratio in 1,4-dioxane (dioxane) yields a compound of composition $[Cu_2(Hhba)_4(dioxane)_2]\cdot 4(dioxane)$ (**1**). As anticipated, the dimeric copper carboxylate complex is formed, as indicated in Fig. 3, with the $Hhba^-$ units extending towards the vertices of an approximate square. Each phenolic group of the coordinated $Hhba^-$ serves as a proton donor to a dioxane molecule. In addition, a dioxane molecule is coordinated to opposing axial sites on each of the symmetry-related Cu^{II} centres.

The compound $[NEt_4][Cu_2(Hhba)_2(H_{0.5}hba)_2(CH_3OH)(H_2O)]\cdot 2(dioxane)$ (**2**) is formed by the combination of $Cu_2(OAc)_4(H_2O)_2$ and H_2hba in dioxane, followed by the addition of NEt_4OH in an aqueous methanol solution. It was anticipated that the addition of the base (NEt_4OH) would serve to generate at least some hba^{2-} anions. The Cu dimeric complex present in **1** is also evident in **2**, however, with disordered water and methanol molecules occupying the axial sites in place of the dioxane molecules. A centre of inversion lies between the two Cu^{II} centres. The complex has formally lost one phenolic H atom, resulting in the formation of the anionic complex $[Cu_2(Hhba)_2(H_{0.5}hba)_2(CH_3OH)(H_2O)]^-$. The four organic groups extend outwards towards the vertices of an approximate square and thus the complex may be con-

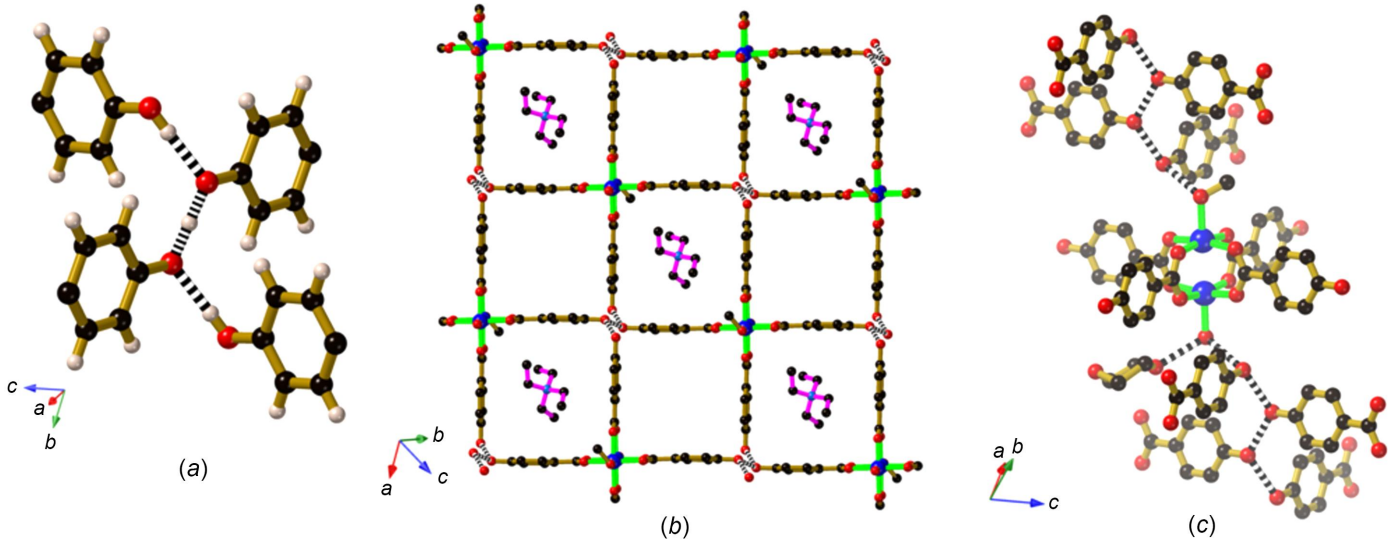


Figure 4
The structure of $[NEt_4][Cu_2(Hhba)_2(H_{0.5}hba)_2(CH_3OH)(H_2O)]\cdot 2(dioxane)$ (**2**), showing (*a*) the hydrogen-bonded 4-connected node, (*b*) the 4,4-network with the dioxane molecules and NEt_4^+ ions in the square cavities (only one of the two orientations of each tetraethylammonium ion is shown), and (*c*) the intersheet hydrogen-bond interactions involving the coordinated water/methanol molecules which extend into sheets above and below.

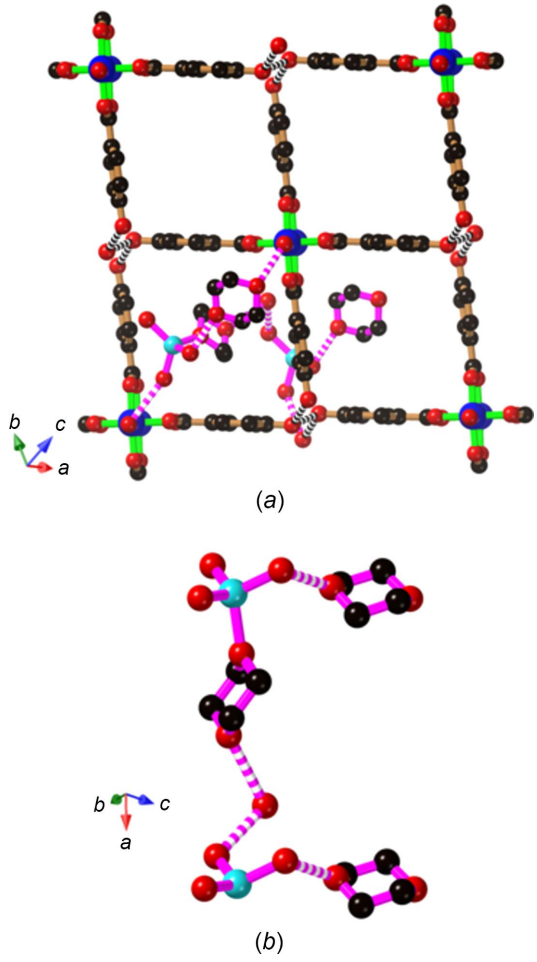


Figure 5
The structure of Li[Cu_2(Hhba)$_2$($H_{0.5}$hba)$_2$(H_2O)$_2$]·3(dioxane)·4H_2O (**3**), showing (*a*) the square-grid structure with the Li^+ cation coordinated by water and dioxane molecules located in the larger of the two types of square cavities, and (*b*) part of the infinite Li^+–solvent chain.

sidered as a 4-connecting node. Hydrogen bonding between phenolic and phenolate groups results in the generation of a second 4-connecting node within a two-dimensional (2D) network. As indicated in Fig. 4(*a*), the second type of 4-connecting node consists of three H atoms that bind four O atoms in a zigzag arrangement. The two central O atoms are separated by a H atom, which was refined to a position midway between the two symmetry-related O atoms. The O···H···O separation of 2.446 (5) Å is indicative of a strong interaction, and the dimer unit formed by this interaction may be represented as ($H_{0.5}$hba)$_2^{3-}$. Interestingly, the O···O separation between O atoms is similar to that seen in ($H_{1.5}$hba)$_2^-$ (Abrahams *et al.*, 2021). Phenolic groups above and below the central pair of O atoms form additional hydrogen bonds which are a little longer [O—H···O = 2.601 (4) Å], resulting in a short hydrogen-bonded planar zigzag chain (O—H···O···H···O···H—O).

An anionic 2D 4,4-network (square-grid network) is generated by linking the [Cu_2(Hhba)$_2$($H_{0.5}$hba)$_2$(CH_3OH)(H_2O)]$^-$ complexes through hydrogen bonds involving the phenolic/phenolate groups [Fig. 4(*b*)]. Two types of square-shaped cavities are formed within the network, with orientationally disordered tetraethylammonium counter-ions occupying the larger cavities. Disordered dioxane molecules, which were crystallographically modelled with partial occupancies, occupy the slightly smaller square cavities and space between networks.

The 2D square-grid layers described above form hydrogen bonds with adjacent parallel layers through the coordinated water/methanol molecules. The coordinated molecules serve as hydrogen-bond donors, bonding to a terminus of the hydrogen-bonded O—H···O···H···O···H—O chain. In the case of the coordinated water molecule, a hydrogen bond extends to a dioxane molecule located in one of the small square cavities of an adjacent sheet, as indicated in Fig. 4(*c*).

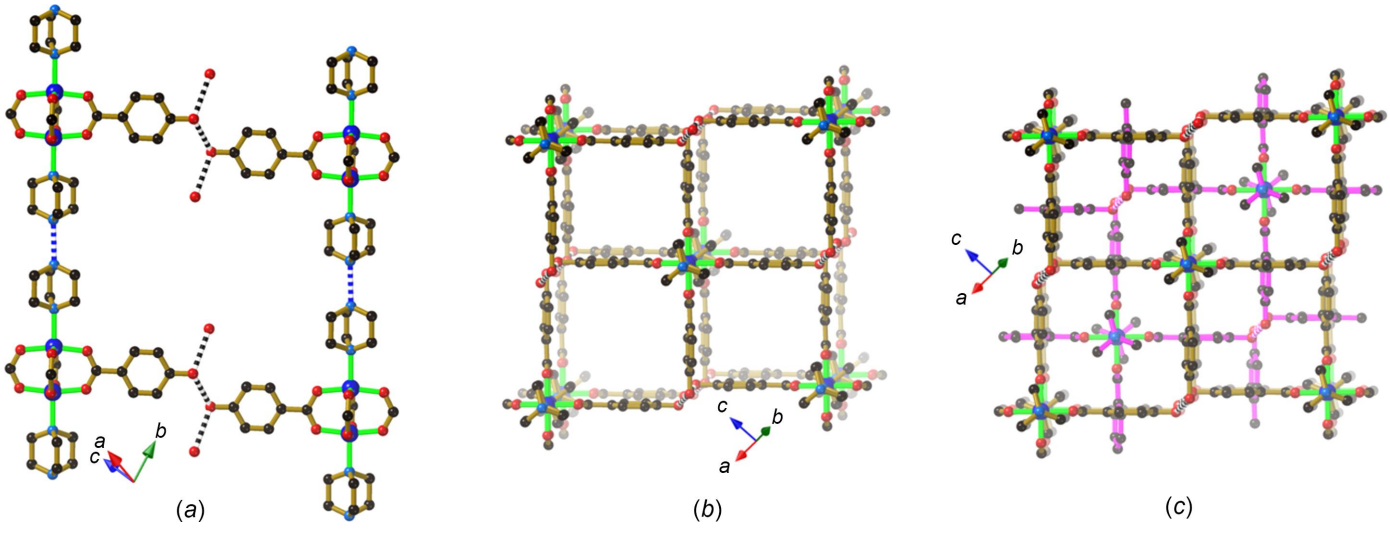


Figure 6
The structure of [Cu_2(Hhba)$_2$($H_{0.5}$hba)$_2$($H_{0.5}$DABCO)$_2$]·3CH_3OH (**4**), showing (*a*) [DABCO···H···DABCO]$^+$ links between [Cu_2(Hhba)$_2$($H_{0.5}$hba)$_2$]$^-$ hydrogen-bonded sheets, (*b*) a single network with [Cu_2($H_{0.5}$hba)$_2$(Hhba)$_2$]$^-$ hydrogen-bonded sheets linked by [DABCO···H···DABCO]$^+$ connections and (*c*) a pair of interpenetrating networks. Only a single orientation of each of the rotationally disordered DABCO units is represented in each figure.

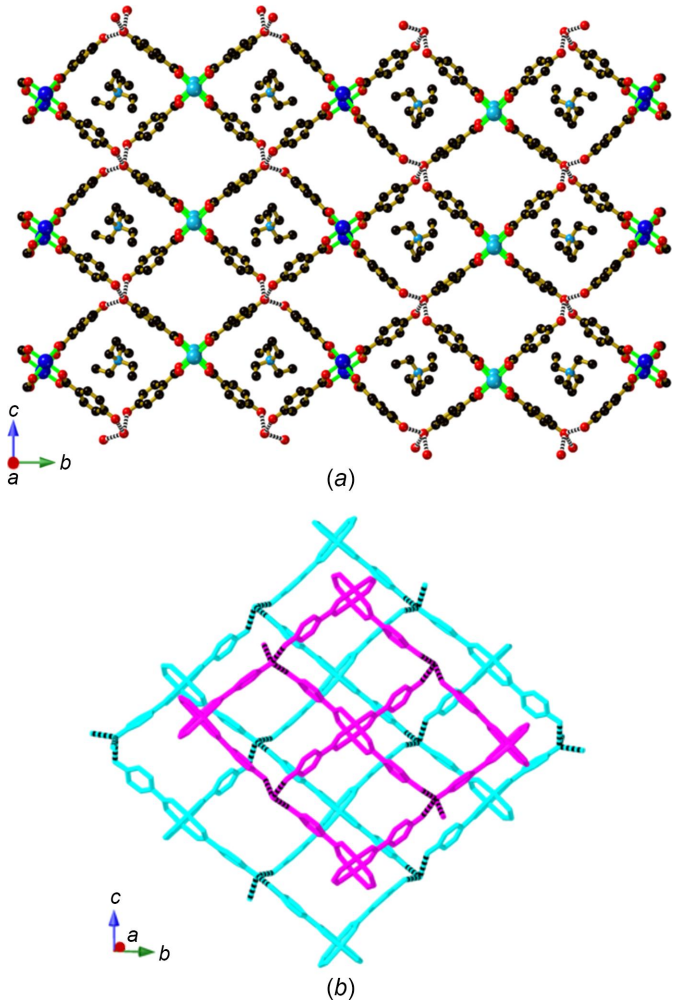


Figure 7
The structure of $[Et_4N]_2[Cu_2(Hhba)_2(hba)_2(dioxane)_2][Cu_2(Hhba)_4(dioxane)(H_2O)]\cdot CH_3OH$ (**5**), showing (*a*) the anionic 2D structure and (*b*) a pair of sheets indicating the *ABAB* . . . stacking.

Crystals of $Li[Cu_2(Hhba)_2(H_{0.5}hba)_2(H_2O)_2]\cdot 3(dioxane)\cdot 4H_2O$ (**3**) are generated using LiOH as a base. A similar 4,4-network [Fig. 5(*a*)] to that found in **2** is formed. The binuclear unit is once again located on a centre of inversion and thus the asymmetric unit within the anionic network is very similar to that of compound **2**. As with **2**, there are two types of cavities in the sheet, one slightly larger than the other. The larger cavities, which are all crystallographically identical, are occupied by dioxane molecules, each of which is bound to a Li centre through one of the O atoms. Three water molecules complete the coordination sphere of the Li centre. The other O atom of the dioxane serves as a hydrogen-bond acceptor to a water molecule. A centre of inversion lies at the centre of the dioxane and thus the Li^+ centre is disordered across a pair of symmetry-related sites. These components are part of an infinite chain consisting of water molecules, Li^+ ions and bridging and branching dioxane molecules [Fig. 5(*b*)]. The chains pass through the larger square cavities of adjacent 4,4-networks, as indicated in Fig. 5(*a*). Branching dioxane molecules occupy the smaller square cavities.

The compound $[Cu_2(Hhba)_2(H_{0.5}hba)_2(H_{0.5}DABCO)_2]\cdot 3CH_3OH$ (**4**) is formed from the reaction of $Cu_2(OAc)_4\cdot 2H_2O$ and $(H_{0.5}DABCO)(H_{1.5}hba)$ (**12**) in methanol. DABCO molecules occupy the axial sites on the Cu dimer complex and are rotationally disordered around the axis which passes through the two N centres. A similar $[Cu_2(Hhba)_2(H_{0.5}hba)_2]^-$ hydrogen-bonded 2D network to that present in compounds **2** and **3** is apparent in **4**, but rather than cations occupying square cavities, the charge on the anionic complex is balanced by a proton located between a pair of coordinated DABCO ligands ($[DABCO\cdots H\cdots DABCO]^+$), which forms a linear link between anionic sheets, as indicated in Fig. 6(*a*). The binuclear Cu complex now serves as a 6-connecting node, with the DABCO ligands extending in a direction perpendicular to

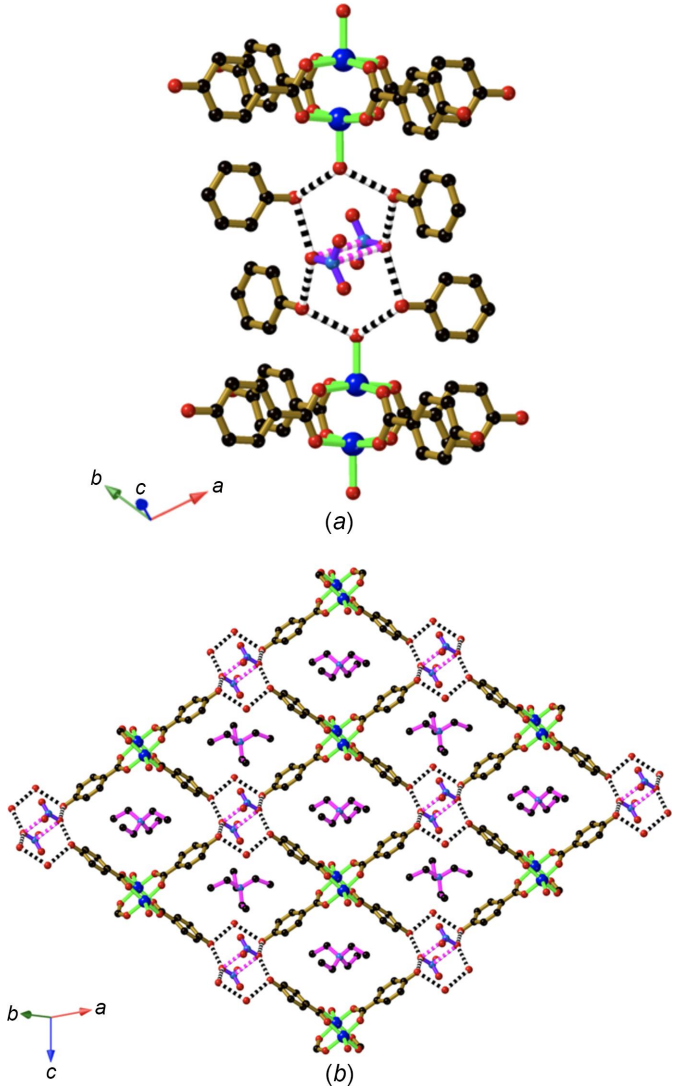


Figure 8
The structure of $[Cu_2(Hhba)_4(H_2O)_2]\cdot 2(Et_4NNO_3)$ (**6**), showing (*a*) the hydrogen bonding from four phenolic groups to a pair of nitrate anions, and from water molecules to pairs of phenolic groups, and (*b*) the 2D network. Hydrogen bonds are indicated by black and white striped connections, whilst close interionic contacts between nitrate anions are indicated by pink and white striped connections. Only a single orientation of each of the disordered Et_4N^+ units is represented in part (*b*).

the familiar 2D network which was a feature of compounds **2** and **3** [Fig. 6(*b*)]. The result is a three-dimensional (3D) hydrogen-bonded network with relatively large intraframework voids that are large enough to accommodate a second equivalent network, as depicted in Fig. 6(*c*).

An anionic 2D hydrogen-bonded network is also a feature of the compound $[Et_4N]_2[Cu_2(Hhba)_2(hba)_2(dioxane)_2][Cu_2(Hhba)_4(dioxane)(H_2O)]\cdot CH_3OH$ (**5**), but there are clear differences in the 4-connecting nodes that are formed by the aggregation of phenolic and phenolate groups. In each of the compounds **1**–**4**, there is only one crystallographically unique binuclear complex, each possessing a centre of symmetry between the two Cu^{II} centres. In **5**, however, two crystallographically distinct binuclear Cu^{II} complexes are present. The first of these consists of four $Hhba^-$ anions bound to a pair of centrosymmetrically-related Cu^{II} centres which are represented by dark-blue spheres in Fig. 7(*a*). The second complex is comprised of two crystallographically distinct Cu^{II} centres (the larger light-blue spheres) which are bound to two $Hhba^-$ anions and two hba^{2-} anions. The hba^{2-} anions are *cis* to each other in the binuclear complex. The phenolate O atoms of the hba^{2-} anions serve as hydrogen-bond acceptors from three phenolic groups, resulting in a 4-connecting hydrogen-bonding node. The O···O separations in the $[O^-\cdots(H-O)_3]$ node are in the range 2.571 (2)–2.616 (2) Å. Charge balance for the anionic network is achieved with tetraethylammonium ions that occupy every second square cavity of the 2D network, which extends in the *bc* plane. The 2D sheets stack in an *ABAB*... fashion [Fig. 7(*b*)], with tetraethylammonium ions sandwiched between $[O^-\cdots(H-O)_3]$ nodes of adjacent sheets. Dioxane and water molecules, which are bound to the axial positions of the binuclear complexes, exhibit varying degrees of disorder and protrude into the vacant square cavities of adjacent sheets.

The compound $[Cu_2(Hhba)_4(H_2O)_2]\cdot 2Et_4N(NO_3)$ (**6**) is formed when $Cu(NO_3)_2\cdot 3H_2O$ is used as the source of Cu^{II} and the reaction mixture includes H_2hba and Et_4NOH. The expected binuclear complex, with water molecules occupying the axial positions, is formed with a centre of symmetry between the metal centres. Each of the carboxylate ligands retains the phenolic proton to give the neutral $[Cu_2(Hhba)_4(H_2O)_2]$ complex. Four phenolic groups combine with a pair of nitrate anions and a pair of coordinated water molecules, from adjacent metal complexes, to form an unusual hydrogen-bonded motif, as shown in Fig. 8(*a*). An interesting feature of this motif is the association of nitrate anion pairs, which make relatively close face-to-face contacts, with N···O separations of 2.883 (2) Å. Inspection of the Cambridge Structural Database (CSD, Version 5.45, March 2024 release; Groom *et al.*, 2016) reveals numerous examples of similar face-to-face

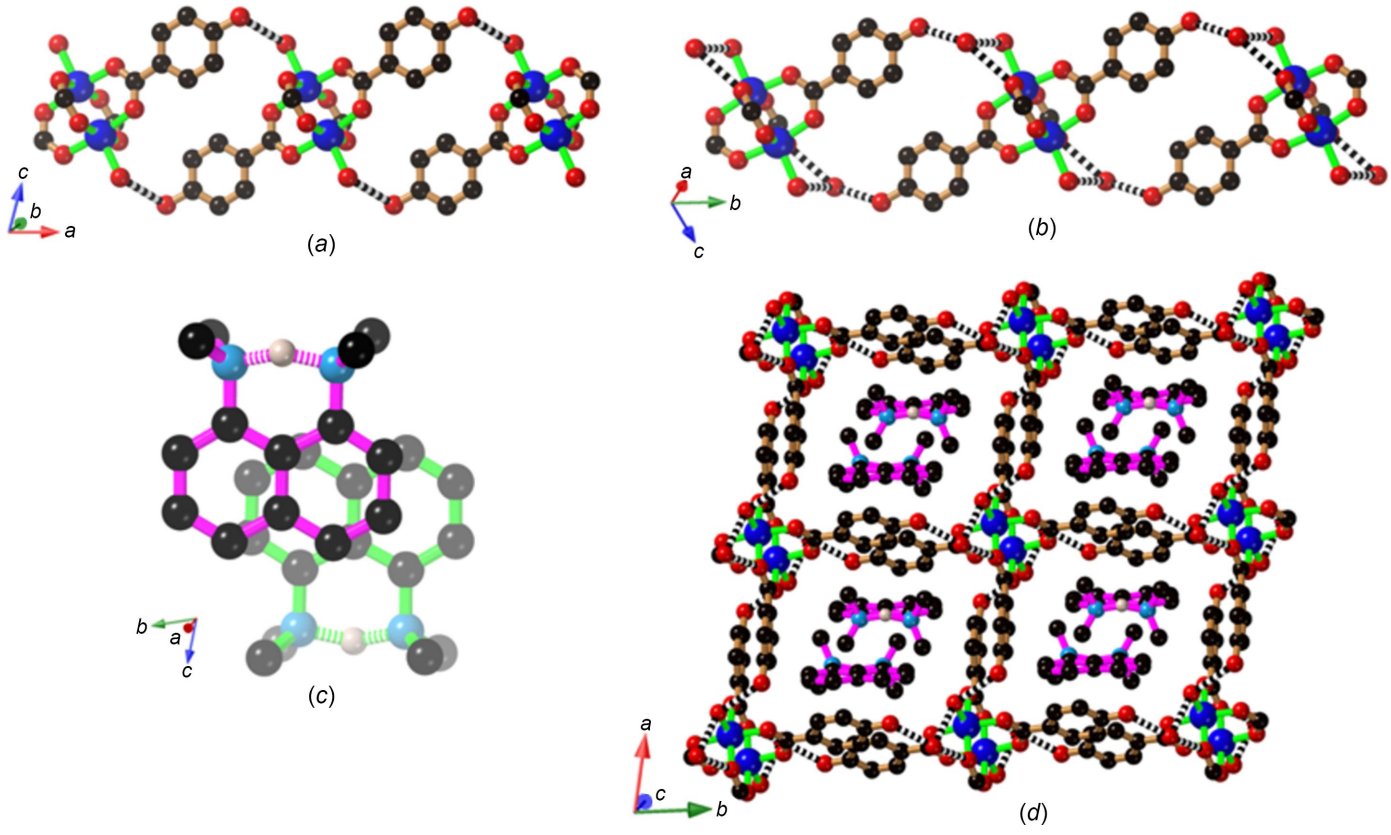


Figure 9
The structure of $[Hbdn]_2[Cu_2(Hhba)_2(hba)_2(H_2O)_2]\cdot 3(dioxane)\cdot H_2O$ (**7**), showing (*a*) the double hba^{2-} bridges between Cu^{II} pairs along the *a* axis, (*b*) the double $Hhba^-$ bridges between Cu^{II} pairs along the *b* axis, with an additional water molecule between the phenolic group and the coordinated water molecule, (*c*) a pair of face-to-face $Hbdn^+$ cations and (*d*) the 2D network extending in the *ab* plane, with the cations located in the cavities.

interactions of nitrate anions. The aggregation of the phenolic groups, the two nitrate anions and a pair of coordinated water molecules leads once again to the generation of a 2D network, as depicted in Fig. 8(*b*). If the link from the coordinated water molecule to adjacent parallel layers is considered, then the overall structure is that of a 3D network. To balance the charge of the nitrate anion pairs, a tetraethylammonium cation is required for every square cavity. There are two crystallographically distinct tetraethylammonium ions, each of which is disordered over a pair of sites in each square cavity.

The employment of the organic base 1,8-bis(dimethylamino)naphthalene (bdn), more commonly known as a proton sponge, leads to the formation of crystals of composition $[Hbdn]_2[Cu_2(Hhba)_2(hba)_2(H_2O)_2]\cdot 3(dioxane)\cdot H_2O$ (**7**). In this compound, the binuclear complex carries an overall 2− charge, with two hba^{2-} ligands *trans* to each other and extending along the *a*-axis direction. The *trans*-phenolate groups act as hydrogen-bond acceptors from coordinated water molecules belonging to complexes on either side along the direction of the *a* axis. In addition to acting as a proton acceptor *via* the *trans* hba^{2-} anions, the complex also serves as a proton donor to the complexes on either side through the coordinated water molecules. Thus, each complex forms a pair of double hba^{2-} bridges, as indicated in Fig. 9(*a*). In addition to the centre of symmetry between the Cu^{II} centres, there is a centre of symmetry between the pair of hba^{2-} anions. Similarly, along the *b* axis, there are $Hhba^{-}$ double bridges between complexes with a non-coordinated water molecule interspersed between the phenolic group and the coordinated water molecule [Fig. 9(*b*)]. The presence of these two types of double bridges results in the generation of a 4,4-network in which the binuclear complex serves as the sole 4-connecting node. Pairs of $Hbdn^{+}$ cations, in a face-to-face arrangement, occupy the approximately square cavities [Figs. 9(*c*) and 9(*d*)]. The 2D layers that are generated are separated by disordered solvent molecules.

The addition of 4,4′-bipyridine *N*,*N*′-dioxide (O-bipy) to $Cu(OAc)_2\cdot 2H_2O$ and H_2hba under solvothermal conditions yields a one-dimensional (1D) coordination polymer of composition $[Cu_2(Hhba)_4(O\text{-bipy})]\cdot H_2O$ (**8**). The O atoms of O-bipy coordinate to the axial positions of a binuclear $Cu_2(Hhba)_4$ complex, resulting in a coordination polymer chain that extends in the [201] direction [Fig. 10(*a*)]. Hydrogen-bonding interactions involving *trans*-$Hhba^{-}$ anions lead to the generation of chains, as indicated in Fig. 10(*b*). Within the crystal structure, these hydrogen-bonded chains extend in both the [110] and the [1$\bar{1}$0] directions. If the links between $Cu_2(Hhba)_4$ units, shown in Figs. 10(*a*) and 10(*b*), are taken into account, the binuclear unit may be considered as a 4-connected node within a network which has the same topology as the $CdSO_4$ net, *i.e.* Schlafli (point) symbol $6^5\cdot 8^1$. Whilst the $CdSO_4$ network is not as common as some of the better-known networks based on 4-connecting nodes, there are examples reported in the literature including one based upon a Cu^{II}–tetracarboxylate dimer (Moulton *et al.*, 2003). The remaining two $Hhba^{-}$ arms of the complex participate in hydrogen-bonding interactions with non-coordinated water molecules, which, in turn, form hydrogen-bonded links to the coordinated O atoms of the O-bipy ligands. The result is a complex 3D hydrogen-bonded network structure.

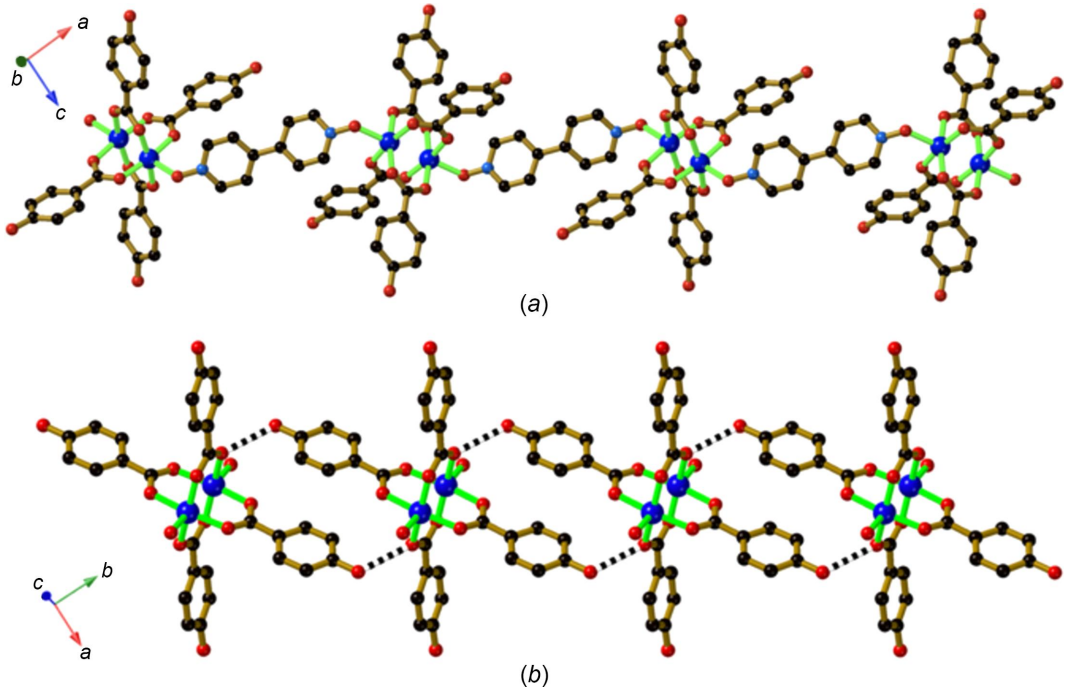


Figure 10
The structure of $[Cu_2(Hhba)_4(O\text{-bipy})]\cdot H_2O$ (**8**), showing (*a*) the 1D chains formed by the bridging O-bipy ligands and (*b*) the hydrogen-bonded chains linking $Cu_2(Hhba)_4$ units together. The chains depicted in part (*b*) extend in both the [110] and the [1$\bar{1}$0] directions.

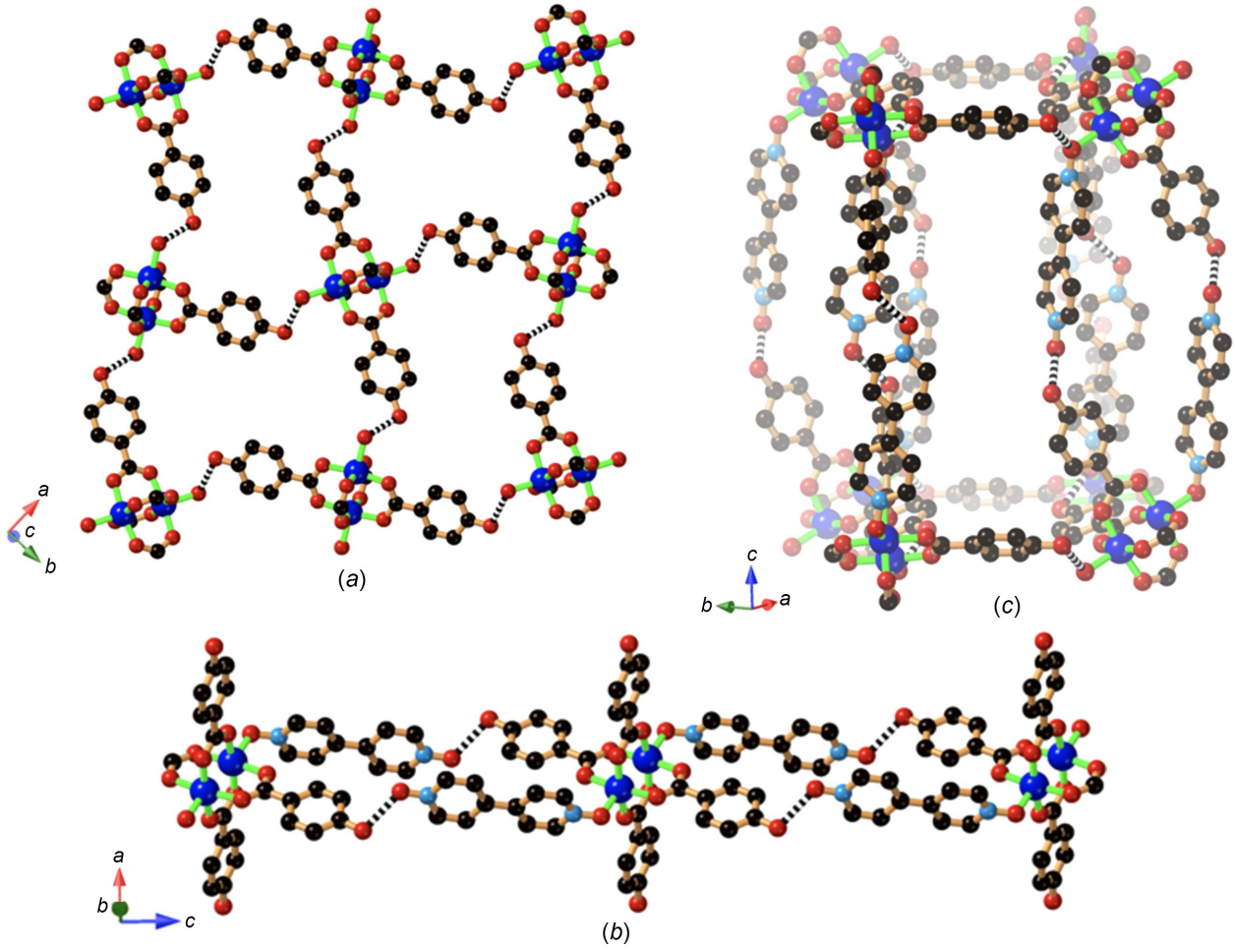


Figure 11
The structure of $[Cu_2(Hhba)_4(O\text{-bipy})_2]\cdot 2(\text{dioxane})$ (**9**), showing (*a*) part of the hydrogen-bonding 2D network that extends parallel to the *ab* plane, (*b*) double hydrogen-bonded bridges that link the sheets depicted in part (*a*), and (*c*) part of the resulting 3D network.

Under solvothermal conditions using dioxane as solvent, O-bipy reacts with $Cu(OAc)_2\cdot 2H_2O$ and H_2hba to form crystals of composition $[Cu_2(Hhba)_4(O\text{-bipy})_2]\cdot 2(\text{dioxane})$ (**9**), in which only one O atom of each O-bipy is coordinated to the neutral binuclear complex. Within this structure, a square-grid hydrogen-bonded network lying parallel to the *ab* plane is generated, with links between phenolic groups and the coordinated O-bipy O atom, as indicated in Fig. 11(*a*). Additional connections from each binuclear unit extend along the *c* axis. These connections are double bridges and consist of a hydrogen-bonding link between the non-coordinated O atom of O-bipy and a phenolic group of a $Hhba^-$ ligand [Fig. 11(*b*)]. The double Hhba···O-bipy bridges extending from the binuclear units to symmetry-related parallel units result in a 3D network that has the topology of the simple (primitive) cubic net, which is also known as the α-polonium net [Fig. 11(*c*)]. The intraframework space is occupied by a symmetry-related second network.

In the preparation of the copper compounds described above, except for **6**, the binuclear copper acetate complex served as the source of Cu^{II}. In the following two examples (**10** and **11**), only partial replacement of the acetate by the $Hhba^-$ ligands has occurred. In $[Cu_2(Hhba)_3(OAc)(\text{dioxane})]\cdot 3.5(\text{dioxane})$ (**10**), the binuclear unit is again present, but with the Cu^{II} ions coordinated by three $Hhba^-$ anions and one acetate anion. In this case, the binuclear units are bridged by dioxane molecules to form a chain that extends along the *a* axis, as indicated in Fig. 12(*a*). Hydrogen-bonding interactions involving the phenolic groups and acetate O atoms result in linked parallel chains [Fig. 12(*b*)]. The remaining phenolic groups interact with disordered dioxane solvent molecules.

In $[Cu_2(Hhba)_2(OAc)_2(DABCO)_2]\cdot 10(\text{dioxane})$ (**11**), two *trans*-acetate anions remain coordinated, with coordinated DABCO molecules occupying the axial positions in the complex. The $[Cu_2(Hhba)_2(OAc)_2(DABCO)_2]$ unit serves as a 4-connecting node within a 2D 4,4-network, as depicted in Fig. 13(*a*), with hydrogen bonds formed between the phenolic groups and the non-coordinated DABCO N atom. Not surprisingly, there is a pronounced bend in the connection between the complexes associated with the C—O—H bond angle. The 2D layers stack on top of each other, with the methyl groups of the acetate ion extending above and below the mean plane of the 2D network [Fig. 13(*b*)]. Dioxane solvent molecules occupy the voids between neighbouring sheets.

The final compound described in this article is the 1:1 cocrystal formed from the combination of DABCO and

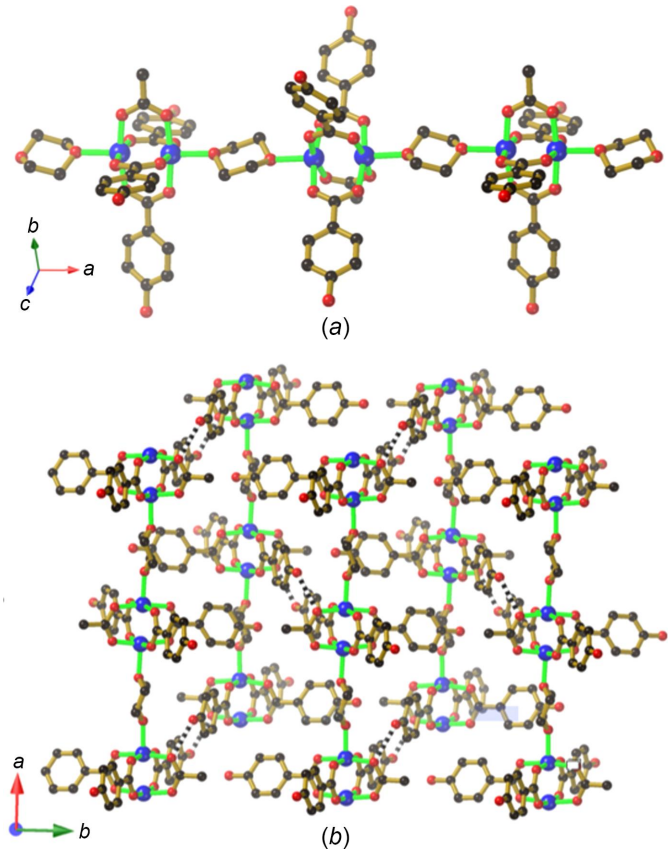


Figure 12
The linear chain structure of $[Cu_2(Hhba)_3(OAc)(dioxane)]\cdot 3.5(dioxane)$ (**10**), showing (*a*) the chain formed by bridging dioxane ligands that extends along the *a* axis and (*b*) the hydrogen-bonded corrugated sheet network. Non-coordinated dioxane molecules have been omitted.

H_2hba, which was used as a reactant in the formation of compound **4**.

The crystal structure determination indicates two DABCO and two 4-hydroxybenzoic acid molecules in the asymmetric unit. Hydrogen-bonded chains that extend in the $[10\bar{1}]$ direction are generated as indicated in Fig. 14. Each chain contains only one type of DABCO and 4-hydroxybenzoic acid molecule. The orientations of the 4-hydroxybenzoic acid in the two crystallographically distinct chains, as represented in Fig. 14, are opposite to each other and thus the two chains are antiparallel. The cocrystal adopts the centrosymmetric space group $P2_1/n$ and thus each chain depicted in Fig. 14 also has a symmetry-related counterpart which is antiparallel.

In the structure refinement, peaks of electron density near C atoms were consistent with the expected positions for H atoms. Similarly, electron-density peaks corresponding to the phenolic H atom appeared at a distance consistent with an O—H covalent bond. For each of the two carboxylic acid groups, peaks of electron density are located at positions further from the O atom than expected based upon normal O—H covalent bonds. These peaks were assigned as H atoms and the position of each was refined, leading to O···H separations of 1.28 (3) Å. The assigned H atoms are 1.25 (3) and 1.24 (3) Å from the N atoms of the neighbouring DABCO molecules. The two N···O separations of 2.518 (2) and 2.515 (2) Å are remarkably short for N···O hydrogen bonds. The N···O separations between the phenolic O atom and the neighbouring N atom of the DABCO are significantly longer [2.640 (2) and 2.619 (2) Å], but still relatively short for an N···O hydrogen bond.

4. General structural comments

For the Cu dimer compounds considered, compound **1** is the only one not to form a network-based material involving coordinate and/or hydrogen-bond interactions. The failure of **1** to form a network may be attributed to the dioxane molecules which bind to the phenolic groups of the neutral $Cu_2(Hhba)_4$ units and block any prospect of network formation. In compounds **7**–**11**, the Cu dimer unit serves as a single node within a 2D (**7**, **10** and **11**) or 3D (**8** and **9**) network, whilst in **2**–**6**, there are two types of nodes.

In compounds **2**–**6**, the Cu dimer serves as a node, with a second node formed from either the aggregation of phenolic and phenolate groups (**2**–**5**) or, alternatively, from the involvement of nitrate anions (**6**). In the networks involving two types of nodes (**2**–**6**), cations are present in the structure. In the structures of **2**, **3**, **5** and **6**, the cations occupy square-shaped cavities, whereas in **4**, a proton shared between the

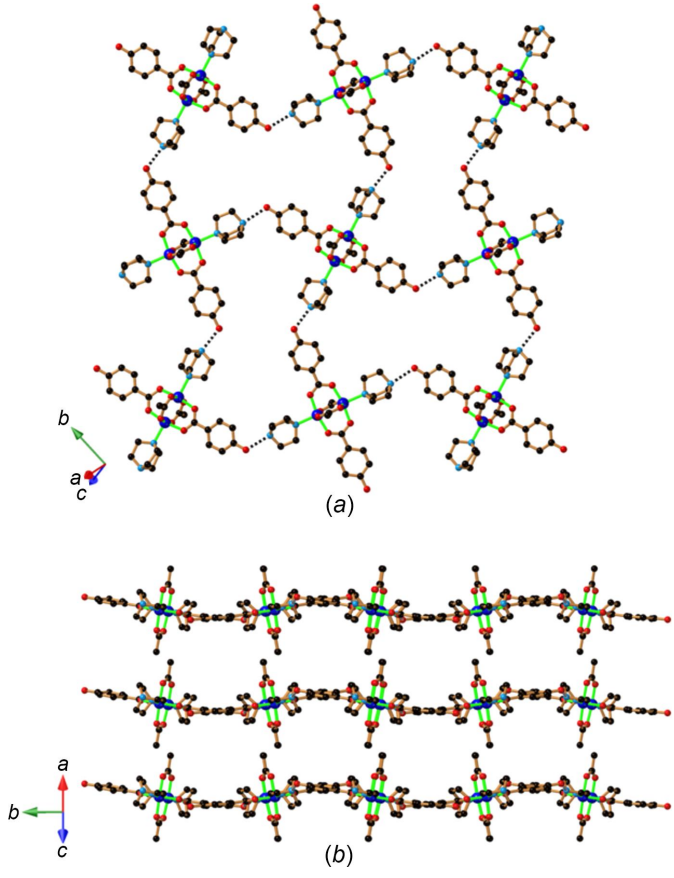


Figure 13
The structure of $[Cu_2(Hhba)_2(OAc)_2(DABCO)_2]\cdot 10(dioxane)$ (**11**), showing (*a*) the 2D hydrogen-bonded network and (*b*) the crystal packing of the sheets.

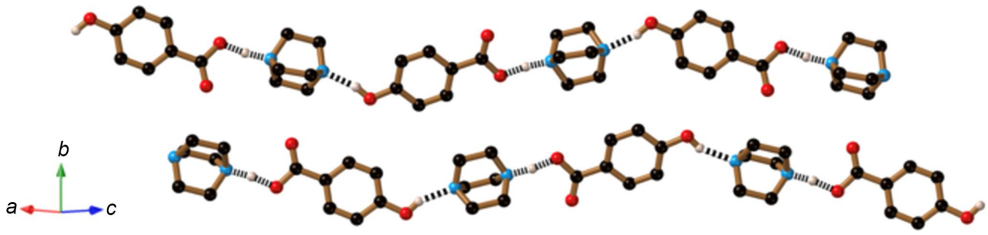


Figure 14
The crystal structure of $(H_{0.5}DABCO)(H_{1.5}hba)$ (**12**), showing the formation of hydrogen-bonded chains that extend in the $[10\bar{1}]$ direction.

non-coordinated N atoms of DABCO ligands serves as a link between Cu dimer nodes.

An average charge of 1− per Cu dimer unit appears to favour the formation of a second type of node in the hydrogen-bonded networks that are generated. In each of **2**–**5**, four O atoms are held together by three H atoms. Whilst the hydrogen-bonded nodes formed in **2**–**4** are similar, consisting of a planar zigzag arrangement of O atoms with a short hydrogen bond between the central two O atoms, the corresponding node in **5** consists of a clearly defined phenolate O atom serving as a hydrogen-bond acceptor from three phenol groups. In the remaining structures (**7**–**11**), the phenol groups only serve to provide links between Cu dimer nodes.

In compounds **8**–**11**, the Cu dimer unit is neutral, with all hba ligands present as $Hhba^-$. These circumstances appear to favour single node (Cu dimer) networks with phenolic groups facilitating links between nodes rather than forming nodes based upon the aggregation of phenolic groups. Although four phenolic groups are involved in a second type of node in **6**, the nitrate ion pairs play a key role in the generation of the second type of node.

Compound **7** represents a special case in which the Cu dimer has a 2− charge, *i.e.* $[Cu_2(Hhba)_2(hba)_2]^{2-}$. This dianion is also present in **5**, but the presence of equal numbers of neutral $[Cu_2(Hhba)_4]$ means that the average charge on a Cu dimer in **5** is 1−. On the basis of the other structures, it might be expected that the presence of phenolic and phenolate groups in **7** would promote the formation of a second type of node, but instead, only one type of node is apparent with double ligand bridges between the Cu dimer nodes. A possible explanation for the adoption of the network, which is depicted in Fig. 9(*b*), is the complementary interaction between pairs of face-to-face $Hbdn^+$ cations and the anionic network containing appropriately sized cavities.

Compounds **10** and **11** represent rare examples of mixed carboxylate systems for binuclear copper(II) dimers. According to Hassanein *et al.* (2015), of the 1300 Cu carboxylate dimers reported, only nine were heteroleptic. In the specific case of acetate-containing dimers, the CSD contains just one compound (refcode HIQQEE02; Luo *et al.*, 2010) with one acetate ligand in combination with three other ferrocenecarboxylate ligands.

In the structures described in this investigation, there are examples of H atoms refining to a position halfway or nearly halfway between a pair of atoms which form a hydrogen bond. The limitations of X-ray diffraction data in accurately determining the positions of H atoms make it difficult to distinguish between disordered asymmetric hydrogen bonds and truly symmetric hydrogen bonds. In the cases where we have refined the structure with a symmetric (or close to symmetric) hydrogen bond, we wish to stress that the assignments of the H-atom positions are only tentative.

5. Conclusion

The network structures described in this work demonstrate that Cu dimer units involving the hba^{2-}/$Hhba^-$ ligands can form a variety of networks. Clearly, hydrogen bonding has played a major role in the formation of the networks. Of particular importance has been the network nodes formed from multiple hydrogen bonds. In compounds **2**–**4**, the planar zigzag motif has shown itself to be an effective 4-connecting node with the ligands extending outwards to the corners of an approximate square. A different type of hydrogen-bonding motif involving three phenol groups serving as donors to a single phenolate group can also produce a 4-connecting node, as seen in **5**.

An unusual and beautiful example of a hydrogen-bonding node is apparent in **6**, where a pair of nitrate anions, four phenol groups and a pair of coordinated water molecules combine to generate a network which accommodates tetraethylammonium cations in square windows. In the remaining structures involving copper dimer complexes, hydrogen bonding plays a key structural role in the arrangement of the molecular components although they do not contain hydrogen-bonded nodes within networks.

Clearly there are numerous opportunities for exploiting the ability of the phenolic/phenolate groups within Cu^{II} tetracarboxylate dimers to create a wide variety of extended structures. Furthermore, the work has demonstrated, using coligands such as DABCO/$HDABCO^+$ and O-bipy, that additional connectivity of the Cu dimer can be achieved by utilizing the axial positions on the Cu centres. The use of different countercations provides further opportunities for crystal engineering. For these reasons, and given the diversity of structures described in this current work, there would appear to be plenty of scope for extending this family of compounds.

Acknowledgements

This research was undertaken in part using the MX1 and MX2 beamlines at the Australian Synchrotron (Aragão *et al.*, 2018; Cowieson *et al.*, 2015; McPhillips *et al.*, 2002), part of ANSTO,

and made use of the Australian Cancer Research Foundation (ACRF) detector. Open access publishing facilitated by The University of Melbourne, as part of the Wiley–The University of Melbourne agreement via the Council of Australian University Librarians.

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supporting information

Acta Cryst. (2024). C**80**, 239-253 [https://doi.org/10.1107/S2053229624004534]

Supramolecular hydrogen-bonded networks formed from copper(II) carboxylate dimers

Brendan F. Abrahams, Christopher J. Commons, Timothy A. Hudson and Robin Sanchez-Arlt

Computing details

Tetrakis(*μ*-4-hydroxybenzoato)bis[(dioxane)copper(II)] dioxane tetrasolvate (compound_1)

Crystal data

$[Cu_2(C_7H_5O_3)_4(C_4H_8O_2)_2]\cdot 4C_4H_8O_2$
$M_r = 1204.14$
Orthorhombic, *Pccn*
$a = 27.7701\ (8)$ Å
$b = 21.2278\ (4)$ Å
$c = 9.3888\ (1)$ Å
$V = 5534.7\ (2)$ Å^3
$Z = 4$
$F(000) = 2520$
$D_x = 1.445$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 4835 reflections
$\theta = 5.2–74.7°$
$\mu = 1.66$ mm^{-1}
$T = 100$ K
Irregular, clear blue
0.14 × 0.13 × 0.05 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
$T_{min} = 0.567$, $T_{max} = 1.000$
18728 measured reflections
5586 independent reflections
4112 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 77.9°$, $\theta_{min} = 2.6°$
$h = -35\rightarrow28$
$k = -26\rightarrow19$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.187$
$S = 1.04$
5586 reflections
354 parameters
2 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0975P)^2 + 4.3605P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.50401 (2)	0.45526 (2)	0.59237 (5)	0.0458 (2)
O7	0.50598 (10)	0.37930 (11)	0.7532 (2)	0.0490 (6)
O4	0.54939 (10)	0.41558 (10)	0.4607 (2)	0.0505 (6)
O5	0.54229 (10)	0.49330 (10)	0.2997 (2)	0.0518 (6)
O6	0.66319 (11)	0.29296 (12)	−0.0478 (3)	0.0599 (7)
H6	0.668464	0.312064	−0.124535	0.090*
O2	0.54782 (10)	0.58260 (11)	0.5194 (2)	0.0529 (6)
O8	0.47914 (12)	0.27813 (11)	0.9315 (3)	0.0571 (7)
O3	0.67261 (11)	0.71553 (12)	0.9950 (3)	0.0608 (7)
H3	0.673537	0.752619	0.963883	0.091*
O9	0.68611 (11)	0.83420 (12)	0.8980 (3)	0.0612 (7)
O1	0.55547 (11)	0.50408 (11)	0.6795 (2)	0.0543 (7)
O11	0.69134 (11)	0.34846 (14)	−0.2826 (3)	0.0616 (7)
O12	0.72613 (16)	0.43718 (16)	−0.4748 (4)	0.0933 (12)
C9	0.58665 (14)	0.40313 (15)	0.2377 (3)	0.0441 (8)
C10	0.60184 (14)	0.34260 (15)	0.2709 (3)	0.0446 (8)
H10	0.594393	0.325537	0.361853	0.054*
C2	0.59535 (15)	0.59955 (16)	0.7219 (3)	0.0507 (9)
C1	0.56448 (15)	0.55920 (16)	0.6338 (4)	0.0496 (9)
C16	0.48813 (16)	0.33951 (16)	0.9880 (3)	0.0515 (9)
H16A	0.522070	0.342274	1.019622	0.062*
H16B	0.467273	0.346584	1.072004	0.062*
C14	0.59734 (15)	0.42700 (16)	0.1026 (3)	0.0499 (9)
H14	0.587022	0.468190	0.077453	0.060*
C3	0.61863 (15)	0.57564 (17)	0.8414 (4)	0.0535 (9)
H3A	0.616246	0.531981	0.862540	0.064*
C8	0.55766 (14)	0.44018 (16)	0.3394 (3)	0.0464 (8)
C13	0.62292 (15)	0.39108 (17)	0.0049 (4)	0.0547 (9)
H13	0.630185	0.407712	−0.086565	0.066*
C12	0.63782 (14)	0.33080 (16)	0.0413 (4)	0.0474 (8)
C4	0.64526 (16)	0.61450 (17)	0.9303 (4)	0.0548 (9)
H4	0.661817	0.597262	1.009843	0.066*
C11	0.62742 (14)	0.30678 (16)	0.1752 (3)	0.0477 (8)
H11	0.637937	0.265716	0.200681	0.057*
C7	0.59887 (17)	0.66394 (17)	0.6935 (4)	0.0581 (10)
H7	0.583379	0.680893	0.611805	0.070*
O10	0.7010 (2)	0.9435 (2)	0.7379 (7)	0.144 (3)
C18	0.49903 (16)	0.31738 (16)	0.6977 (4)	0.0525 (9)
H18A	0.521433	0.310258	0.617282	0.063*

H18B	0.465720	0.313232	0.661358	0.063*
C15	0.47853 (16)	0.38964 (16)	0.8791 (3)	0.0517 (9)
H15A	0.443826	0.389838	0.854902	0.062*
H15B	0.486774	0.431350	0.919453	0.062*
C5	0.64764 (16)	0.67873 (17)	0.9027 (4)	0.0548 (9)
C17	0.50779 (16)	0.26872 (17)	0.8114 (4)	0.0568 (10)
H17A	0.501140	0.226371	0.771792	0.068*
H17B	0.542108	0.270035	0.839761	0.068*
C6	0.62463 (17)	0.70335 (17)	0.7826 (4)	0.0600 (11)
H6A	0.626689	0.747099	0.762227	0.072*
C19	0.64733 (17)	0.8696 (2)	0.8392 (4)	0.0654 (11)
H19A	0.619030	0.866925	0.903022	0.078*
H19B	0.638175	0.851694	0.745667	0.078*
C22	0.72803 (17)	0.8409 (3)	0.8115 (6)	0.0833 (15)
H22A	0.721990	0.822756	0.716070	0.100*
H22B	0.755268	0.817883	0.855291	0.100*
C23	0.74174 (18)	0.3544 (2)	−0.3066 (6)	0.0742 (12)
H23A	0.751884	0.325377	−0.383462	0.089*
H23B	0.759605	0.343188	−0.218927	0.089*
C26	0.6644 (2)	0.3658 (2)	−0.4058 (5)	0.0791 (14)
H26A	0.629503	0.361967	−0.385441	0.095*
H26B	0.672262	0.336957	−0.485339	0.095*
C24	0.7528 (2)	0.4210 (2)	−0.3480 (6)	0.0830 (14)
H24A	0.743885	0.449817	−0.269421	0.100*
H24B	0.787774	0.425563	−0.366055	0.100*
C25	0.6757 (2)	0.4309 (3)	−0.4466 (7)	0.0988 (19)
H25A	0.657138	0.442523	−0.532673	0.119*
H25B	0.666371	0.459876	−0.368743	0.119*
C20	0.6614 (3)	0.9360 (2)	0.8214 (8)	0.129 (3)
H20A	0.634225	0.959429	0.778626	0.155*
H20B	0.667934	0.954400	0.916282	0.155*
C21	0.7406 (2)	0.9099 (3)	0.7976 (9)	0.138 (3)
H21A	0.748471	0.927386	0.892523	0.166*
H21B	0.769217	0.914722	0.735716	0.166*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0770 (4)	0.0316 (3)	0.0288 (3)	0.0038 (2)	0.0171 (2)	0.00427 (18)
O7	0.0881 (19)	0.0312 (11)	0.0277 (11)	0.0018 (11)	0.0178 (11)	0.0022 (9)
O4	0.0803 (18)	0.0385 (12)	0.0327 (11)	0.0105 (11)	0.0152 (11)	0.0054 (9)
O5	0.0864 (18)	0.0348 (12)	0.0343 (11)	0.0113 (11)	0.0193 (12)	0.0066 (9)
O6	0.086 (2)	0.0487 (14)	0.0447 (13)	0.0097 (13)	0.0227 (14)	−0.0031 (11)
O2	0.0821 (18)	0.0397 (12)	0.0368 (12)	−0.0028 (11)	0.0169 (12)	0.0059 (10)
O8	0.098 (2)	0.0358 (12)	0.0373 (12)	−0.0020 (12)	0.0150 (13)	0.0033 (10)
O3	0.091 (2)	0.0485 (15)	0.0430 (13)	−0.0062 (14)	0.0130 (13)	−0.0039 (11)
O9	0.0794 (19)	0.0479 (14)	0.0564 (15)	−0.0014 (13)	−0.0054 (14)	0.0073 (12)
O1	0.0881 (19)	0.0365 (12)	0.0382 (11)	−0.0033 (12)	0.0110 (12)	0.0067 (9)

O11	0.0657 (17)	0.0747 (18)	0.0443 (13)	−0.0046 (14)	0.0104 (13)	0.0094 (12)
O12	0.133 (3)	0.075 (2)	0.072 (2)	−0.037 (2)	0.010 (2)	0.0163 (17)
C9	0.066 (2)	0.0341 (16)	0.0323 (15)	0.0000 (14)	0.0100 (15)	−0.0006 (12)
C10	0.065 (2)	0.0381 (17)	0.0310 (14)	0.0016 (15)	0.0073 (15)	−0.0004 (12)
C2	0.081 (3)	0.0379 (17)	0.0330 (15)	−0.0011 (16)	0.0170 (17)	−0.0005 (13)
C1	0.077 (2)	0.0359 (16)	0.0363 (16)	0.0016 (15)	0.0221 (17)	−0.0010 (13)
C16	0.085 (3)	0.0401 (18)	0.0296 (15)	0.0044 (16)	0.0098 (16)	0.0025 (13)
C14	0.072 (2)	0.0388 (17)	0.0385 (16)	0.0045 (16)	0.0157 (17)	0.0024 (14)
C3	0.076 (3)	0.0411 (18)	0.0440 (18)	0.0019 (17)	0.0187 (18)	0.0001 (15)
C8	0.069 (2)	0.0379 (17)	0.0322 (15)	0.0028 (15)	0.0098 (15)	0.0022 (13)
C13	0.078 (3)	0.0478 (19)	0.0379 (17)	0.0017 (18)	0.0205 (17)	0.0021 (15)
C12	0.064 (2)	0.0386 (17)	0.0399 (16)	0.0029 (15)	0.0118 (16)	−0.0042 (13)
C4	0.075 (3)	0.049 (2)	0.0405 (18)	0.0026 (18)	0.0159 (17)	−0.0002 (15)
C11	0.066 (2)	0.0392 (17)	0.0378 (16)	0.0035 (15)	0.0065 (16)	−0.0017 (13)
C7	0.096 (3)	0.045 (2)	0.0341 (16)	−0.0017 (19)	0.0130 (18)	0.0027 (14)
O10	0.150 (4)	0.088 (3)	0.196 (6)	−0.060 (3)	−0.110 (4)	0.082 (3)
C18	0.093 (3)	0.0305 (16)	0.0337 (16)	0.0031 (16)	0.0123 (17)	−0.0009 (13)
C15	0.086 (3)	0.0406 (18)	0.0290 (15)	0.0057 (17)	0.0151 (17)	−0.0009 (13)
C5	0.079 (3)	0.0469 (19)	0.0391 (17)	−0.0038 (18)	0.0196 (17)	−0.0061 (15)
C17	0.099 (3)	0.0354 (17)	0.0365 (17)	0.0028 (17)	0.0112 (18)	0.0011 (14)
C6	0.103 (3)	0.0396 (18)	0.0372 (17)	−0.0054 (19)	0.0167 (19)	−0.0006 (14)
C19	0.074 (3)	0.072 (3)	0.051 (2)	0.014 (2)	0.001 (2)	−0.0067 (19)
C22	0.058 (3)	0.094 (4)	0.099 (4)	−0.003 (2)	−0.002 (3)	0.043 (3)
C23	0.070 (3)	0.075 (3)	0.078 (3)	−0.002 (2)	0.008 (2)	0.006 (2)
C26	0.093 (4)	0.073 (3)	0.072 (3)	−0.017 (3)	−0.023 (3)	0.019 (2)
C24	0.081 (3)	0.076 (3)	0.092 (4)	−0.018 (3)	−0.001 (3)	0.006 (3)
C25	0.123 (5)	0.073 (3)	0.101 (4)	−0.019 (3)	−0.039 (4)	0.030 (3)
C20	0.217 (9)	0.040 (3)	0.131 (6)	0.019 (4)	−0.102 (6)	−0.020 (3)
C21	0.106 (5)	0.129 (5)	0.180 (7)	−0.052 (4)	−0.078 (5)	0.099 (5)

Geometric parameters (Å, º)

Cu1—Cu1^{i}	2.5818 (9)	C13—H13	0.9500
Cu1—O7	2.210 (2)	C13—C12	1.388 (5)
Cu1—O4	1.956 (2)	C12—C11	1.387 (5)
Cu1—O5^{i}	1.968 (2)	C4—H4	0.9500
Cu1—O2^{i}	1.954 (3)	C4—C5	1.389 (5)
Cu1—O1	1.946 (3)	C11—H11	0.9500
O7—C18	1.427 (4)	C7—H7	0.9500
O7—C15	1.423 (4)	C7—C6	1.383 (6)
O4—C8	1.273 (4)	O10—C20	1.359 (10)
O5—C8	1.262 (4)	O10—C21	1.424 (7)
O6—H6	0.8400	C18—H18A	0.9900
O6—C12	1.357 (4)	C18—H18B	0.9900
O2—C1	1.270 (4)	C18—C17	1.505 (5)
O8—C16	1.429 (4)	C15—H15A	0.9900
O8—C17	1.395 (4)	C15—H15B	0.9900
O3—H3	0.8400	C5—C6	1.397 (6)

O3—C5	1.357 (5)	C17—H17A	0.9900
O9—C19	1.425 (5)	C17—H17B	0.9900
O9—C22	1.426 (5)	C6—H6A	0.9500
O1—C1	1.271 (4)	C19—H19A	0.9900
O11—C23	1.423 (5)	C19—H19B	0.9900
O11—C26	1.426 (5)	C19—C20	1.471 (8)
O12—C24	1.445 (6)	C22—H22A	0.9900
O12—C25	1.431 (7)	C22—H22B	0.9900
C9—C10	1.388 (5)	C22—C21	1.511 (8)
C9—C14	1.397 (4)	C23—H23A	0.9900
C9—C8	1.476 (5)	C23—H23B	0.9900
C10—H10	0.9500	C23—C24	1.498 (7)
C10—C11	1.375 (5)	C26—H26A	0.9900
C2—C1	1.467 (5)	C26—H26B	0.9900
C2—C3	1.391 (5)	C26—C25	1.469 (7)
C2—C7	1.396 (5)	C24—H24A	0.9900
C16—H16A	0.9900	C24—H24B	0.9900
C16—H16B	0.9900	C25—H25A	0.9900
C16—C15	1.500 (5)	C25—H25B	0.9900
C14—H14	0.9500	C20—H20A	0.9900
C14—C13	1.389 (5)	C20—H20B	0.9900
C3—H3A	0.9500	C21—H21A	0.9900
C3—C4	1.387 (6)	C21—H21B	0.9900

O7—Cu1—Cu1^{i}	176.39 (8)	O7—C18—H18B	109.5
O4—Cu1—Cu1^{i}	87.01 (7)	O7—C18—C17	110.6 (3)
O4—Cu1—O7	95.85 (9)	H18A—C18—H18B	108.1
O4—Cu1—O5^{i}	170.18 (9)	C17—C18—H18A	109.5
O5^{i}—Cu1—Cu1^{i}	83.17 (7)	C17—C18—H18B	109.5
O5^{i}—Cu1—O7	93.97 (9)	O7—C15—C16	111.2 (3)
O2^{i}—Cu1—Cu1^{i}	83.00 (7)	O7—C15—H15A	109.4
O2^{i}—Cu1—O7	94.89 (10)	O7—C15—H15B	109.4
O2^{i}—Cu1—O4	87.58 (11)	C16—C15—H15A	109.4
O2^{i}—Cu1—O5^{i}	91.34 (12)	C16—C15—H15B	109.4
O1—Cu1—Cu1^{i}	87.37 (7)	H15A—C15—H15B	108.0
O1—Cu1—O7	94.76 (10)	O3—C5—C4	118.0 (4)
O1—Cu1—O4	91.26 (12)	O3—C5—C6	122.2 (3)
O1—Cu1—O5^{i}	88.16 (12)	C4—C5—C6	119.7 (4)
O1—Cu1—O2^{i}	170.35 (10)	O8—C17—C18	112.5 (3)
C18—O7—Cu1	114.77 (19)	O8—C17—H17A	109.1
C15—O7—Cu1	116.20 (19)	O8—C17—H17B	109.1
C15—O7—C18	111.9 (3)	C18—C17—H17A	109.1
C8—O4—Cu1	120.3 (2)	C18—C17—H17B	109.1
C8—O5—Cu1^{i}	124.4 (2)	H17A—C17—H17B	107.8
C12—O6—H6	109.5	C7—C6—C5	119.9 (3)
C1—O2—Cu1^{i}	124.2 (2)	C7—C6—H6A	120.0
C17—O8—C16	109.3 (3)	C5—C6—H6A	120.0
C5—O3—H3	109.5	O9—C19—H19A	109.6

C19—O9—C22	110.1 (3)	O9—C19—H19B	109.6
C1—O1—Cu1	119.5 (3)	O9—C19—C20	110.4 (5)
C23—O11—C26	111.4 (3)	H19A—C19—H19B	108.1
C25—O12—C24	109.1 (4)	C20—C19—H19A	109.6
C10—C9—C14	118.4 (3)	C20—C19—H19B	109.6
C10—C9—C8	120.9 (3)	O9—C22—H22A	109.8
C14—C9—C8	120.7 (3)	O9—C22—H22B	109.8
C9—C10—H10	119.3	O9—C22—C21	109.6 (5)
C11—C10—C9	121.4 (3)	H22A—C22—H22B	108.2
C11—C10—H10	119.3	C21—C22—H22A	109.8
C3—C2—C1	120.9 (3)	C21—C22—H22B	109.8
C3—C2—C7	118.6 (4)	O11—C23—H23A	109.9
C7—C2—C1	120.3 (3)	O11—C23—H23B	109.9
O2—C1—O1	125.0 (4)	O11—C23—C24	109.1 (4)
O2—C1—C2	117.5 (3)	H23A—C23—H23B	108.3
O1—C1—C2	117.5 (3)	C24—C23—H23A	109.9
O8—C16—H16A	109.4	C24—C23—H23B	109.9
O8—C16—H16B	109.4	O11—C26—H26A	109.7
O8—C16—C15	111.3 (3)	O11—C26—H26B	109.7
H16A—C16—H16B	108.0	O11—C26—C25	110.0 (4)
C15—C16—H16A	109.4	H26A—C26—H26B	108.2
C15—C16—H16B	109.4	C25—C26—H26A	109.7
C9—C14—H14	119.7	C25—C26—H26B	109.7
C13—C14—C9	120.6 (3)	O12—C24—C23	109.5 (4)
C13—C14—H14	119.7	O12—C24—H24A	109.8
C2—C3—H3A	119.5	O12—C24—H24B	109.8
C4—C3—C2	121.1 (3)	C23—C24—H24A	109.8
C4—C3—H3A	119.5	C23—C24—H24B	109.8
O4—C8—C9	117.3 (3)	H24A—C24—H24B	108.2
O5—C8—O4	124.7 (3)	O12—C25—C26	110.2 (5)
O5—C8—C9	118.0 (3)	O12—C25—H25A	109.6
C14—C13—H13	120.1	O12—C25—H25B	109.6
C12—C13—C14	119.8 (3)	C26—C25—H25A	109.6
C12—C13—H13	120.1	C26—C25—H25B	109.6
O6—C12—C13	123.3 (3)	H25A—C25—H25B	108.1
O6—C12—C11	116.7 (3)	O10—C20—C19	113.1 (6)
C11—C12—C13	120.0 (3)	O10—C20—H20A	109.0
C3—C4—H4	120.1	O10—C20—H20B	109.0
C3—C4—C5	119.8 (4)	C19—C20—H20A	109.0
C5—C4—H4	120.1	C19—C20—H20B	109.0
C10—C11—C12	119.8 (3)	H20A—C20—H20B	107.8
C10—C11—H11	120.1	O10—C21—C22	109.9 (4)
C12—C11—H11	120.1	O10—C21—H21A	109.7
C2—C7—H7	119.6	O10—C21—H21B	109.7
C6—C7—C2	120.8 (4)	C22—C21—H21A	109.7
C6—C7—H7	119.6	C22—C21—H21B	109.7
C20—O10—C21	109.8 (5)	H21A—C21—H21B	108.2
O7—C18—H18A	109.5		

Cu1—O7—C18—C17	172.2 (3)	C14—C9—C10—C11	0.9 (6)
Cu1—O7—C15—C16	−172.0 (3)	C14—C9—C8—O4	−179.8 (4)
Cu1—O4—C8—O5	7.6 (5)	C14—C9—C8—O5	1.2 (6)
Cu1—O4—C8—C9	−171.3 (3)	C14—C13—C12—O6	−179.5 (4)
Cu1^{i}—O5—C8—O4	−7.7 (6)	C14—C13—C12—C11	−0.4 (6)
Cu1^{i}—O5—C8—C9	171.2 (2)	C3—C2—C1—O2	−173.9 (3)
Cu1^{i}—O2—C1—O1	11.5 (5)	C3—C2—C1—O1	7.9 (5)
Cu1^{i}—O2—C1—C2	−166.6 (2)	C3—C2—C7—C6	−0.5 (6)
Cu1—O1—C1—O2	−11.9 (5)	C3—C4—C5—O3	177.7 (3)
Cu1—O1—C1—C2	166.2 (2)	C3—C4—C5—C6	−2.3 (6)
O7—C18—C17—O8	55.9 (5)	C8—C9—C10—C11	177.7 (4)
O6—C12—C11—C10	180.0 (4)	C8—C9—C14—C13	−177.3 (4)
O8—C16—C15—O7	−56.0 (5)	C13—C12—C11—C10	0.8 (6)
O3—C5—C6—C7	−178.8 (4)	C4—C5—C6—C7	1.3 (6)
O9—C19—C20—O10	57.5 (6)	C7—C2—C1—O2	11.1 (5)
O9—C22—C21—O10	−58.0 (8)	C7—C2—C1—O1	−167.1 (3)
O11—C23—C24—O12	−58.8 (6)	C7—C2—C3—C4	−0.6 (6)
O11—C26—C25—O12	58.7 (6)	C18—O7—C15—C16	53.5 (4)
C9—C10—C11—C12	−1.1 (6)	C15—O7—C18—C17	−52.7 (4)
C9—C14—C13—C12	0.3 (6)	C17—O8—C16—C15	57.8 (4)
C10—C9—C14—C13	−0.6 (6)	C19—O9—C22—C21	56.7 (5)
C10—C9—C8—O4	3.5 (6)	C22—O9—C19—C20	−55.6 (6)
C10—C9—C8—O5	−175.5 (4)	C23—O11—C26—C25	−58.2 (6)
C2—C3—C4—C5	2.0 (6)	C26—O11—C23—C24	58.1 (5)
C2—C7—C6—C5	0.2 (6)	C24—O12—C25—C26	−60.1 (6)
C1—C2—C3—C4	−175.7 (3)	C25—O12—C24—C23	60.0 (6)
C1—C2—C7—C6	174.6 (4)	C20—O10—C21—C22	58.2 (8)
C16—O8—C17—C18	−58.0 (4)	C21—O10—C20—C19	−58.5 (7)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Tetraethylammonium aquatris(*μ*-4-hydroxybenzoato)methanol(*μ*-4-oxidobenzoato)dicopper(II) dioxane disolvate (compound_2)

Crystal data

$(C_8H_{20}N)[Cu_2(C_7H_5O_3)_2(C_7H_{4.5}O_3)_2(CH_4O)(H_2O)]\cdot 2C_4H_8O_2$
M_r = 1031.02
Triclinic, $P\overline{1}$
a = 10.1659 (4) Å
b = 10.7383 (6) Å
c = 12.9878 (4) Å
α = 76.132 (4)°
β = 69.646 (3)°
γ = 75.304 (4)°
V = 1267.74 (10) Å^3
Z = 1
$F(000)$ = 540
D_x = 1.350 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 7244 reflections
θ = 3.7–77.8°
μ = 1.63 mm^{-1}
T = 100 K
Irregular, clear blue
0.17 × 0.12 × 0.05 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.885, T_{max} = 1.000
17988 measured reflections
5262 independent reflections
4672 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 78.0°, θ_{min} = 5.8°
h = −12→12
k = −13→13
l = −13→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.200
S = 1.06
5262 reflections
459 parameters
419 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1206P)^2 + 1.2732P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −1.00 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. Occupancies of the axial sites of the complex are shared almost equally by H2O and CH3OH molecules. The NEt4 group is disordered across a scentre of inversion and is in PART -1. Both 1,4-dioxane molecules are disordered and their disordered components have been placed in parts.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.55520 (4)	0.10317 (4)	0.44807 (3)	0.03624 (19)	
O6	−0.1090 (2)	0.2374 (3)	0.18088 (19)	0.0451 (5)	
H6	−0.083253	0.289897	0.121568	0.068*	
O3	−0.0352 (3)	0.4006 (2)	0.99674 (18)	0.0455 (5)	
O5	0.3102 (3)	−0.0326 (2)	0.46488 (19)	0.0430 (5)	
O8	0.6421 (3)	0.2782 (2)	0.3614 (2)	0.0450 (5)	0.5
H8A	0.718610	0.258591	0.306740	0.067*	
H8B	0.693510	0.293540	0.397200	0.067*	0.5
O2	0.3211 (2)	0.0083 (2)	0.66128 (18)	0.0424 (5)	
O1	0.4181 (3)	0.1857 (2)	0.5731 (2)	0.0520 (6)	
O4	0.4064 (3)	0.1441 (3)	0.3743 (2)	0.0514 (6)	
C12	−0.0038 (3)	0.2006 (3)	0.2307 (3)	0.0405 (7)	
C1	0.3299 (3)	0.1261 (3)	0.6517 (3)	0.0390 (7)	
C13	−0.0043 (4)	0.0848 (4)	0.3072 (3)	0.0440 (7)	
H13	−0.075254	0.034120	0.322676	0.053*	
C2	0.2303 (3)	0.2005 (3)	0.7402 (3)	0.0405 (7)	
C8	0.3148 (3)	0.0736 (3)	0.3971 (3)	0.0401 (7)	
C9	0.2033 (3)	0.1182 (3)	0.3399 (3)	0.0397 (7)	
C5	0.0506 (4)	0.3380 (3)	0.9122 (3)	0.0424 (7)	

C14	0.0996 (4)	0.0446 (3)	0.3603 (3)	0.0423 (7)	
H14	0.100262	−0.034953	0.411719	0.051*	
C3	0.2446 (4)	0.3258 (3)	0.7414 (3)	0.0478 (8)	
H3A	0.316244	0.365169	0.682944	0.057*	
C6	0.0339 (4)	0.2132 (4)	0.9099 (3)	0.0494 (8)	
H6A	−0.039305	0.174616	0.967266	0.059*	
C11	0.0988 (4)	0.2758 (4)	0.2101 (3)	0.0468 (8)	
H11	0.098196	0.355615	0.158966	0.056*	
C10	0.2006 (4)	0.2342 (4)	0.2640 (3)	0.0479 (8)	
H10	0.270647	0.285612	0.249253	0.057*	
C7	0.1223 (4)	0.1456 (4)	0.8255 (3)	0.0468 (8)	
H7	0.109673	0.060998	0.825413	0.056*	
C4	0.1565 (4)	0.3940 (3)	0.8260 (3)	0.0480 (8)	
H4	0.168138	0.479203	0.825243	0.058*	
N1	0.5009 (17)	0.0024 (14)	1.0087 (13)	0.0599 (19)	0.5
C28	0.4884 (15)	0.1146 (13)	1.0638 (9)	0.098 (4)	0.5
H28A	0.473903	0.196396	1.010975	0.117*	0.5
H28B	0.401301	0.116172	1.129078	0.117*	0.5
C29	0.601 (2)	0.1155 (18)	1.0992 (11)	0.110 (5)	0.5
H29A	0.661689	0.028477	1.102014	0.166*	0.5
H29B	0.564592	0.139682	1.173625	0.166*	0.5
H29C	0.657728	0.179149	1.047189	0.166*	0.5
C30	0.5447 (9)	−0.1286 (9)	1.0730 (7)	0.061 (2)	0.5
H30A	0.553635	−0.196311	1.029685	0.073*	0.5
H30B	0.640639	−0.132763	1.078163	0.073*	0.5
C31	0.4511 (16)	−0.162 (2)	1.1841 (15)	0.065 (4)	0.5
H31A	0.442541	−0.096612	1.228873	0.097*	0.5
H31B	0.490697	−0.248096	1.218277	0.097*	0.5
H31C	0.356621	−0.162182	1.180436	0.097*	0.5
C24	0.3558 (15)	0.0152 (14)	0.9974 (18)	0.141 (7)	0.5
H24A	0.285781	0.040750	1.067429	0.169*	0.5
H24B	0.345776	0.090811	0.937786	0.169*	0.5
C25	0.3076 (19)	−0.0882 (17)	0.9745 (17)	0.137 (8)	0.5
H25A	0.302477	−0.161189	1.036869	0.206*	0.5
H25B	0.374806	−0.118281	0.906230	0.206*	0.5
H25C	0.212723	−0.055201	0.964780	0.206*	0.5
C26	0.6057 (15)	0.0192 (11)	0.8945 (7)	0.090 (4)	0.5
H26A	0.701856	0.001752	0.903545	0.107*	0.5
H26B	0.603984	−0.050203	0.856995	0.107*	0.5
C27	0.592 (2)	0.142 (2)	0.8181 (16)	0.090 (7)	0.5
H27A	0.670504	0.137026	0.748239	0.135*	0.5
H27B	0.595785	0.213382	0.851822	0.135*	0.5
H27C	0.500650	0.159704	0.802766	0.135*	0.5
H3	0.000000	0.500000	1.000000	0.09 (2)*	
O14	0.5243 (8)	0.4043 (7)	0.9358 (6)	0.0728 (17)	0.5
C34	0.398 (2)	0.4956 (17)	0.9555 (18)	0.077 (4)	0.5
H34A	0.319904	0.456490	0.955359	0.093*	0.5
H34B	0.408427	0.571531	0.894432	0.093*	0.5

C32	0.3596 (13)	0.5413 (12)	1.0644 (11)	0.080 (3)	0.5
H32A	0.271056	0.608322	1.074551	0.096*	0.5
H32B	0.342807	0.467156	1.126593	0.096*	0.5
O13	0.4625 (11)	0.3789 (10)	1.0541 (10)	0.128 (4)	0.5
C35	0.438 (2)	0.563 (2)	0.9224 (19)	0.154 (10)	0.5
H35A	0.363689	0.635523	0.902420	0.184*	0.5
H35B	0.490819	0.522107	0.855438	0.184*	0.5
C33	0.371 (4)	0.475 (3)	1.001 (3)	0.172 (18)	0.5
H33A	0.326123	0.430674	0.967518	0.206*	0.5
H33B	0.294405	0.520442	1.058562	0.206*	0.5
C15	0.6890 (17)	0.3335 (11)	0.4300 (10)	0.098 (4)	0.5
H15A	0.607134	0.361106	0.492573	0.148*	0.5
H15B	0.731581	0.409197	0.385658	0.148*	0.5
H15C	0.760219	0.267967	0.458586	0.148*	0.5
O7	0.6421 (3)	0.2782 (2)	0.3614 (2)	0.0450 (5)	0.5
O10	0.8206 (14)	0.4688 (14)	0.6446 (14)	0.071 (3)	0.25
O9	0.748 (3)	0.385 (3)	0.489 (3)	0.070 (3)	0.25
C17	0.6706 (18)	0.496 (2)	0.6509 (17)	0.071 (3)	0.25
H17A	0.620951	0.426873	0.702692	0.085*	0.25
H17B	0.621969	0.581239	0.675776	0.085*	0.25
C16	0.674 (3)	0.500 (3)	0.5373 (18)	0.073 (3)	0.25
H16A	0.574587	0.518662	0.535516	0.088*	0.25
H16B	0.719192	0.574521	0.489595	0.088*	0.25
C19	0.886 (2)	0.347 (2)	0.5048 (18)	0.074 (3)	0.25
H19A	0.949676	0.397624	0.441985	0.089*	0.25
H19B	0.919569	0.254449	0.494465	0.089*	0.25
C18	0.914 (2)	0.355 (2)	0.6033 (19)	0.079 (3)	0.25
H18A	0.896272	0.276315	0.659827	0.095*	0.25
H18B	1.014992	0.363034	0.585653	0.095*	0.25
O11	0.741 (4)	0.403 (3)	0.480 (3)	0.071 (3)	0.25
C20	0.637 (2)	0.480 (3)	0.5570 (19)	0.072 (3)	0.25
H20A	0.564277	0.428706	0.607319	0.087*	0.25
H20B	0.588377	0.557561	0.515925	0.087*	0.25
C23	0.825 (2)	0.306 (2)	0.5402 (18)	0.073 (3)	0.25
H23A	0.914974	0.272411	0.484547	0.088*	0.25
H23B	0.773344	0.232200	0.574225	0.088*	0.25
C22	0.866 (2)	0.336 (2)	0.6286 (18)	0.075 (3)	0.25
H22A	0.887819	0.253077	0.678298	0.090*	0.25
H22B	0.955753	0.371003	0.593313	0.090*	0.25
C21	0.702 (3)	0.523 (2)	0.6224 (19)	0.075 (3)	0.25
H21A	0.777256	0.570931	0.570094	0.090*	0.25
H21B	0.628310	0.586357	0.666357	0.090*	0.25
O12	0.7639 (17)	0.4268 (14)	0.6966 (14)	0.078 (3)	0.25

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0303 (3)	0.0357 (3)	0.0339 (3)	0.00316 (18)	−0.00424 (19)	−0.00778 (18)

O6	0.0370 (11)	0.0558 (14)	0.0363 (11)	−0.0026 (10)	−0.0081 (9)	−0.0072 (10)
O3	0.0450 (12)	0.0474 (13)	0.0348 (11)	0.0027 (10)	−0.0036 (9)	−0.0149 (9)
O5	0.0455 (12)	0.0401 (11)	0.0414 (12)	−0.0041 (9)	−0.0159 (10)	−0.0030 (9)
O8	0.0398 (12)	0.0388 (11)	0.0436 (12)	−0.0010 (9)	−0.0011 (9)	−0.0074 (9)
O2	0.0391 (11)	0.0456 (12)	0.0363 (11)	−0.0056 (9)	−0.0014 (9)	−0.0134 (9)
O1	0.0507 (14)	0.0361 (12)	0.0460 (13)	0.0018 (10)	0.0092 (11)	−0.0110 (10)
O4	0.0393 (12)	0.0496 (14)	0.0600 (15)	−0.0088 (10)	−0.0218 (11)	0.0112 (11)
C12	0.0330 (14)	0.0474 (17)	0.0339 (14)	0.0029 (12)	−0.0055 (12)	−0.0119 (12)
C1	0.0352 (15)	0.0411 (16)	0.0336 (14)	0.0043 (12)	−0.0074 (12)	−0.0097 (12)
C13	0.0429 (17)	0.0484 (18)	0.0375 (16)	−0.0086 (14)	−0.0078 (13)	−0.0079 (13)
C2	0.0367 (15)	0.0423 (16)	0.0353 (15)	0.0028 (12)	−0.0072 (12)	−0.0097 (12)
C8	0.0323 (14)	0.0415 (16)	0.0369 (15)	0.0039 (12)	−0.0040 (12)	−0.0102 (12)
C9	0.0342 (15)	0.0407 (16)	0.0362 (15)	0.0015 (12)	−0.0047 (12)	−0.0097 (12)
C5	0.0394 (16)	0.0486 (17)	0.0323 (14)	0.0023 (13)	−0.0054 (12)	−0.0134 (13)
C14	0.0441 (17)	0.0410 (16)	0.0362 (15)	−0.0037 (13)	−0.0082 (13)	−0.0066 (12)
C3	0.0461 (18)	0.0410 (17)	0.0401 (17)	0.0031 (14)	0.0020 (14)	−0.0119 (13)
C6	0.0462 (18)	0.056 (2)	0.0393 (17)	−0.0112 (15)	0.0013 (14)	−0.0161 (15)
C11	0.0387 (17)	0.0439 (17)	0.0497 (18)	−0.0009 (13)	−0.0119 (14)	−0.0026 (14)
C10	0.0391 (17)	0.0446 (18)	0.0539 (19)	−0.0048 (14)	−0.0144 (15)	−0.0001 (15)
C7	0.0453 (18)	0.0514 (19)	0.0405 (17)	−0.0049 (15)	−0.0052 (14)	−0.0179 (14)
C4	0.0484 (19)	0.0392 (16)	0.0440 (17)	0.0002 (14)	−0.0020 (14)	−0.0112 (13)
N1	0.051 (3)	0.066 (3)	0.056 (5)	0.016 (2)	−0.020 (3)	−0.020 (3)
C28	0.140 (11)	0.088 (7)	0.049 (5)	−0.015 (7)	−0.002 (6)	−0.029 (5)
C29	0.165 (16)	0.118 (11)	0.070 (7)	−0.035 (11)	−0.068 (9)	0.000 (7)
C30	0.055 (4)	0.070 (5)	0.051 (4)	0.016 (4)	−0.021 (3)	−0.023 (4)
C31	0.055 (7)	0.067 (8)	0.065 (6)	0.010 (6)	−0.022 (5)	−0.016 (5)
C24	0.077 (7)	0.098 (9)	0.241 (18)	−0.020 (7)	−0.092 (10)	0.052 (10)
C25	0.118 (12)	0.147 (14)	0.168 (16)	−0.070 (12)	−0.100 (12)	0.069 (13)
C26	0.115 (9)	0.076 (6)	0.047 (4)	0.031 (6)	−0.010 (5)	−0.028 (4)
C27	0.105 (16)	0.080 (10)	0.061 (7)	0.025 (10)	−0.024 (8)	−0.018 (6)
O14	0.070 (4)	0.068 (4)	0.080 (4)	−0.004 (3)	−0.021 (3)	−0.023 (3)
C34	0.072 (8)	0.053 (8)	0.122 (12)	−0.013 (6)	−0.045 (9)	−0.019 (9)
C32	0.059 (6)	0.065 (6)	0.099 (8)	−0.011 (5)	−0.004 (5)	−0.014 (6)
O13	0.104 (7)	0.107 (7)	0.166 (11)	−0.052 (6)	−0.056 (8)	0.047 (7)
C35	0.17 (2)	0.138 (15)	0.19 (2)	−0.065 (15)	−0.140 (18)	0.055 (15)
C33	0.105 (17)	0.096 (15)	0.32 (5)	−0.043 (13)	−0.12 (2)	0.07 (2)
C15	0.136 (11)	0.069 (7)	0.088 (8)	−0.049 (7)	0.002 (7)	−0.029 (6)
O7	0.0398 (12)	0.0388 (11)	0.0436 (12)	−0.0010 (9)	−0.0011 (9)	−0.0074 (9)
O10	0.064 (5)	0.063 (5)	0.087 (6)	0.014 (4)	−0.028 (4)	−0.033 (5)
O9	0.067 (5)	0.063 (6)	0.074 (5)	0.006 (4)	−0.011 (4)	−0.036 (5)
C17	0.069 (5)	0.063 (6)	0.076 (6)	0.010 (5)	−0.021 (5)	−0.029 (5)
C16	0.070 (6)	0.065 (6)	0.078 (5)	0.010 (5)	−0.018 (5)	−0.030 (5)
C19	0.068 (5)	0.059 (6)	0.079 (6)	0.013 (5)	−0.006 (5)	−0.031 (5)
C18	0.069 (6)	0.065 (6)	0.092 (6)	0.013 (5)	−0.021 (5)	−0.026 (5)
O11	0.069 (5)	0.060 (6)	0.073 (5)	0.008 (4)	−0.009 (4)	−0.030 (5)
C20	0.067 (6)	0.066 (6)	0.076 (5)	0.011 (5)	−0.018 (5)	−0.029 (5)
C23	0.068 (6)	0.060 (6)	0.080 (6)	0.009 (5)	−0.010 (5)	−0.030 (5)
C22	0.064 (6)	0.063 (6)	0.088 (6)	0.013 (5)	−0.017 (5)	−0.030 (5)

C21	0.073 (5)	0.067 (6)	0.084 (6)	0.016 (5)	−0.027 (5)	−0.032 (5)
O12	0.069 (5)	0.068 (6)	0.091 (6)	0.017 (4)	−0.028 (5)	−0.030 (5)

Geometric parameters (Å, º)

Cu1—Cu1^{i}	2.6010 (9)	C24—C25	1.449 (15)
Cu1—O5^{i}	1.965 (2)	C25—H25A	0.9800
Cu1—O8	2.172 (2)	C25—H25B	0.9800
Cu1—O2^{i}	1.960 (2)	C25—H25C	0.9800
Cu1—O1	1.963 (2)	C26—H26A	0.9900
Cu1—O4	1.967 (2)	C26—H26B	0.9900
Cu1—O7	2.172 (2)	C26—C27	1.459 (15)
O6—H6	0.8400	C27—H27A	0.9800
O6—C12	1.365 (4)	C27—H27B	0.9800
O3—C5	1.344 (4)	C27—H27C	0.9800
O3—H3	1.223 (3)	O14—C34	1.39 (2)
O5—C8	1.263 (4)	O14—C32^{ii}	1.442 (15)
O8—H8A	0.8704	C34—H34A	0.9900
O8—H8B	0.8746	C34—H34B	0.9900
O2—C1	1.264 (4)	C34—C32	1.50 (2)
O1—C1	1.264 (4)	C32—H32A	0.9900
O4—C8	1.259 (4)	C32—H32B	0.9900
C12—C13	1.393 (5)	O13—C35^{ii}	1.46 (2)
C12—C11	1.393 (5)	O13—C33	1.42 (3)
C1—C2	1.484 (4)	C35—H35A	0.9900
C13—H13	0.9500	C35—H35B	0.9900
C13—C14	1.382 (5)	C35—C33	1.34 (3)
C2—C3	1.394 (5)	C33—H33A	0.9900
C2—C7	1.396 (5)	C33—H33B	0.9900
C8—C9	1.486 (5)	C15—H15A	0.9800
C9—C14	1.391 (5)	C15—H15B	0.9800
C9—C10	1.391 (5)	C15—H15C	0.9800
C5—C6	1.401 (5)	C15—O7	1.437 (13)
C5—C4	1.394 (5)	O7—H8A	0.8704
C14—H14	0.9500	O10—C17	1.454 (14)
C3—H3A	0.9500	O10—C18	1.430 (14)
C3—C4	1.383 (5)	O9—C16	1.434 (15)
C6—H6A	0.9500	O9—C19	1.431 (16)
C6—C7	1.379 (5)	C17—H17A	0.9900
C11—H11	0.9500	C17—H17B	0.9900
C11—C10	1.371 (5)	C17—C16	1.453 (17)
C10—H10	0.9500	C16—H16A	0.9900
C7—H7	0.9500	C16—H16B	0.9900
C4—H4	0.9500	C19—H19A	0.9900
N1—C28	1.501 (14)	C19—H19B	0.9900
N1—C30	1.500 (13)	C19—C18	1.433 (17)
N1—C24	1.501 (14)	C18—H18A	0.9900
N1—C26	1.496 (14)	C18—H18B	0.9900

C28—H28A	0.9900	O11—C20	1.426 (16)
C28—H28B	0.9900	O11—C23	1.436 (16)
C28—C29	1.380 (15)	C20—H20A	0.9900
C29—H29A	0.9800	C20—H20B	0.9900
C29—H29B	0.9800	C20—C21	1.449 (17)
C29—H29C	0.9800	C23—H23A	0.9900
C30—H30A	0.9900	C23—H23B	0.9900
C30—H30B	0.9900	C23—C22	1.476 (17)
C30—C31	1.443 (14)	C22—H22A	0.9900
C31—H31A	0.9800	C22—H22B	0.9900
C31—H31B	0.9800	C22—O12	1.439 (14)
C31—H31C	0.9800	C21—H21A	0.9900
C24—H24A	0.9900	C21—H21B	0.9900
C24—H24B	0.9900	C21—O12	1.417 (15)

O5^{i}—Cu1—Cu1^{i}	84.72 (7)	C25—C24—H24B	106.3
O5^{i}—Cu1—O8	96.44 (10)	C24—C25—H25A	109.5
O5^{i}—Cu1—O4	169.41 (10)	C24—C25—H25B	109.5
O5^{i}—Cu1—O7	96.44 (10)	C24—C25—H25C	109.5
O8—Cu1—Cu1^{i}	178.59 (7)	H25A—C25—H25B	109.5
O2^{i}—Cu1—Cu1^{i}	84.69 (7)	H25A—C25—H25C	109.5
O2^{i}—Cu1—O5^{i}	88.27 (10)	H25B—C25—H25C	109.5
O2^{i}—Cu1—O8	96.14 (9)	N1—C26—H26A	107.2
O2^{i}—Cu1—O1	169.50 (10)	N1—C26—H26B	107.2
O2^{i}—Cu1—O4	90.21 (11)	H26A—C26—H26B	106.9
O2^{i}—Cu1—O7	96.14 (9)	C27—C26—N1	120.4 (12)
O1—Cu1—Cu1^{i}	84.85 (8)	C27—C26—H26A	107.2
O1—Cu1—O5^{i}	89.95 (11)	C27—C26—H26B	107.2
O1—Cu1—O8	94.34 (10)	C26—C27—H27A	109.5
O1—Cu1—O4	89.64 (12)	C26—C27—H27B	109.5
O1—Cu1—O7	94.34 (10)	C26—C27—H27C	109.5
O4—Cu1—Cu1^{i}	84.70 (8)	H27A—C27—H27B	109.5
O4—Cu1—O8	94.14 (10)	H27A—C27—H27C	109.5
O4—Cu1—O7	94.14 (10)	H27B—C27—H27C	109.5
C12—O6—H6	109.5	C34—O14—C32^{ii}	110.4 (10)
C5—O3—H3	114.8 (2)	O14—C34—H34A	109.2
C8—O5—Cu1^{i}	122.7 (2)	O14—C34—H34B	109.2
Cu1—O8—H8A	109.4	O14—C34—C32	112.2 (14)
Cu1—O8—H8B	109.6	H34A—C34—H34B	107.9
H8A—O8—H8B	90.7	C32—C34—H34A	109.2
C1—O2—Cu1^{i}	122.8 (2)	C32—C34—H34B	109.2
C1—O1—Cu1	122.5 (2)	O14^{ii}—C32—C34	108.5 (11)
C8—O4—Cu1	122.7 (2)	O14^{ii}—C32—H32A	110.0
O6—C12—C13	117.3 (3)	O14^{ii}—C32—H32B	110.0
O6—C12—C11	122.6 (3)	C34—C32—H32A	110.0
C11—C12—C13	120.1 (3)	C34—C32—H32B	110.0
O2—C1—C2	117.8 (3)	H32A—C32—H32B	108.4
O1—C1—O2	125.1 (3)	C33—O13—C35^{ii}	111.5 (16)

O1—C1—C2	117.1 (3)	O13^{ii}—C35—H35A	107.6
C12—C13—H13	120.4	O13^{ii}—C35—H35B	107.6
C14—C13—C12	119.2 (3)	H35A—C35—H35B	107.0
C14—C13—H13	120.4	C33—C35—H35A	107.6
C3—C2—C1	120.6 (3)	C33—C35—H35B	107.6
C3—C2—C7	118.4 (3)	O13—C33—H33A	108.8
C7—C2—C1	120.9 (3)	O13—C33—H33B	108.8
O5—C8—C9	117.2 (3)	C35—C33—O13	114 (2)
O4—C8—O5	125.1 (3)	C35—C33—H33A	108.8
O4—C8—C9	117.7 (3)	C35—C33—H33B	108.8
C14—C9—C8	120.8 (3)	H33A—C33—H33B	107.7
C14—C9—C10	118.4 (3)	H15A—C15—H15B	109.5
C10—C9—C8	120.8 (3)	H15A—C15—H15C	109.5
O3—C5—C6	119.6 (3)	H15B—C15—H15C	109.5
O3—C5—C4	121.7 (3)	O7—C15—H15A	109.5
C4—C5—C6	118.7 (3)	O7—C15—H15B	109.5
C13—C14—C9	121.3 (3)	O7—C15—H15C	109.5
C13—C14—H14	119.3	Cu1—O7—H8A	109.4
C9—C14—H14	119.3	C15—O7—Cu1	113.1 (5)
C2—C3—H3A	119.4	C15—O7—H8A	104.8
C4—C3—C2	121.2 (3)	C18—O10—C17	120.7 (17)
C4—C3—H3A	119.4	C19—O9—C16	109 (2)
C5—C6—H6A	119.6	O10—C17—H17A	111.0
C7—C6—C5	120.8 (3)	O10—C17—H17B	111.0
C7—C6—H6A	119.6	H17A—C17—H17B	109.0
C12—C11—H11	120.1	C16—C17—O10	103.7 (17)
C10—C11—C12	119.7 (3)	C16—C17—H17A	111.0
C10—C11—H11	120.1	C16—C17—H17B	111.0
C9—C10—H10	119.3	O9—C16—C17	117 (3)
C11—C10—C9	121.3 (3)	O9—C16—H16A	108.1
C11—C10—H10	119.3	O9—C16—H16B	108.1
C2—C7—H7	119.7	C17—C16—H16A	108.1
C6—C7—C2	120.6 (3)	C17—C16—H16B	108.1
C6—C7—H7	119.7	H16A—C16—H16B	107.3
C5—C4—H4	119.9	O9—C19—H19A	106.2
C3—C4—C5	120.2 (3)	O9—C19—H19B	106.2
C3—C4—H4	119.9	O9—C19—C18	124.3 (19)
C28—N1—C24	105.1 (13)	H19A—C19—H19B	106.4
C30—N1—C28	113.5 (12)	C18—C19—H19A	106.2
C30—N1—C24	110.9 (12)	C18—C19—H19B	106.2
C26—N1—C28	108.9 (11)	O10—C18—C19	107.2 (16)
C26—N1—C30	109.8 (11)	O10—C18—H18A	110.3
C26—N1—C24	108.4 (14)	O10—C18—H18B	110.3
N1—C28—H28A	108.0	C19—C18—H18A	110.3
N1—C28—H28B	108.0	C19—C18—H18B	110.3
H28A—C28—H28B	107.2	H18A—C18—H18B	108.5
C29—C28—N1	117.2 (13)	C20—O11—C23	107 (2)
C29—C28—H28A	108.0	O11—C20—H20A	109.4

C29—C28—H28B	108.0	O11—C20—H20B	109.4
C28—C29—H29A	109.5	O11—C20—C21	111 (2)
C28—C29—H29B	109.5	H20A—C20—H20B	108.0
C28—C29—H29C	109.5	C21—C20—H20A	109.4
H29A—C29—H29B	109.5	C21—C20—H20B	109.4
H29A—C29—H29C	109.5	O11—C23—H23A	106.9
H29B—C29—H29C	109.5	O11—C23—H23B	106.9
N1—C30—H30A	108.2	O11—C23—C22	121.6 (19)
N1—C30—H30B	108.2	H23A—C23—H23B	106.7
H30A—C30—H30B	107.3	C22—C23—H23A	106.9
C31—C30—N1	116.6 (10)	C22—C23—H23B	106.9
C31—C30—H30A	108.2	C23—C22—H22A	108.1
C31—C30—H30B	108.2	C23—C22—H22B	108.1
C30—C31—H31A	109.5	H22A—C22—H22B	107.3
C30—C31—H31B	109.5	O12—C22—C23	117.0 (16)
C30—C31—H31C	109.5	O12—C22—H22A	108.1
H31A—C31—H31B	109.5	O12—C22—H22B	108.1
H31A—C31—H31C	109.5	C20—C21—H21A	107.8
H31B—C31—H31C	109.5	C20—C21—H21B	107.8
N1—C24—H24A	106.3	H21A—C21—H21B	107.2
N1—C24—H24B	106.3	O12—C21—C20	118 (2)
H24A—C24—H24B	106.4	O12—C21—H21A	107.8
C25—C24—N1	124.1 (13)	O12—C21—H21B	107.8
C25—C24—H24A	106.3	C21—O12—C22	105.2 (17)

Cu1^{i}—O5—C8—O4	2.4 (5)	C11—C12—C13—C14	−1.3 (5)
Cu1^{i}—O5—C8—C9	−177.73 (19)	C10—C9—C14—C13	−0.2 (5)
Cu1^{i}—O2—C1—O1	−0.7 (5)	C7—C2—C3—C4	1.4 (6)
Cu1^{i}—O2—C1—C2	−179.9 (2)	C4—C5—C6—C7	1.5 (6)
Cu1—O1—C1—O2	1.9 (5)	C28—N1—C30—C31	59.9 (19)
Cu1—O1—C1—C2	−179.0 (2)	C28—N1—C24—C25	−164.0 (17)
Cu1—O4—C8—O5	−3.2 (5)	C28—N1—C26—C27	−51 (2)
Cu1—O4—C8—C9	177.0 (2)	C30—N1—C28—C29	49.3 (17)
O6—C12—C13—C14	−179.4 (3)	C30—N1—C24—C25	−41 (2)
O6—C12—C11—C10	179.1 (3)	C30—N1—C26—C27	−175.9 (17)
O3—C5—C6—C7	−178.6 (3)	C24—N1—C28—C29	170.7 (13)
O3—C5—C4—C3	178.8 (3)	C24—N1—C30—C31	−58.1 (19)
O5—C8—C9—C14	−0.5 (4)	C24—N1—C26—C27	63 (2)
O5—C8—C9—C10	180.0 (3)	C26—N1—C28—C29	−73.3 (16)
O2—C1—C2—C3	172.9 (3)	C26—N1—C30—C31	−177.9 (15)
O2—C1—C2—C7	−5.7 (5)	C26—N1—C24—C25	80 (2)
O1—C1—C2—C3	−6.4 (5)	O14—C34—C32—O14^{ii}	57.8 (17)
O1—C1—C2—C7	175.0 (3)	C32^{ii}—O14—C34—C32	−58.8 (18)
O4—C8—C9—C14	179.4 (3)	O13^{ii}—C35—C33—O13	−46 (5)
O4—C8—C9—C10	−0.2 (5)	C35^{ii}—O13—C33—C35	42 (5)
C12—C13—C14—C9	0.8 (5)	O10—C17—C16—O9	58 (4)
C12—C11—C10—C9	−0.4 (6)	O9—C19—C18—O10	−34 (3)
C1—C2—C3—C4	−177.2 (3)	C17—O10—C18—C19	45 (3)

C1—C2—C7—C6	177.4 (3)	C16—O9—C19—C18	38 (4)
C13—C12—C11—C10	1.1 (5)	C19—O9—C16—C17	−50 (4)
C2—C3—C4—C5	−0.1 (6)	C18—O10—C17—C16	−57 (3)
C8—C9—C14—C13	−179.7 (3)	O11—C20—C21—O12	−66 (4)
C8—C9—C10—C11	179.5 (3)	O11—C23—C22—O12	35 (4)
C5—C6—C7—C2	−0.2 (6)	C20—O11—C23—C22	−38 (4)
C14—C9—C10—C11	0.0 (5)	C20—C21—O12—C22	56 (3)
C3—C2—C7—C6	−1.2 (5)	C23—O11—C20—C21	49 (4)
C6—C5—C4—C3	−1.3 (6)	C23—C22—O12—C21	−39 (3)

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

Triaqua(dioxane)lithium tris(*μ*-4-hydroxybenzoato)(*μ*-4-oxidobenzoato)bis[aquacopper(II)] dioxane disolvate monohydrate (compound_3)

Crystal data

$[Li(C_4H_8O_2)(H_2O)_3]$ $[Cu_2(C_7H_5O_3)_2(C_7H_{4.5}O_3)_2(H_2O)_2]\cdot 2C_4H_8O_2\cdot H_2O$
M_r = 1047.81
Triclinic, $P\overline{1}$
a = 9.7711 (8) Å
b = 10.1924 (6) Å
c = 12.5863 (7) Å
α = 73.615 (5)°
β = 79.528 (6)°
γ = 83.412 (6)°
V = 1179.86 (15) Å^3
Z = 1
F(000) = 542
D_x = 1.475 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 3510 reflections
θ = 4.6–75.1°
μ = 1.85 mm^{-1}
T = 100 K
Irregular, clear blue
0.14 × 0.07 × 0.03 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2023)
T_{min} = 0.610, T_{max} = 1.000
10093 measured reflections
4145 independent reflections
3048 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 68.2°, θ_{min} = 4.6°
h = −11→11
k = −12→12
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.089
$wR(F^2)$ = 0.261
S = 1.09
4145 reflections
338 parameters
42 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1435P)^2 + 2.3152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.21 e Å^{-3}
$\Delta\rho_{min}$ = −1.32 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. The H atoms on the disordered water molecules containing O11 and O13 were not modelled. A Li atom and 3 coordinated H2O molecules are disordered across a centre of inversion and placed in Part -1. A water molecule (containing O13) is present when this Li group is absent.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.38413 (8)	0.44096 (8)	1.04454 (7)	0.0407 (3)	
O4	0.3050 (4)	0.5680 (4)	0.9199 (3)	0.0399 (9)	
O3	0.7321 (5)	−0.1046 (4)	0.6841 (3)	0.0462 (10)	
O2	0.6606 (5)	0.4217 (4)	0.8704 (3)	0.0477 (10)	
O6	0.1137 (4)	1.0334 (5)	0.5107 (3)	0.0481 (11)	
O5	0.5051 (4)	0.6668 (5)	0.8416 (4)	0.0609 (14)	
O1	0.4633 (5)	0.3197 (4)	0.9507 (5)	0.0602 (13)	
O7	0.2001 (5)	0.3273 (5)	1.1236 (5)	0.0673 (15)	
H7A	0.173763	0.283559	1.077023	0.101*	
H7B	0.217203	0.255346	1.183695	0.101*	
C9	0.3070 (6)	0.7543 (5)	0.7571 (4)	0.0366 (12)	
C5	0.6987 (6)	0.0029 (6)	0.7287 (5)	0.0439 (14)	
C8	0.3781 (6)	0.6559 (5)	0.8459 (4)	0.0392 (13)	
C2	0.6203 (7)	0.2170 (6)	0.8296 (5)	0.0464 (14)	
C1	0.5794 (6)	0.3279 (6)	0.8863 (5)	0.0473 (15)	
C10	0.1669 (7)	0.7495 (7)	0.7551 (5)	0.0482 (15)	
H10	0.114814	0.681778	0.810525	0.058*	
C7	0.7338 (7)	0.2261 (7)	0.7429 (5)	0.0525 (16)	
H7	0.785553	0.305501	0.718947	0.063*	
C12	0.1768 (6)	0.9412 (6)	0.5911 (4)	0.0410 (13)	
C14	0.3812 (6)	0.8539 (6)	0.6753 (5)	0.0453 (14)	
H14	0.477583	0.858547	0.675817	0.054*	
C6	0.7714 (7)	0.1217 (7)	0.6921 (5)	0.0515 (16)	
H6A	0.847165	0.130358	0.631925	0.062*	
C4	0.5883 (8)	−0.0078 (6)	0.8167 (6)	0.0578 (18)	
H4	0.539242	−0.088896	0.843512	0.069*	
C13	0.3165 (6)	0.9466 (6)	0.5929 (4)	0.0429 (14)	
H13	0.368694	1.014212	0.537429	0.051*	
C3	0.5500 (8)	0.0985 (6)	0.8652 (6)	0.0590 (18)	
H3A	0.473322	0.090364	0.924595	0.071*	
O8	0.0820 (8)	0.2064 (10)	0.9889 (8)	0.125 (3)	
C11	0.1015 (7)	0.8421 (8)	0.6732 (5)	0.0572 (18)	
H11	0.004893	0.838042	0.672981	0.069*	
O10	0.6328 (9)	0.5374 (9)	0.4925 (8)	0.116 (3)	
O9	0.0984 (11)	0.2964 (12)	0.7549 (9)	0.146 (3)	
C20	0.6142 (13)	0.4296 (12)	0.4539 (10)	0.104 (4)	

H20A	0.696253	0.362893	0.461404	0.125*	
H20B	0.604375	0.462237	0.373547	0.125*	
C17	0.0420 (14)	0.3807 (15)	0.8225 (11)	0.121 (3)	
H17A	−0.031199	0.445081	0.787530	0.145*	
H17B	0.115637	0.435331	0.829864	0.145*	
C18	−0.0188 (13)	0.2999 (15)	0.9347 (10)	0.119 (3)	
H18A	−0.059153	0.362762	0.981288	0.143*	
H18B	−0.095151	0.248610	0.927384	0.143*	
C19	0.5160 (16)	0.6390 (12)	0.4789 (13)	0.118 (4)	
H19A	0.505849	0.673732	0.398712	0.142*	
H19B	0.531282	0.717240	0.506369	0.142*	
C15	0.1383 (18)	0.1230 (17)	0.9084 (15)	0.150 (4)	
H15A	0.209901	0.053351	0.940044	0.180*	
H15B	0.062027	0.074383	0.897454	0.180*	
C16	0.1989 (19)	0.2079 (18)	0.8021 (16)	0.163 (4)	
H16	0.294502	0.202828	0.769955	0.195*	
H3	0.78 (2)	−0.08 (2)	0.619 (8)	0.244*	
H6	0.029 (14)	1.02 (3)	0.51 (3)	0.244*	0.5
O11	0.9549 (12)	0.4163 (10)	0.5358 (8)	0.064 (3)	0.5
O12	0.8884 (13)	0.7314 (10)	0.4357 (7)	0.065 (3)	0.5
H12A	0.925599	0.746213	0.365501	0.097*	0.5
H12B	0.895816	0.807350	0.452242	0.097*	0.5
Li1	0.819 (3)	0.567 (3)	0.5324 (18)	0.058 (6)	0.5
O14	0.7460 (14)	0.6145 (10)	0.6690 (9)	0.065 (3)	0.5
H14A	0.733666	0.559870	0.736424	0.098*	0.5
H14B	0.721000	0.695829	0.678221	0.098*	0.5
O13	0.8171 (17)	0.5735 (14)	0.6329 (10)	0.085 (4)	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0322 (5)	0.0349 (5)	0.0401 (5)	0.0003 (3)	−0.0017 (3)	0.0102 (3)
O4	0.036 (2)	0.041 (2)	0.0295 (18)	0.0003 (16)	0.0006 (15)	0.0068 (15)
O3	0.047 (2)	0.048 (2)	0.0320 (19)	0.0004 (18)	0.0031 (17)	0.0015 (17)
O2	0.048 (2)	0.052 (2)	0.034 (2)	−0.0111 (19)	0.0020 (17)	0.0018 (17)
O6	0.042 (2)	0.060 (2)	0.0263 (18)	−0.0031 (19)	−0.0058 (16)	0.0132 (17)
O5	0.035 (2)	0.054 (3)	0.063 (3)	−0.0014 (18)	−0.009 (2)	0.033 (2)
O1	0.042 (3)	0.041 (2)	0.086 (3)	−0.0046 (18)	0.015 (2)	−0.013 (2)
O7	0.034 (2)	0.054 (3)	0.083 (3)	−0.0033 (19)	−0.002 (2)	0.027 (2)
C9	0.043 (3)	0.034 (3)	0.024 (2)	0.006 (2)	−0.002 (2)	0.001 (2)
C5	0.042 (3)	0.047 (3)	0.032 (3)	−0.001 (2)	−0.002 (2)	0.004 (2)
C8	0.037 (3)	0.038 (3)	0.033 (3)	0.007 (2)	−0.003 (2)	0.002 (2)
C2	0.045 (3)	0.040 (3)	0.041 (3)	−0.001 (2)	0.000 (2)	0.007 (2)
C1	0.044 (3)	0.040 (3)	0.043 (3)	0.000 (3)	−0.009 (3)	0.014 (2)
C10	0.043 (3)	0.056 (3)	0.031 (3)	−0.005 (3)	−0.002 (2)	0.011 (2)
C7	0.056 (4)	0.051 (4)	0.040 (3)	−0.014 (3)	−0.001 (3)	0.004 (3)
C12	0.045 (3)	0.047 (3)	0.022 (2)	0.000 (2)	−0.004 (2)	0.002 (2)
C14	0.036 (3)	0.048 (3)	0.041 (3)	0.000 (2)	−0.003 (2)	0.004 (3)

C6	0.051 (4)	0.062 (4)	0.032 (3)	−0.014 (3)	0.005 (3)	0.000 (3)
C4	0.060 (4)	0.041 (3)	0.055 (4)	−0.008 (3)	0.018 (3)	0.000 (3)
C13	0.041 (3)	0.045 (3)	0.030 (3)	−0.003 (2)	−0.003 (2)	0.008 (2)
C3	0.057 (4)	0.042 (3)	0.061 (4)	−0.006 (3)	0.019 (3)	−0.003 (3)
O8	0.069 (4)	0.161 (6)	0.140 (6)	0.012 (4)	−0.011 (4)	−0.045 (5)
C11	0.037 (3)	0.079 (5)	0.037 (3)	−0.010 (3)	−0.005 (2)	0.017 (3)
O10	0.095 (5)	0.108 (6)	0.159 (8)	0.001 (4)	−0.029 (5)	−0.056 (5)
O9	0.117 (6)	0.190 (7)	0.144 (6)	−0.016 (5)	0.038 (5)	−0.101 (6)
C20	0.116 (9)	0.103 (8)	0.089 (7)	0.030 (7)	0.002 (6)	−0.045 (6)
C17	0.097 (6)	0.166 (8)	0.119 (6)	0.009 (6)	−0.005 (5)	−0.083 (6)
C18	0.076 (5)	0.181 (7)	0.111 (6)	0.030 (5)	−0.012 (5)	−0.075 (6)
C19	0.139 (11)	0.087 (7)	0.135 (11)	0.001 (7)	−0.009 (9)	−0.050 (7)
C15	0.111 (7)	0.142 (8)	0.184 (8)	−0.004 (6)	0.031 (6)	−0.057 (7)
C16	0.129 (8)	0.169 (8)	0.179 (8)	0.002 (6)	0.056 (7)	−0.083 (7)
O11	0.078 (7)	0.052 (5)	0.051 (5)	−0.006 (5)	0.005 (5)	−0.003 (4)
O12	0.095 (8)	0.057 (6)	0.034 (4)	−0.014 (5)	0.007 (5)	−0.008 (4)
Li1	0.072 (16)	0.068 (14)	0.034 (10)	−0.007 (11)	0.000 (10)	−0.018 (10)
O14	0.097 (9)	0.046 (5)	0.041 (5)	−0.015 (6)	0.006 (6)	−0.001 (4)
O13	0.112 (11)	0.079 (8)	0.054 (7)	−0.003 (7)	0.022 (7)	−0.022 (6)

Geometric parameters (Å, º)

Cu1—O4	1.949 (4)	C13—H13	0.9500
Cu1—O2^{i}	1.958 (5)	C3—H3A	0.9500
Cu1—O5^{i}	1.959 (4)	O8—C18	1.415 (15)
Cu1—O1	1.948 (5)	O8—C15	1.492 (16)
Cu1—O7	2.178 (4)	C11—H11	0.9500
O4—C8	1.271 (7)	O10—C20	1.363 (13)
O3—C5	1.349 (8)	O10—C19	1.451 (15)
O3—H3	0.850 (15)	O10—Li1	2.05 (3)
O2—C1	1.263 (8)	O9—C17	1.379 (13)
O6—C12	1.358 (7)	O9—C16	1.36 (2)
O6—H6	0.85 (2)	C20—H20A	0.9900
O5—C8	1.248 (7)	C20—H20B	0.9900
O1—C1	1.265 (8)	C20—C19^{ii}	1.517 (18)
O7—H7A	0.9149	C17—H17A	0.9900
O7—H7B	0.9178	C17—H17B	0.9900
C9—C8	1.491 (7)	C17—C18	1.476 (18)
C9—C10	1.381 (9)	C18—H18A	0.9900
C9—C14	1.387 (8)	C18—H18B	0.9900
C5—C6	1.395 (9)	C19—H19A	0.9900
C5—C4	1.389 (9)	C19—H19B	0.9900
C2—C1	1.481 (9)	C15—H15A	0.9900
C2—C7	1.398 (9)	C15—H15B	0.9900
C2—C3	1.380 (9)	C15—C16	1.44 (2)
C10—H10	0.9500	C16—H16	0.9500
C10—C11	1.384 (8)	O11—Li1	1.91 (3)
C7—H7	0.9500	O12—H12A	0.8696

C7—C6	1.373 (10)	O12—H12B	0.8703
C12—C13	1.377 (9)	O12—Li1	1.89 (3)
C12—C11	1.390 (8)	Li1—O14	1.91 (2)
C14—H14	0.9500	Li1—O13	1.28 (3)
C14—C13	1.384 (8)	O14—H14A	0.8699
C6—H6A	0.9500	O14—H14B	0.8705
C4—H4	0.9500	O14—O13	0.889 (14)
C4—C3	1.372 (10)		

O4—Cu1—O2^{i}	89.60 (17)	C10—C11—H11	119.9
O4—Cu1—O5^{i}	169.14 (17)	C12—C11—H11	119.9
O4—Cu1—O7	97.95 (18)	C20—O10—C19	110.9 (10)
O2^{i}—Cu1—O5^{i}	88.7 (2)	C20—O10—Li1	123.0 (10)
O2^{i}—Cu1—O7	95.2 (2)	C19—O10—Li1	124.9 (10)
O5^{i}—Cu1—O7	92.89 (18)	C16—O9—C17	109.9 (12)
O1—Cu1—O4	90.9 (2)	O10—C20—H20A	109.8
O1—Cu1—O2^{i}	168.96 (18)	O10—C20—H20B	109.8
O1—Cu1—O5^{i}	88.7 (2)	O10—C20—C19^{ii}	109.2 (9)
O1—Cu1—O7	95.7 (2)	H20A—C20—H20B	108.3
C8—O4—Cu1	121.3 (4)	C19^{ii}—C20—H20A	109.8
C5—O3—H3	112 (10)	C19^{ii}—C20—H20B	109.8
C1—O2—Cu1^{i}	120.0 (4)	O9—C17—H17A	109.5
C12—O6—H6	115 (5)	O9—C17—H17B	109.5
C8—O5—Cu1^{i}	124.2 (3)	O9—C17—C18	110.9 (12)
C1—O1—Cu1	126.8 (5)	H17A—C17—H17B	108.0
Cu1—O7—H7A	111.6	C18—C17—H17A	109.5
Cu1—O7—H7B	112.4	C18—C17—H17B	109.5
H7A—O7—H7B	101.9	O8—C18—C17	111.8 (10)
C10—C9—C8	121.2 (5)	O8—C18—H18A	109.3
C10—C9—C14	118.6 (5)	O8—C18—H18B	109.3
C14—C9—C8	120.2 (5)	C17—C18—H18A	109.3
O3—C5—C6	123.2 (5)	C17—C18—H18B	109.3
O3—C5—C4	117.8 (6)	H18A—C18—H18B	107.9
C4—C5—C6	119.0 (6)	O10—C19—C20^{ii}	107.7 (10)
O4—C8—C9	117.7 (5)	O10—C19—H19A	110.2
O5—C8—O4	125.1 (5)	O10—C19—H19B	110.2
O5—C8—C9	117.2 (5)	C20^{ii}—C19—H19A	110.2
C7—C2—C1	121.5 (6)	C20^{ii}—C19—H19B	110.2
C3—C2—C1	120.1 (5)	H19A—C19—H19B	108.5
C3—C2—C7	118.3 (6)	O8—C15—H15A	109.4
O2—C1—O1	124.0 (6)	O8—C15—H15B	109.4
O2—C1—C2	119.7 (5)	H15A—C15—H15B	108.0
O1—C1—C2	116.3 (6)	C16—C15—O8	111.1 (13)
C9—C10—H10	119.6	C16—C15—H15A	109.4
C9—C10—C11	120.8 (5)	C16—C15—H15B	109.4
C11—C10—H10	119.6	O9—C16—C15	109.4 (14)
C2—C7—H7	119.6	O9—C16—H16	125.3
C6—C7—C2	120.8 (6)	C15—C16—H16	125.3

C6—C7—H7	119.6	H12A—O12—H12B	104.5
O6—C12—C13	119.9 (5)	Li1—O12—H12A	127.5
O6—C12—C11	120.8 (6)	Li1—O12—H12B	127.9
C13—C12—C11	119.3 (5)	O11—Li1—O10	114.4 (12)
C9—C14—H14	119.6	O12—Li1—O10	109.6 (13)
C13—C14—C9	120.9 (6)	O12—Li1—O11	111.6 (13)
C13—C14—H14	119.6	O12—Li1—O14	103.7 (12)
C5—C6—H6A	119.9	O14—Li1—O10	97.5 (12)
C7—C6—C5	120.1 (6)	O14—Li1—O11	118.6 (13)
C7—C6—H6A	119.9	O13—Li1—O10	116.5 (17)
C5—C4—H4	119.9	O13—Li1—O11	97.0 (17)
C3—C4—C5	120.2 (6)	O13—Li1—O12	107.0 (16)
C3—C4—H4	119.9	O13—Li1—O14	23.3 (9)
C12—C13—C14	120.2 (5)	Li1—O14—H14A	127.6
C12—C13—H13	119.9	Li1—O14—H14B	127.8
C14—C13—H13	119.9	H14A—O14—H14B	104.5
C2—C3—H3A	119.3	O13—O14—Li1	34.7 (14)
C4—C3—C2	121.4 (6)	O13—O14—H14A	104.0
C4—C3—H3A	119.3	O13—O14—H14B	138.1
C18—O8—C15	103.7 (11)	O14—O13—Li1	122 (2)
C10—C11—C12	120.2 (6)		

Cu1—O4—C8—O5	3.3 (8)	C7—C2—C3—C4	−0.7 (11)
Cu1—O4—C8—C9	−176.6 (4)	C14—C9—C8—O4	178.9 (5)
Cu1^{i}—O2—C1—O1	4.2 (8)	C14—C9—C8—O5	−0.9 (8)
Cu1^{i}—O2—C1—C2	−174.3 (4)	C14—C9—C10—C11	0.0 (10)
Cu1^{i}—O5—C8—O4	−5.0 (9)	C6—C5—C4—C3	1.4 (11)
Cu1^{i}—O5—C8—C9	174.9 (4)	C4—C5—C6—C7	0.0 (10)
Cu1—O1—C1—O2	−2.6 (9)	C13—C12—C11—C10	−0.5 (11)
Cu1—O1—C1—C2	175.9 (4)	C3—C2—C1—O2	164.9 (6)
O3—C5—C6—C7	−178.5 (6)	C3—C2—C1—O1	−13.7 (9)
O3—C5—C4—C3	180.0 (6)	C3—C2—C7—C6	2.1 (10)
O6—C12—C13—C14	179.3 (6)	O8—C15—C16—O9	−64 (2)
O6—C12—C11—C10	−179.5 (6)	C11—C12—C13—C14	0.3 (10)
C9—C10—C11—C12	0.4 (11)	O10—Li1—O13—O14	−38 (3)
C9—C14—C13—C12	0.0 (10)	O9—C17—C18—O8	59.4 (16)
C5—C4—C3—C2	−1.0 (12)	C20—O10—C19—C20^{ii}	60.5 (14)
C8—C9—C10—C11	179.2 (6)	C17—O9—C16—C15	61.7 (18)
C8—C9—C14—C13	−179.5 (5)	C18—O8—C15—C16	59.6 (18)
C2—C7—C6—C5	−1.8 (10)	C19—O10—C20—C19^{ii}	−61.4 (15)
C1—C2—C7—C6	179.2 (6)	C15—O8—C18—C17	−56.5 (14)
C1—C2—C3—C4	−177.9 (7)	C16—O9—C17—C18	−59.2 (16)
C10—C9—C8—O4	−0.3 (8)	O11—Li1—O13—O14	−159.6 (17)
C10—C9—C8—O5	179.8 (6)	O12—Li1—O13—O14	85 (2)
C10—C9—C14—C13	−0.2 (9)	Li1—O10—C20—C19^{ii}	130.4 (12)

C7—C2—C1—O2	−12.2 (9)	Li1—O10—C19—C20^{ii}	−131.6 (12)
C7—C2—C1—O1	169.3 (6)		

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

(1-Aza-4-azoniabicyclo[2.2.2]octane)(1,4-diazabicyclo[2.2.2]octane)tris(*μ*-4-hydroxybenzoato)(*μ*-4-oxidobenzoato)dicopper(II) methanol trisolvate (compound_4)

Crystal data

$[Cu_2(C_7H_5O_3)_2(C_7H_{4.5}O_3)_2(C_6H_{12.5}N_2)_2]\cdot 3CH_4O$
M_r = 995.99
Triclinic, $P\overline{1}$
a = 8.8860 (18) Å
b = 10.702 (2) Å
c = 12.360 (3) Å
α = 73.13 (3)°
β = 73.07 (3)°
γ = 81.68 (3)°
V = 1073.8 (4) Å^3
Z = 2
F(000) = 520
D_x = 1.540 Mg m^{-3}
Synchrotron radiation, *λ* = 0.7108 Å
Cell parameters from 1048 reflections
μ = 1.07 mm^{-1}
T = 100 K
Irregular, dark green
0.18 × 0.11 × 0.07 mm

Data collection

MX1 beamline, Australian Synchrotron diffractometer
Radiation source: MX1 Beamline Australian Synchrotron
Silicon Double Crystal monochromator
Detector resolution: 13.3 pixels mm^{-1}
ω Scan scans
Absorption correction: multi-scan (XDS; Kabsch, 2010)
T_{min} = 0.321, T_{max} = 0.432
15021 measured reflections
3849 independent reflections
2994 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 26.0°, θ_{min} = 2.0°
h = −10→10
k = −13→13
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.089
$wR(F^2)$ = 0.263
S = 1.05
3849 reflections
325 parameters
364 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.171P)^2 + 2.1393P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.042 (8)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.91525 (8)	0.40962 (7)	0.50033 (6)	0.0423 (4)	
O1	0.8428 (6)	0.5494 (4)	0.3804 (4)	0.0574 (12)	

C2	0.8255 (8)	0.7566 (6)	0.2492 (5)	0.0477 (14)	
O2	0.9863 (7)	0.7028 (5)	0.3797 (5)	0.0665 (15)	
C3	0.7122 (9)	0.7192 (7)	0.2095 (6)	0.0607 (18)	
H3	0.678943	0.632476	0.240430	0.073*	
O3	0.6426 (8)	1.0210 (6)	−0.0079 (5)	0.0847 (19)	
H3A	0.543 (5)	1.010 (9)	0.008 (8)	0.127*	0.5
C4	0.6478 (11)	0.8062 (8)	0.1261 (7)	0.074 (2)	
H4	0.568257	0.779865	0.101610	0.089*	
O4	0.7604 (5)	0.4726 (4)	0.6205 (3)	0.0450 (10)	
C5	0.6990 (11)	0.9344 (8)	0.0764 (7)	0.072 (2)	
O5	0.9074 (6)	0.6316 (5)	0.6200 (4)	0.0545 (11)	
C6	0.8155 (11)	0.9693 (8)	0.1147 (7)	0.069 (2)	
H6	0.854603	1.053888	0.080615	0.083*	
O6	0.2991 (10)	0.7280 (8)	1.0103 (6)	0.101 (2)	
C7	0.8749 (9)	0.8828 (7)	0.2012 (6)	0.0603 (18)	
H7	0.951005	0.909894	0.228449	0.072*	
C1	0.8900 (7)	0.6624 (6)	0.3432 (5)	0.0444 (13)	
C9	0.6517 (9)	0.6047 (7)	0.7562 (6)	0.0547 (16)	
C10	0.5215 (9)	0.5350 (9)	0.8104 (7)	0.067 (2)	
H10	0.511431	0.458053	0.790262	0.081*	
C11	0.4014 (11)	0.5794 (11)	0.8975 (8)	0.085 (3)	
H11	0.310126	0.531598	0.937259	0.102*	
C12	0.4177 (13)	0.6931 (10)	0.9244 (7)	0.080 (3)	
C13	0.5484 (13)	0.7608 (9)	0.8695 (8)	0.079 (3)	
H13	0.559052	0.837438	0.889875	0.095*	
C14	0.6642 (11)	0.7198 (8)	0.7853 (7)	0.068 (2)	
H14	0.754270	0.769191	0.745923	0.081*	
C8	0.7799 (8)	0.5664 (6)	0.6607 (5)	0.0481 (15)	
N1	0.7671 (6)	0.2586 (5)	0.4988 (4)	0.0461 (11)	0.555 (7)
C17	0.731 (2)	0.1618 (13)	0.6134 (10)	0.081 (3)	0.555 (7)
H17A	0.678059	0.207125	0.675185	0.097*	0.555 (7)
H17B	0.830428	0.117030	0.630255	0.097*	0.555 (7)
C15	0.8402 (17)	0.1984 (14)	0.4014 (12)	0.086 (3)	0.555 (7)
H15A	0.946828	0.159761	0.406807	0.104*	0.555 (7)
H15B	0.851251	0.266193	0.326017	0.104*	0.555 (7)
C16	0.6165 (13)	0.3210 (11)	0.4750 (15)	0.074 (3)	0.555 (7)
H16A	0.637701	0.385495	0.397365	0.089*	0.555 (7)
H16B	0.560983	0.368217	0.534929	0.089*	0.555 (7)
C20	0.625 (2)	0.0609 (15)	0.6157 (10)	0.082 (3)	0.555 (7)
H20A	0.676906	−0.027865	0.635981	0.098*	0.555 (7)
H20B	0.524460	0.064754	0.676297	0.098*	0.555 (7)
C18	0.7403 (14)	0.0925 (15)	0.4052 (13)	0.089 (3)	0.555 (7)
H18A	0.714610	0.110836	0.329127	0.107*	0.555 (7)
H18B	0.801005	0.006758	0.417889	0.107*	0.555 (7)
C19	0.5127 (14)	0.2189 (8)	0.4768 (14)	0.071 (3)	0.555 (7)
H19A	0.412688	0.220663	0.538407	0.085*	0.555 (7)
H19B	0.487187	0.239789	0.400419	0.085*	0.555 (7)
N2	0.5933 (6)	0.0867 (5)	0.4999 (5)	0.0559 (14)	0.555 (7)

N3	0.7671 (6)	0.2586 (5)	0.4988 (4)	0.0461 (11)	0.445 (7)
C22	0.659 (2)	0.2161 (17)	0.6165 (10)	0.079 (3)	0.445 (7)
H22A	0.587474	0.291190	0.636086	0.095*	0.445 (7)
H22B	0.719355	0.182541	0.675806	0.095*	0.445 (7)
C23	0.8617 (16)	0.1350 (11)	0.4899 (17)	0.071 (3)	0.445 (7)
H23A	0.909291	0.102047	0.556891	0.085*	0.445 (7)
H23B	0.948073	0.150815	0.416766	0.085*	0.445 (7)
C21	0.721 (2)	0.2961 (15)	0.3879 (12)	0.078 (3)	0.445 (7)
H21A	0.816011	0.304454	0.320791	0.094*	0.445 (7)
H21B	0.659043	0.381386	0.379801	0.094*	0.445 (7)
C25	0.562 (2)	0.1089 (17)	0.6177 (10)	0.080 (3)	0.445 (7)
H25A	0.587637	0.026534	0.673075	0.096*	0.445 (7)
H25B	0.448520	0.133583	0.645779	0.096*	0.445 (7)
C26	0.7583 (12)	0.0335 (14)	0.4900 (18)	0.079 (3)	0.445 (7)
H26A	0.799594	0.005350	0.416665	0.095*	0.445 (7)
H26B	0.762038	−0.044203	0.556586	0.095*	0.445 (7)
C24	0.622 (2)	0.1906 (14)	0.3892 (12)	0.087 (3)	0.445 (7)
H24A	0.519977	0.231650	0.375672	0.104*	0.445 (7)
H24B	0.677070	0.150851	0.323891	0.104*	0.445 (7)
N4	0.5933 (6)	0.0867 (5)	0.4999 (5)	0.0559 (14)	0.445 (7)
H2	0.500000	0.000000	0.500000	0.11 (5)*	
H6A	0.276 (19)	0.807 (5)	1.015 (13)	0.160*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0498 (5)	0.0343 (5)	0.0516 (5)	−0.0111 (3)	−0.0270 (3)	−0.0070 (3)
O1	0.075 (3)	0.039 (2)	0.071 (3)	−0.018 (2)	−0.049 (3)	0.003 (2)
C2	0.054 (3)	0.043 (3)	0.050 (3)	−0.001 (3)	−0.022 (3)	−0.011 (3)
O2	0.088 (4)	0.042 (3)	0.084 (3)	−0.018 (2)	−0.061 (3)	0.006 (2)
C3	0.076 (5)	0.050 (4)	0.066 (4)	−0.001 (4)	−0.041 (4)	−0.010 (3)
O3	0.127 (5)	0.063 (3)	0.070 (3)	0.021 (4)	−0.057 (4)	−0.010 (3)
C4	0.100 (6)	0.061 (5)	0.075 (5)	0.002 (4)	−0.056 (5)	−0.011 (4)
O4	0.056 (2)	0.041 (2)	0.043 (2)	−0.0215 (19)	−0.0165 (18)	−0.0070 (17)
C5	0.094 (6)	0.064 (5)	0.057 (4)	0.029 (4)	−0.039 (4)	−0.015 (3)
O5	0.057 (3)	0.056 (3)	0.062 (3)	−0.011 (2)	−0.013 (2)	−0.031 (2)
C6	0.085 (5)	0.053 (4)	0.067 (4)	−0.008 (4)	−0.028 (4)	−0.002 (3)
O6	0.116 (5)	0.098 (5)	0.070 (4)	0.034 (5)	−0.023 (4)	−0.012 (4)
C7	0.070 (4)	0.050 (4)	0.061 (4)	−0.007 (3)	−0.026 (3)	−0.003 (3)
C1	0.047 (3)	0.042 (3)	0.051 (3)	−0.006 (3)	−0.024 (3)	−0.010 (3)
C9	0.065 (4)	0.049 (4)	0.060 (4)	0.002 (3)	−0.035 (3)	−0.013 (3)
C10	0.063 (4)	0.068 (5)	0.068 (5)	−0.002 (4)	−0.025 (4)	−0.007 (4)
C11	0.073 (5)	0.095 (7)	0.068 (5)	0.000 (5)	−0.018 (4)	0.002 (5)
C12	0.105 (7)	0.073 (6)	0.057 (4)	0.019 (5)	−0.037 (5)	−0.005 (4)
C13	0.106 (7)	0.067 (5)	0.071 (5)	0.017 (5)	−0.042 (5)	−0.022 (4)
C14	0.091 (6)	0.058 (4)	0.065 (4)	0.006 (4)	−0.041 (4)	−0.018 (3)
C8	0.052 (3)	0.047 (4)	0.050 (3)	−0.005 (3)	−0.029 (3)	−0.004 (3)
N1	0.052 (3)	0.045 (3)	0.048 (3)	−0.017 (2)	−0.023 (2)	−0.005 (2)

C17	0.094 (6)	0.054 (5)	0.105 (6)	−0.028 (5)	−0.051 (5)	−0.001 (5)
C15	0.092 (5)	0.063 (5)	0.123 (6)	−0.025 (5)	−0.038 (5)	−0.034 (5)
C16	0.076 (5)	0.044 (5)	0.124 (6)	−0.025 (4)	−0.063 (5)	−0.009 (5)
C20	0.092 (6)	0.057 (6)	0.110 (6)	−0.031 (5)	−0.058 (5)	0.001 (5)
C18	0.089 (6)	0.066 (5)	0.129 (6)	−0.025 (5)	−0.034 (5)	−0.035 (5)
C19	0.071 (5)	0.037 (5)	0.124 (7)	−0.019 (4)	−0.060 (5)	−0.010 (5)
N2	0.057 (3)	0.045 (3)	0.076 (4)	−0.017 (3)	−0.026 (3)	−0.016 (3)
N3	0.052 (3)	0.045 (3)	0.048 (3)	−0.017 (2)	−0.023 (2)	−0.005 (2)
C22	0.091 (6)	0.051 (6)	0.108 (6)	−0.026 (5)	−0.045 (5)	−0.012 (5)
C23	0.078 (5)	0.038 (4)	0.118 (6)	−0.019 (3)	−0.046 (5)	−0.027 (5)
C21	0.092 (6)	0.055 (6)	0.114 (7)	−0.027 (6)	−0.065 (5)	−0.013 (6)
C25	0.091 (7)	0.053 (6)	0.111 (6)	−0.026 (5)	−0.043 (6)	−0.016 (5)
C26	0.077 (5)	0.056 (4)	0.127 (7)	−0.030 (4)	−0.047 (5)	−0.025 (5)
C24	0.094 (6)	0.060 (5)	0.127 (6)	−0.030 (5)	−0.061 (5)	−0.012 (5)
N4	0.057 (3)	0.045 (3)	0.076 (4)	−0.017 (3)	−0.026 (3)	−0.016 (3)

Geometric parameters (Å, º)

Cu1—O1	1.959 (4)	C17—H17B	0.9900
Cu1—O2^{i}	1.957 (4)	C17—C20	1.521 (12)
Cu1—O4	1.917 (4)	C15—H15A	0.9900
Cu1—O5^{i}	1.925 (5)	C15—H15B	0.9900
Cu1—N1	2.235 (5)	C15—C18	1.520 (12)
Cu1—N3	2.235 (5)	C16—H16A	0.9900
O1—C1	1.250 (7)	C16—H16B	0.9900
C2—C3	1.390 (10)	C16—C19	1.521 (12)
C2—C7	1.387 (10)	C20—H20A	0.9900
C2—C1	1.495 (8)	C20—H20B	0.9900
O2—C1	1.252 (7)	C20—N2	1.479 (8)
C3—H3	0.9500	C18—H18A	0.9900
C3—C4	1.375 (9)	C18—H18B	0.9900
O3—H3A	0.87 (2)	C18—N2	1.473 (8)
O3—C5	1.344 (8)	C19—H19A	0.9900
C4—H4	0.9500	C19—H19B	0.9900
C4—C5	1.413 (12)	C19—N2	1.479 (8)
O4—C8	1.294 (8)	N2—H2	1.330 (5)
C5—C6	1.390 (12)	N3—C22	1.477 (8)
O5—C8	1.310 (8)	N3—C23	1.474 (8)
C6—H6	0.9500	N3—C21	1.472 (8)
C6—C7	1.375 (10)	C22—H22A	0.9900
O6—C12	1.357 (11)	C22—H22B	0.9900
O6—H6A	0.86 (2)	C22—C25	1.523 (12)
C7—H7	0.9500	C23—H23A	0.9900
C9—C10	1.361 (11)	C23—H23B	0.9900
C9—C14	1.408 (11)	C23—C26	1.521 (12)
C9—C8	1.490 (10)	C21—H21A	0.9900
C10—H10	0.9500	C21—H21B	0.9900
C10—C11	1.415 (12)	C21—C24	1.519 (12)

C11—H11	0.9500	C25—H25A	0.9900
C11—C12	1.389 (15)	C25—H25B	0.9900
C12—C13	1.354 (14)	C25—N4	1.483 (8)
C13—H13	0.9500	C26—H26A	0.9900
C13—C14	1.358 (12)	C26—H26B	0.9900
C14—H14	0.9500	C26—N4	1.478 (8)
N1—C17	1.470 (8)	C24—H24A	0.9900
N1—C15	1.469 (8)	C24—H24B	0.9900
N1—C16	1.476 (8)	C24—N4	1.473 (8)
C17—H17A	0.9900	N4—H2	1.330 (5)

O1—Cu1—N1	95.47 (18)	C18—C15—H15B	109.6
O1—Cu1—N3	95.47 (18)	N1—C16—H16A	109.5
O2^{i}—Cu1—O1	168.33 (19)	N1—C16—H16B	109.5
O2^{i}—Cu1—N1	96.20 (19)	N1—C16—C19	110.5 (8)
O2^{i}—Cu1—N3	96.20 (19)	H16A—C16—H16B	108.1
O4—Cu1—O1	90.0 (2)	C19—C16—H16A	109.5
O4—Cu1—O2^{i}	89.0 (2)	C19—C16—H16B	109.5
O4—Cu1—O5^{i}	170.21 (18)	C17—C20—H20A	109.6
O4—Cu1—N1	95.04 (19)	C17—C20—H20B	109.6
O4—Cu1—N3	95.04 (19)	H20A—C20—H20B	108.1
O5^{i}—Cu1—O1	89.2 (2)	N2—C20—C17	110.1 (9)
O5^{i}—Cu1—O2^{i}	89.9 (2)	N2—C20—H20A	109.6
O5^{i}—Cu1—N1	94.8 (2)	N2—C20—H20B	109.6
O5^{i}—Cu1—N3	94.8 (2)	C15—C18—H18A	109.4
C1—O1—Cu1	124.4 (4)	C15—C18—H18B	109.4
C3—C2—C1	120.1 (6)	H18A—C18—H18B	108.0
C7—C2—C3	118.7 (6)	N2—C18—C15	111.0 (10)
C7—C2—C1	121.2 (6)	N2—C18—H18A	109.4
C1—O2—Cu1^{i}	122.5 (4)	N2—C18—H18B	109.4
C2—C3—H3	119.6	C16—C19—H19A	109.5
C4—C3—C2	120.8 (7)	C16—C19—H19B	109.5
C4—C3—H3	119.6	H19A—C19—H19B	108.1
C5—O3—H3A	107 (2)	N2—C19—C16	110.7 (8)
C3—C4—H4	119.7	N2—C19—H19A	109.5
C3—C4—C5	120.5 (8)	N2—C19—H19B	109.5
C5—C4—H4	119.7	C20—N2—H2	111.2 (6)
C8—O4—Cu1	124.4 (4)	C18—N2—C20	111.7 (10)
O3—C5—C4	122.4 (8)	C18—N2—C19	106.1 (9)
O3—C5—C6	119.6 (8)	C18—N2—H2	111.8 (6)
C6—C5—C4	118.0 (7)	C19—N2—C20	106.6 (10)
C8—O5—Cu1^{i}	122.5 (4)	C19—N2—H2	109.2 (5)
C5—C6—H6	119.6	C22—N3—Cu1	108.7 (7)
C7—C6—C5	120.8 (7)	C23—N3—Cu1	111.1 (6)
C7—C6—H6	119.6	C23—N3—C22	100.5 (11)
C12—O6—H6A	123 (10)	C21—N3—Cu1	109.6 (6)
C2—C7—H7	119.4	C21—N3—C22	125.0 (11)
C6—C7—C2	121.1 (7)	C21—N3—C23	100.8 (11)

C6—C7—H7	119.4	N3—C22—H22A	109.9
O1—C1—C2	117.7 (5)	N3—C22—H22B	109.9
O1—C1—O2	124.8 (5)	N3—C22—C25	109.0 (11)
O2—C1—C2	117.6 (6)	H22A—C22—H22B	108.3
C10—C9—C14	120.1 (8)	C25—C22—H22A	109.9
C10—C9—C8	121.6 (7)	C25—C22—H22B	109.9
C14—C9—C8	118.2 (7)	N3—C23—H23A	109.6
C9—C10—H10	120.6	N3—C23—H23B	109.6
C9—C10—C11	118.8 (8)	N3—C23—C26	110.1 (10)
C11—C10—H10	120.6	H23A—C23—H23B	108.1
C10—C11—H11	120.2	C26—C23—H23A	109.6
C12—C11—C10	119.6 (9)	C26—C23—H23B	109.6
C12—C11—H11	120.2	N3—C21—H21A	110.1
O6—C12—C11	116.1 (10)	N3—C21—H21B	110.1
C13—C12—O6	123.2 (10)	N3—C21—C24	108.2 (10)
C13—C12—C11	120.6 (9)	H21A—C21—H21B	108.4
C12—C13—H13	119.8	C24—C21—H21A	110.1
C12—C13—C14	120.5 (9)	C24—C21—H21B	110.1
C14—C13—H13	119.8	C22—C25—H25A	109.2
C9—C14—H14	119.8	C22—C25—H25B	109.2
C13—C14—C9	120.4 (9)	H25A—C25—H25B	107.9
C13—C14—H14	119.8	N4—C25—C22	112.0 (11)
O4—C8—O5	122.9 (6)	N4—C25—H25A	109.2
O4—C8—C9	119.3 (6)	N4—C25—H25B	109.2
O5—C8—C9	117.8 (6)	C23—C26—H26A	109.4
C17—N1—Cu1	110.3 (6)	C23—C26—H26B	109.4
C17—N1—C16	108.0 (9)	H26A—C26—H26B	108.0
C15—N1—Cu1	111.5 (6)	N4—C26—C23	111.2 (10)
C15—N1—C17	112.8 (10)	N4—C26—H26A	109.4
C15—N1—C16	104.0 (9)	N4—C26—H26B	109.4
C16—N1—Cu1	110.0 (5)	C21—C24—H24A	108.9
N1—C17—H17A	109.4	C21—C24—H24B	108.9
N1—C17—H17B	109.4	H24A—C24—H24B	107.7
N1—C17—C20	111.2 (9)	N4—C24—C21	113.3 (11)
H17A—C17—H17B	108.0	N4—C24—H24A	108.9
C20—C17—H17A	109.4	N4—C24—H24B	108.9
C20—C17—H17B	109.4	C25—N4—H2	111.5 (7)
N1—C15—H15A	109.6	C26—N4—C25	96.9 (11)
N1—C15—H15B	109.6	C26—N4—H2	112.5 (6)
N1—C15—C18	110.4 (10)	C24—N4—C25	124.6 (11)
H15A—C15—H15B	108.1	C24—N4—C26	97.4 (11)
C18—C15—H15A	109.6	C24—N4—H2	111.5 (7)
Cu1—O1—C1—C2	178.5 (4)	C12—C13—C14—C9	−1.5 (11)
Cu1—O1—C1—O2	−2.2 (10)	C14—C9—C10—C11	−1.1 (11)
Cu1^{i}—O2—C1—O1	2.2 (10)	C14—C9—C8—O4	−169.9 (6)
Cu1^{i}—O2—C1—C2	−178.6 (4)	C14—C9—C8—O5	9.2 (8)
Cu1—O4—C8—O5	2.3 (8)	C8—C9—C10—C11	−177.7 (6)

Cu1—O4—C8—C9	−178.7 (4)	C8—C9—C14—C13	178.1 (6)
Cu1^{i}—O5—C8—O4	−2.6 (8)	N1—C17—C20—N2	2.0 (14)
Cu1^{i}—O5—C8—C9	178.3 (4)	N1—C15—C18—N2	4.1 (15)
Cu1—N1—C17—C20	177.9 (9)	N1—C16—C19—N2	−0.9 (14)
Cu1—N1—C15—C18	177.8 (9)	C17—N1—C15—C18	53.0 (14)
Cu1—N1—C16—C19	−178.5 (8)	C17—N1—C16—C19	−58.1 (13)
Cu1—N3—C22—C25	−177.6 (9)	C17—C20—N2—C18	54.7 (13)
Cu1—N3—C23—C26	179.5 (9)	C17—C20—N2—C19	−60.7 (12)
Cu1—N3—C21—C24	179.3 (9)	C15—N1—C17—C20	−56.7 (14)
C2—C3—C4—C5	1.9 (13)	C15—N1—C16—C19	61.9 (12)
C3—C2—C7—C6	−1.0 (12)	C15—C18—N2—C20	−58.5 (14)
C3—C2—C1—O1	2.6 (10)	C15—C18—N2—C19	57.3 (13)
C3—C2—C1—O2	−176.7 (7)	C16—N1—C17—C20	57.6 (13)
C3—C4—C5—O3	177.9 (7)	C16—N1—C15—C18	−63.7 (12)
C3—C4—C5—C6	−0.1 (13)	C16—C19—N2—C20	60.2 (12)
O3—C5—C6—C7	179.7 (7)	C16—C19—N2—C18	−58.9 (12)
C4—C5—C6—C7	−2.2 (13)	N3—C22—C25—N4	−5.2 (15)
C5—C6—C7—C2	2.8 (13)	N3—C23—C26—N4	−0.5 (16)
O6—C12—C13—C14	179.0 (7)	N3—C21—C24—N4	3.1 (16)
C7—C2—C3—C4	−1.4 (12)	C22—N3—C23—C26	64.7 (13)
C7—C2—C1—O1	−178.4 (6)	C22—N3—C21—C24	−49.0 (17)
C7—C2—C1—O2	2.4 (10)	C22—C25—N4—C26	67.1 (13)
C1—C2—C3—C4	177.7 (7)	C22—C25—N4—C24	−36.8 (18)
C1—C2—C7—C6	179.9 (7)	C23—N3—C22—C25	−60.9 (13)
C9—C10—C11—C12	0.9 (11)	C23—N3—C21—C24	62.1 (13)
C10—C9—C14—C13	1.4 (11)	C23—C26—N4—C25	−63.4 (13)
C10—C9—C8—O4	6.8 (9)	C23—C26—N4—C24	62.9 (14)
C10—C9—C8—O5	−174.1 (6)	C21—N3—C22—C25	50.4 (16)
C10—C11—C12—O6	−178.8 (7)	C21—N3—C23—C26	−64.4 (13)
C10—C11—C12—C13	−1.0 (12)	C21—C24—N4—C25	38.2 (19)
C11—C12—C13—C14	1.3 (12)	C21—C24—N4—C26	−65.5 (14)

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.

Bis(tetraethylammonium) bis(*μ*-4-hydroxybenzoato)bis(*μ*-4-oxidobenzoato)bis[(dioxane)copper(II)] aqua(dioxane)tetrakis(*μ*-4-hydroxybenzoato)dicopper(II) methanol monosolvate (compound_5)

Crystal data

$(C_8H_{20}N)[Cu_2(C_7H_4O_3)_2(C_7H_5O_3)_2(C_4H_8O_2)_2]$
$[Cu_2(C_7H_5O_3)_4(C_4H_8O_2)(H_2O)]\cdot CH_4O$
M_r = 1923.89
Orthorhombic, *Pnma*
a = 12.833 (3) Å
b = 54.548 (11) Å
c = 12.119 (2) Å
V = 8483 (3) Å^3
Z = 4
F(000) = 4016
D_x = 1.506 Mg m^{-3}
Synchrotron radiation, *λ* = 0.7108 Å
Cell parameters from 2355 reflections
μ = 1.08 mm^{-1}
T = 100 K
Irregular, clear blue
0.13 × 0.09 × 0.06 mm

Data collection

MX2 beamline, Australian Synchrotron diffractometer
Radiation source: MX2 Beamline Australian Synchrotron
Silicon Double Crystal monochromator
Detector resolution: 13.3 pixels mm^{-1}
ω Scan scans
Absorption correction: multi-scan (XDS; Kabsch, 2010)
T_{min} = 0.321, T_{max} = 0.432
151309 measured reflections
13753 independent reflections
11493 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 32.4°, θ_{min} = 0.8°
h = −19→19
k = −75→75
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.148
S = 1.02
13753 reflections
585 parameters
67 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0883P)^2 + 6.6788P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 1.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.84 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. The methanol molecule is disordered across a mirror plane and modelled in Part -1.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	1.09438 (2)	0.50228 (2)	0.46389 (2)	0.02254 (7)	
Cu3	0.77549 (3)	0.750000	0.39070 (3)	0.02583 (8)	
Cu2	0.97413 (3)	0.750000	0.42875 (3)	0.02737 (9)	
O11	0.93206 (12)	0.63271 (2)	0.00021 (12)	0.0286 (3)	
O6	0.79458 (13)	0.59894 (3)	0.04796 (13)	0.0305 (3)	
O5	0.87919 (12)	0.51933 (3)	0.40980 (14)	0.0344 (3)	
O1	1.10134 (12)	0.53053 (3)	0.56100 (13)	0.0310 (3)	
O4	1.04170 (12)	0.52395 (3)	0.34824 (13)	0.0327 (3)	
O7	1.25994 (13)	0.50399 (3)	0.41904 (15)	0.0351 (3)	
O2	0.93938 (13)	0.52620 (3)	0.62668 (14)	0.0351 (3)	
O17	0.6112 (2)	0.750000	0.3665 (2)	0.0372 (5)	
H17C	0.567 (2)	0.750000	0.424 (3)	0.056*	
H17	0.583 (3)	0.7564 (10)	0.310 (3)	0.056*	0.5
O3	1.09462 (14)	0.61876 (3)	0.89328 (14)	0.0369 (4)	
H3	1.047567	0.622646	0.936981	0.077 (13)*	
O9	0.98162 (14)	0.72462 (3)	0.31540 (16)	0.0443 (4)	
O10	0.81086 (14)	0.72439 (3)	0.28582 (16)	0.0451 (4)	
O13	0.77289 (15)	0.72506 (4)	0.50558 (17)	0.0460 (4)	
O14	0.79843 (16)	0.63828 (3)	0.84125 (15)	0.0458 (4)	

O18	0.5112 (6)	0.73992 (18)	0.5562 (5)	0.139 (4)	0.5
H18	0.451763	0.744139	0.572141	0.208*	0.5
H14A	0.840123	0.637049	0.896711	0.208*	
O12	0.94340 (16)	0.72509 (4)	0.53854 (17)	0.0498 (5)	
O15	1.1407 (2)	0.750000	0.4589 (3)	0.0510 (7)	
N1	0.94177 (16)	0.62350 (4)	0.45490 (16)	0.0340 (4)	
C12	0.83421 (17)	0.58163 (3)	0.11586 (15)	0.0267 (4)	
C7	0.96608 (16)	0.56600 (3)	0.76684 (16)	0.0274 (4)	
H7	0.902792	0.557028	0.771631	0.033*	
O8	1.41723 (18)	0.47818 (4)	0.5330 (2)	0.0576 (5)	
C2	1.04197 (16)	0.55867 (3)	0.69232 (16)	0.0261 (3)	
C19	0.91121 (17)	0.69439 (3)	0.19854 (16)	0.0283 (4)	
C13	0.94089 (17)	0.57883 (3)	0.13287 (16)	0.0272 (4)	
H13	0.988965	0.589086	0.095133	0.033*	
C8	0.94606 (16)	0.52843 (3)	0.34574 (16)	0.0258 (3)	
C1	1.02630 (16)	0.53685 (3)	0.62135 (15)	0.0253 (3)	
C11	0.76460 (17)	0.56603 (4)	0.16858 (18)	0.0298 (4)	
H11	0.692123	0.567179	0.153469	0.036*	
C25	0.85244 (19)	0.71771 (4)	0.55474 (16)	0.0317 (4)	
C22	0.92602 (17)	0.65267 (3)	0.06385 (16)	0.0270 (4)	
C6	0.98102 (17)	0.58615 (4)	0.83431 (16)	0.0283 (4)	
H6A	0.928236	0.590939	0.884863	0.034*	
C14	0.97644 (16)	0.56107 (3)	0.20488 (16)	0.0270 (4)	
H14	1.049277	0.558911	0.215007	0.032*	
C23	1.01435 (17)	0.66676 (4)	0.08873 (18)	0.0313 (4)	
H23	1.080046	0.662257	0.058802	0.038*	
C26	0.83724 (19)	0.69794 (4)	0.63736 (17)	0.0318 (4)	
C64	0.90060 (17)	0.71611 (4)	0.27185 (16)	0.0288 (4)	
C9	0.90715 (16)	0.54634 (3)	0.26264 (16)	0.0257 (3)	
C5	1.07344 (17)	0.59938 (4)	0.82799 (17)	0.0297 (4)	
C4	1.14971 (18)	0.59259 (4)	0.75125 (18)	0.0316 (4)	
H4	1.212193	0.601828	0.745030	0.038*	
C27	0.7452 (2)	0.68484 (4)	0.63751 (19)	0.0365 (5)	
H27	0.689710	0.689614	0.590260	0.044*	
C24	1.00705 (17)	0.68709 (4)	0.15624 (18)	0.0318 (4)	
H24	1.067993	0.696164	0.173907	0.038*	
C3	1.13368 (17)	0.57242 (4)	0.68471 (17)	0.0292 (4)	
H3A	1.185705	0.567794	0.632966	0.035*	
C10	0.80083 (16)	0.54885 (3)	0.24297 (17)	0.0285 (4)	
H10	0.752775	0.538650	0.281032	0.034*	
C31	0.9161 (2)	0.69161 (4)	0.71040 (18)	0.0354 (4)	
H31	0.979126	0.700732	0.711240	0.043*	
C20	0.82288 (18)	0.68093 (4)	0.17132 (19)	0.0336 (4)	
H20	0.756699	0.686007	0.198006	0.040*	
C39	0.8940 (2)	0.60932 (4)	0.54997 (18)	0.0358 (5)	
H39A	0.950063	0.604951	0.602465	0.043*	
H39B	0.844682	0.620229	0.589215	0.043*	
C21	0.83027 (18)	0.66027 (4)	0.10592 (18)	0.0332 (4)	

H21	0.769260	0.651113	0.089396	0.040*
C42	1.08702 (19)	0.62009 (5)	0.3124 (2)	0.0374 (5)
H42A	1.039807	0.621569	0.249216	0.056*
H42B	1.146988	0.609944	0.291858	0.056*
H42C	1.111212	0.636411	0.334408	0.056*
C29	0.8131 (2)	0.65835 (4)	0.77841 (18)	0.0363 (5)
C30	0.9032 (2)	0.67202 (4)	0.78223 (19)	0.0356 (4)
H30	0.956350	0.668071	0.833815	0.043*
C15	1.30881 (19)	0.48317 (4)	0.3692 (2)	0.0382 (4)
H15A	1.368711	0.488498	0.323664	0.046*
H15B	1.258622	0.474517	0.320926	0.046*
C37	0.8636 (2)	0.62802 (5)	0.3634 (2)	0.0405 (5)
H37A	0.897573	0.637781	0.304673	0.049*
H37B	0.843334	0.612056	0.330932	0.049*
C28	0.7336 (2)	0.66485 (4)	0.7061 (2)	0.0397 (5)
H28	0.671123	0.655505	0.703982	0.048*
C40	0.8368 (2)	0.58612 (5)	0.5178 (2)	0.0445 (5)
H40A	0.778075	0.590266	0.469606	0.067*
H40B	0.810747	0.577962	0.584382	0.067*
H40C	0.884540	0.575124	0.478700	0.067*
C41	1.0304 (2)	0.60833 (5)	0.4071 (2)	0.0428 (5)
H41A	1.081202	0.604906	0.466636	0.051*
H41B	1.001899	0.592412	0.382052	0.051*
C38	0.7663 (2)	0.64124 (5)	0.4007 (2)	0.0425 (5)
H38A	0.718827	0.643180	0.337958	0.064*
H38B	0.785164	0.657431	0.429548	0.064*
H38C	0.731911	0.631705	0.458698	0.064*
O16	1.2961 (4)	0.750000	0.6153 (4)	0.1116 (19)
C17	1.3722 (2)	0.49978 (5)	0.5839 (3)	0.0483 (6)
H17A	1.425049	0.508191	0.629804	0.058*
H17B	1.313166	0.495025	0.631961	0.058*
C35	0.9795 (2)	0.64795 (5)	0.4959 (3)	0.0449 (6)
H35A	0.998623	0.658168	0.431508	0.054*
H35B	0.921343	0.656247	0.534448	0.054*
C36	1.0698 (2)	0.64683 (6)	0.5715 (3)	0.0537 (7)
H36A	1.128135	0.638651	0.534472	0.081*
H36B	1.050704	0.637598	0.637875	0.081*
H36C	1.090526	0.663494	0.592338	0.081*
C18	1.3345 (2)	0.51679 (5)	0.4933 (2)	0.0424 (5)
H18A	1.299981	0.531228	0.526678	0.051*
H18B	1.394838	0.522659	0.449831	0.051*
C16	1.3456 (2)	0.46624 (5)	0.4601 (3)	0.0496 (6)
H16A	1.284709	0.460432	0.502876	0.060*
H16B	1.379874	0.451750	0.426902	0.060*
C32	1.1999 (3)	0.77128 (6)	0.4784 (3)	0.0574 (8)
H32A	1.157007	0.786006	0.463735	0.069*
H32B	1.260763	0.771627	0.428212	0.069*
C33	1.2354 (4)	0.77148 (9)	0.5931 (4)	0.0939 (15)

H33A	1.278058	0.786305	0.606652	0.113*	
H33B	1.174512	0.771984	0.643119	0.113*	
H6	0.8425 (19)	0.6083 (5)	0.035 (3)	0.045 (9)*	
C34	0.5581 (9)	0.7512 (7)	0.6500 (7)	0.139 (4)	0.5
H34A	0.517242	0.747211	0.715856	0.208*	0.5
H34B	0.560602	0.769051	0.640566	0.208*	0.5
H34C	0.629092	0.744851	0.658456	0.208*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02841 (12)	0.01829 (11)	0.02092 (12)	0.00083 (8)	0.00086 (8)	0.00034 (7)
Cu3	0.03776 (18)	0.02005 (15)	0.01968 (15)	0.000	−0.00168 (12)	0.000
Cu2	0.03862 (19)	0.02342 (16)	0.02007 (16)	0.000	−0.00258 (13)	0.000
O11	0.0424 (8)	0.0189 (6)	0.0245 (6)	0.0004 (5)	0.0009 (6)	−0.0010 (5)
O6	0.0390 (8)	0.0234 (6)	0.0292 (7)	0.0006 (6)	−0.0010 (6)	0.0070 (5)
O5	0.0333 (7)	0.0335 (8)	0.0363 (8)	0.0025 (6)	0.0024 (6)	0.0154 (6)
O1	0.0359 (7)	0.0271 (7)	0.0300 (7)	−0.0028 (6)	0.0039 (6)	−0.0079 (6)
O4	0.0343 (7)	0.0360 (8)	0.0278 (7)	0.0021 (6)	0.0004 (6)	0.0096 (6)
O7	0.0341 (7)	0.0300 (7)	0.0413 (9)	0.0029 (6)	0.0075 (6)	0.0015 (6)
O2	0.0367 (8)	0.0287 (7)	0.0400 (8)	−0.0035 (6)	0.0072 (7)	−0.0123 (6)
O17	0.0408 (12)	0.0398 (12)	0.0309 (11)	0.000	−0.0041 (9)	0.000
O3	0.0459 (9)	0.0305 (8)	0.0344 (8)	−0.0079 (6)	0.0056 (7)	−0.0129 (6)
O9	0.0422 (9)	0.0406 (9)	0.0500 (10)	0.0037 (7)	−0.0060 (8)	−0.0229 (8)
O10	0.0415 (9)	0.0454 (10)	0.0485 (10)	0.0043 (7)	−0.0033 (8)	−0.0257 (8)
O13	0.0462 (10)	0.0459 (10)	0.0460 (10)	−0.0038 (8)	−0.0065 (8)	0.0242 (8)
O14	0.0663 (12)	0.0305 (8)	0.0405 (9)	−0.0143 (8)	−0.0184 (8)	0.0139 (7)
O18	0.120 (4)	0.231 (10)	0.066 (3)	−0.083 (6)	−0.004 (3)	−0.009 (4)
O12	0.0462 (10)	0.0558 (11)	0.0473 (11)	−0.0053 (9)	−0.0065 (8)	0.0273 (9)
O15	0.0454 (14)	0.0340 (13)	0.074 (2)	0.000	−0.0177 (13)	0.000
N1	0.0405 (10)	0.0309 (9)	0.0306 (9)	0.0044 (7)	0.0032 (7)	0.0021 (7)
C12	0.0376 (10)	0.0210 (8)	0.0214 (8)	0.0005 (7)	−0.0006 (7)	0.0010 (6)
C7	0.0349 (9)	0.0221 (8)	0.0252 (9)	−0.0010 (7)	0.0008 (7)	−0.0011 (6)
O8	0.0559 (12)	0.0470 (11)	0.0698 (15)	0.0062 (9)	−0.0079 (10)	0.0070 (10)
C2	0.0357 (9)	0.0210 (8)	0.0217 (8)	0.0006 (7)	0.0000 (7)	−0.0004 (6)
C19	0.0393 (10)	0.0220 (8)	0.0238 (8)	0.0009 (7)	−0.0017 (7)	−0.0012 (6)
C13	0.0364 (10)	0.0240 (8)	0.0213 (8)	−0.0036 (7)	−0.0008 (7)	0.0028 (6)
C8	0.0367 (9)	0.0193 (7)	0.0216 (8)	0.0003 (7)	−0.0002 (7)	0.0002 (6)
C1	0.0363 (9)	0.0195 (7)	0.0201 (8)	0.0023 (7)	−0.0009 (7)	0.0007 (6)
C11	0.0330 (9)	0.0260 (9)	0.0304 (10)	0.0020 (7)	0.0006 (8)	0.0049 (7)
C25	0.0491 (12)	0.0243 (9)	0.0218 (8)	0.0014 (8)	−0.0014 (8)	0.0012 (6)
C22	0.0389 (10)	0.0194 (8)	0.0226 (8)	0.0006 (7)	−0.0014 (7)	0.0012 (6)
C6	0.0372 (10)	0.0239 (8)	0.0239 (8)	−0.0002 (7)	0.0023 (7)	−0.0016 (6)
C14	0.0332 (9)	0.0255 (8)	0.0222 (8)	−0.0016 (7)	−0.0027 (7)	0.0008 (6)
C23	0.0355 (10)	0.0263 (9)	0.0322 (10)	0.0015 (7)	−0.0008 (8)	−0.0025 (7)
C26	0.0495 (12)	0.0236 (8)	0.0223 (8)	0.0011 (8)	−0.0027 (8)	0.0024 (7)
C64	0.0419 (10)	0.0221 (8)	0.0225 (9)	0.0006 (7)	0.0001 (7)	0.0004 (6)
C9	0.0359 (9)	0.0195 (7)	0.0215 (8)	0.0012 (7)	−0.0008 (7)	0.0003 (6)

C5	0.0406 (10)	0.0229 (8)	0.0256 (9)	−0.0021 (7)	−0.0005 (8)	−0.0034 (7)
C4	0.0376 (10)	0.0274 (9)	0.0299 (9)	−0.0042 (8)	0.0036 (8)	−0.0043 (7)
C27	0.0506 (13)	0.0309 (10)	0.0282 (10)	−0.0039 (9)	−0.0083 (9)	0.0057 (8)
C24	0.0359 (10)	0.0277 (9)	0.0316 (10)	−0.0006 (7)	−0.0031 (8)	−0.0041 (7)
C3	0.0352 (9)	0.0264 (9)	0.0258 (9)	−0.0008 (7)	0.0030 (7)	−0.0017 (7)
C10	0.0339 (9)	0.0230 (8)	0.0286 (9)	−0.0005 (7)	0.0023 (7)	0.0050 (7)
C31	0.0470 (12)	0.0314 (10)	0.0279 (10)	−0.0020 (8)	−0.0029 (9)	0.0057 (8)
C20	0.0384 (10)	0.0276 (9)	0.0348 (10)	−0.0006 (8)	0.0033 (8)	−0.0064 (8)
C39	0.0469 (12)	0.0340 (11)	0.0265 (10)	−0.0021 (9)	0.0022 (9)	−0.0010 (8)
C21	0.0371 (10)	0.0275 (9)	0.0351 (10)	−0.0045 (8)	0.0024 (8)	−0.0065 (8)
C42	0.0397 (11)	0.0383 (11)	0.0344 (11)	0.0037 (9)	0.0036 (9)	0.0004 (9)
C29	0.0548 (13)	0.0254 (9)	0.0287 (10)	−0.0035 (9)	−0.0072 (9)	0.0050 (7)
C30	0.0477 (12)	0.0301 (10)	0.0289 (10)	−0.0003 (8)	−0.0060 (9)	0.0056 (8)
C15	0.0371 (11)	0.0376 (10)	0.0401 (11)	0.0061 (8)	0.0095 (9)	−0.0016 (8)
C37	0.0478 (13)	0.0444 (13)	0.0292 (10)	0.0116 (10)	−0.0002 (9)	0.0002 (9)
C28	0.0533 (13)	0.0322 (10)	0.0337 (11)	−0.0098 (9)	−0.0105 (10)	0.0076 (8)
C40	0.0530 (14)	0.0351 (11)	0.0453 (13)	−0.0051 (10)	0.0007 (11)	−0.0007 (10)
C41	0.0488 (13)	0.0379 (12)	0.0418 (12)	0.0134 (10)	0.0086 (10)	0.0067 (10)
C38	0.0420 (12)	0.0501 (14)	0.0354 (11)	0.0096 (10)	−0.0012 (10)	−0.0005 (10)
O16	0.112 (4)	0.126 (4)	0.097 (4)	0.000	−0.067 (3)	0.000
C17	0.0447 (13)	0.0476 (14)	0.0527 (16)	0.0032 (11)	0.0027 (12)	0.0163 (12)
C35	0.0463 (13)	0.0326 (11)	0.0559 (16)	0.0007 (10)	−0.0008 (12)	−0.0038 (10)
C36	0.0523 (15)	0.0597 (18)	0.0490 (16)	−0.0058 (13)	−0.0063 (13)	−0.0062 (13)
C18	0.0481 (13)	0.0353 (11)	0.0439 (12)	−0.0003 (9)	0.0038 (9)	0.0016 (8)
C16	0.0515 (15)	0.0384 (13)	0.0590 (17)	0.0033 (11)	0.0050 (13)	−0.0024 (11)
C32	0.0497 (15)	0.0453 (15)	0.077 (2)	−0.0108 (12)	−0.0080 (15)	0.0045 (14)
C33	0.102 (3)	0.092 (3)	0.087 (3)	−0.002 (3)	−0.031 (3)	−0.040 (3)
C34	0.119 (4)	0.230 (10)	0.067 (3)	−0.085 (6)	−0.004 (3)	−0.009 (5)

Geometric parameters (Å, º)

Cu1—O5^{i}	1.9618 (15)	C23—H23	0.9500
Cu1—O1	1.9408 (15)	C23—C24	1.382 (3)
Cu1—O4	1.9542 (15)	C26—C27	1.381 (3)
Cu1—O7	2.1950 (17)	C26—C31	1.388 (3)
Cu1—O2^{i}	1.9508 (15)	C9—C10	1.392 (3)
Cu3—Cu3^{ii}	0.0000 (10)	C5—C4	1.400 (3)
Cu3—O17	2.128 (3)	C4—H4	0.9500
Cu3—O10	1.9425 (16)	C4—C3	1.380 (3)
Cu3—O10^{ii}	1.9426 (16)	C27—H27	0.9500
Cu3—O13	1.9469 (17)	C27—C28	1.379 (3)
Cu3—O13^{ii}	1.9469 (17)	C24—H24	0.9500
Cu2—O9^{ii}	1.9527 (16)	C3—H3A	0.9500
Cu2—O9	1.9526 (16)	C10—H10	0.9500
Cu2—O12	1.9422 (18)	C31—H31	0.9500
Cu2—O12^{ii}	1.9422 (18)	C31—C30	1.388 (3)
Cu2—O15	2.168 (3)	C20—H20	0.9500
O11—C22	1.336 (2)	C20—C21	1.381 (3)

O6—C12	1.352 (2)	C39—H39A	0.9900
O6—H6	0.815 (18)	C39—H39B	0.9900
O5—C8	1.259 (2)	C39—C40	1.514 (3)
O1—C1	1.257 (2)	C21—H21	0.9500
O4—C8	1.252 (3)	C42—H42A	0.9800
O7—C15	1.431 (3)	C42—H42B	0.9800
O7—C18	1.487 (3)	C42—H42C	0.9800
O2—C1	1.259 (3)	C42—C41	1.502 (3)
O17—H17C	0.899 (19)	C29—C30	1.376 (3)
O17—H17	0.847 (19)	C29—C28	1.392 (3)
O3—H3	0.8307	C30—H30	0.9500
O3—C5	1.348 (2)	C15—H15A	0.9900
O9—C64	1.255 (3)	C15—H15B	0.9900
O10—C64	1.248 (3)	C15—C16	1.513 (4)
O13—C25	1.248 (3)	C37—H37A	0.9900
O14—H14A	0.8617	C37—H37B	0.9900
O14—C29	1.347 (3)	C37—C38	1.511 (3)
O18—H18	0.8199	C28—H28	0.9500
O18—C34	1.427 (16)	C40—H40A	0.9800
O12—C25	1.250 (3)	C40—H40B	0.9800
O15—C32^{ii}	1.408 (3)	C40—H40C	0.9800
O15—C32	1.408 (3)	C41—H41A	0.9900
N1—C39	1.517 (3)	C41—H41B	0.9900
N1—C37	1.516 (3)	C38—H38A	0.9800
N1—C41	1.521 (3)	C38—H38B	0.9800
N1—C35	1.504 (3)	C38—H38C	0.9800
C12—C13	1.393 (3)	O16—C33^{ii}	1.432 (6)
C12—C11	1.389 (3)	O16—C33	1.432 (6)
C7—H7	0.9500	C17—H17A	0.9900
C7—C2	1.387 (3)	C17—H17B	0.9900
C7—C6	1.383 (3)	C17—C18	1.517 (4)
O8—C17	1.450 (4)	C35—H35A	0.9900
O8—C16	1.431 (4)	C35—H35B	0.9900
C2—C1	1.482 (3)	C35—C36	1.478 (4)
C2—C3	1.398 (3)	C36—H36A	0.9800
C19—C64	1.487 (3)	C36—H36B	0.9800
C19—C24	1.391 (3)	C36—H36C	0.9800
C19—C20	1.390 (3)	C18—H18A	0.9900
C13—H13	0.9500	C18—H18B	0.9900
C13—C14	1.382 (3)	C16—H16A	0.9900
C8—C9	1.489 (3)	C16—H16B	0.9900
C11—H11	0.9500	C32—H32A	0.9900
C11—C10	1.381 (3)	C32—H32B	0.9900
C25—C26	1.484 (3)	C32—C33	1.462 (6)
C22—C23	1.402 (3)	C33—H33A	0.9900
C22—C21	1.393 (3)	C33—H33B	0.9900
C6—H6A	0.9500	C34—H34A	0.9795
C6—C5	1.390 (3)	C34—H34B	0.9804

C14—H14	0.9500	C34—H34C	0.9799
C14—C9	1.388 (3)		
O5^{i}—Cu1—O7	92.94 (7)	C3—C4—H4	120.2
O1—Cu1—O5^{i}	89.78 (7)	C26—C27—H27	119.9
O1—Cu1—O4	88.31 (7)	C28—C27—C26	120.2 (2)
O1—Cu1—O7	94.12 (6)	C28—C27—H27	119.9
O1—Cu1—O2^{i}	169.60 (7)	C19—C24—H24	119.7
O4—Cu1—O5^{i}	169.44 (7)	C23—C24—C19	120.5 (2)
O4—Cu1—O7	97.56 (7)	C23—C24—H24	119.7
O2^{i}—Cu1—O5^{i}	89.93 (8)	C2—C3—H3A	119.5
O2^{i}—Cu1—O4	90.08 (7)	C4—C3—C2	120.96 (19)
O2^{i}—Cu1—O7	96.28 (7)	C4—C3—H3A	119.5
Cu3^{ii}—Cu3—O17	0 (10)	C11—C10—C9	120.57 (18)
Cu3^{ii}—Cu3—O10	0 (10)	C11—C10—H10	119.7
Cu3^{ii}—Cu3—O10^{ii}	0 (10)	C9—C10—H10	119.7
Cu3^{ii}—Cu3—O13	0 (10)	C26—C31—H31	119.8
Cu3^{ii}—Cu3—O13^{ii}	0 (10)	C26—C31—C30	120.3 (2)
O10—Cu3—O17	98.13 (7)	C30—C31—H31	119.8
O10^{ii}—Cu3—O17	98.12 (7)	C19—C20—H20	119.6
O10—Cu3—O10^{ii}	91.98 (13)	C21—C20—C19	120.7 (2)
O10^{ii}—Cu3—O13	167.03 (8)	C21—C20—H20	119.6
O10—Cu3—O13	88.24 (9)	N1—C39—H39A	108.5
O10—Cu3—O13^{ii}	167.04 (8)	N1—C39—H39B	108.5
O10^{ii}—Cu3—O13^{ii}	88.24 (9)	H39A—C39—H39B	107.5
O13—Cu3—O17	94.67 (8)	C40—C39—N1	115.3 (2)
O13^{ii}—Cu3—O17	94.67 (8)	C40—C39—H39A	108.5
O13^{ii}—Cu3—O13	88.66 (13)	C40—C39—H39B	108.5
O9—Cu2—O9^{ii}	90.31 (13)	C22—C21—H21	119.6
O9—Cu2—O15	94.01 (8)	C20—C21—C22	120.9 (2)
O9^{ii}—Cu2—O15	94.01 (8)	C20—C21—H21	119.6
O12—Cu2—O9	89.77 (10)	H42A—C42—H42B	109.5
O12—Cu2—O9^{ii}	171.10 (8)	H42A—C42—H42C	109.5
O12^{ii}—Cu2—O9^{ii}	89.77 (10)	H42B—C42—H42C	109.5
O12^{ii}—Cu2—O9	171.10 (8)	C41—C42—H42A	109.5
O12—Cu2—O12^{ii}	88.79 (15)	C41—C42—H42B	109.5
O12^{ii}—Cu2—O15	94.86 (9)	C41—C42—H42C	109.5
O12—Cu2—O15	94.86 (9)	O14—C29—C30	122.6 (2)
C12—O6—H6	106 (2)	O14—C29—C28	117.4 (2)
C8—O5—Cu1^{i}	126.88 (14)	C30—C29—C28	120.0 (2)
C1—O1—Cu1	122.35 (13)	C31—C30—H30	120.1
C8—O4—Cu1	118.33 (13)	C29—C30—C31	119.7 (2)
C15—O7—Cu1	119.67 (14)	C29—C30—H30	120.1
C15—O7—C18	110.24 (19)	O7—C15—H15A	110.0
C18—O7—Cu1	119.51 (14)	O7—C15—H15B	110.0
C1—O2—Cu1^{i}	122.35 (13)	O7—C15—C16	108.3 (2)
Cu3^{ii}—O17—Cu3	0.000 (17)	H15A—C15—H15B	108.4
Cu3^{ii}—O17—H17C	121 (3)	C16—C15—H15A	110.0

Cu3—O17—H17C	121 (3)	C16—C15—H15B	110.0
Cu3^{ii}—O17—H17	123 (3)	N1—C37—H37A	108.8
Cu3—O17—H17	123 (3)	N1—C37—H37B	108.8
H17C—O17—H17	110 (5)	H37A—C37—H37B	107.7
C5—O3—H3	115.3	C38—C37—N1	113.9 (2)
C64—O9—Cu2	121.15 (15)	C38—C37—H37A	108.8
Cu3^{ii}—O10—Cu3	0.00 (2)	C38—C37—H37B	108.8
C64—O10—Cu3^{ii}	124.35 (15)	C27—C28—C29	120.1 (2)
C64—O10—Cu3	124.35 (15)	C27—C28—H28	119.9
Cu3^{ii}—O13—Cu3	0.000 (13)	C29—C28—H28	119.9
C25—O13—Cu3^{ii}	123.49 (16)	C39—C40—H40A	109.5
C25—O13—Cu3	123.49 (16)	C39—C40—H40B	109.5
C29—O14—H14A	114.7	C39—C40—H40C	109.5
C34—O18—H18	94.8	H40A—C40—H40B	109.5
C25—O12—Cu2	121.49 (16)	H40A—C40—H40C	109.5
C32—O15—Cu2	124.11 (17)	H40B—C40—H40C	109.5
C32^{ii}—O15—Cu2	124.11 (17)	N1—C41—H41A	108.6
C32^{ii}—O15—C32	111.1 (3)	N1—C41—H41B	108.6
C39—N1—C41	108.29 (18)	C42—C41—N1	114.85 (19)
C37—N1—C39	111.8 (2)	C42—C41—H41A	108.6
C37—N1—C41	107.75 (19)	C42—C41—H41B	108.6
C35—N1—C39	109.36 (19)	H41A—C41—H41B	107.5
C35—N1—C37	108.14 (19)	C37—C38—H38A	109.5
C35—N1—C41	111.6 (2)	C37—C38—H38B	109.5
O6—C12—C13	122.43 (18)	C37—C38—H38C	109.5
O6—C12—C11	117.77 (19)	H38A—C38—H38B	109.5
C11—C12—C13	119.79 (18)	H38A—C38—H38C	109.5
C2—C7—H7	119.5	H38B—C38—H38C	109.5
C6—C7—H7	119.5	C33^{ii}—O16—C33	109.8 (5)
C6—C7—C2	121.10 (19)	O8—C17—H17A	110.0
C16—O8—C17	112.1 (2)	O8—C17—H17B	110.0
C7—C2—C1	120.92 (18)	O8—C17—C18	108.4 (2)
C7—C2—C3	118.65 (17)	H17A—C17—H17B	108.4
C3—C2—C1	120.43 (18)	C18—C17—H17A	110.0
C24—C19—C64	121.95 (19)	C18—C17—H17B	110.0
C20—C19—C64	119.20 (19)	N1—C35—H35A	108.5
C20—C19—C24	118.85 (18)	N1—C35—H35B	108.5
C12—C13—H13	120.2	H35A—C35—H35B	107.5
C14—C13—C12	119.66 (18)	C36—C35—N1	114.9 (2)
C14—C13—H13	120.2	C36—C35—H35A	108.5
O5—C8—C9	116.55 (18)	C36—C35—H35B	108.5
O4—C8—O5	125.20 (18)	C35—C36—H36A	109.5
O4—C8—C9	118.23 (18)	C35—C36—H36B	109.5
O1—C1—O2	125.57 (18)	C35—C36—H36C	109.5
O1—C1—C2	116.98 (18)	H36A—C36—H36B	109.5
O2—C1—C2	117.44 (18)	H36A—C36—H36C	109.5
C12—C11—H11	120.0	H36B—C36—H36C	109.5
C10—C11—C12	119.95 (19)	O7—C18—C17	110.9 (2)

C10—C11—H11	120.0	O7—C18—H18A	109.5
O13—C25—O12	125.8 (2)	O7—C18—H18B	109.5
O13—C25—C26	116.6 (2)	C17—C18—H18A	109.5
O12—C25—C26	117.6 (2)	C17—C18—H18B	109.5
O11—C22—C23	121.57 (19)	H18A—C18—H18B	108.1
O11—C22—C21	120.30 (19)	O8—C16—C15	111.9 (2)
C21—C22—C23	118.12 (18)	O8—C16—H16A	109.2
C7—C6—H6A	120.1	O8—C16—H16B	109.2
C7—C6—C5	119.88 (19)	C15—C16—H16A	109.2
C5—C6—H6A	120.1	C15—C16—H16B	109.2
C13—C14—H14	119.6	H16A—C16—H16B	107.9
C13—C14—C9	120.87 (19)	O15—C32—H32A	109.8
C9—C14—H14	119.6	O15—C32—H32B	109.8
C22—C23—H23	119.6	O15—C32—C33	109.5 (3)
C24—C23—C22	120.8 (2)	H32A—C32—H32B	108.2
C24—C23—H23	119.6	C33—C32—H32A	109.8
C27—C26—C25	119.3 (2)	C33—C32—H32B	109.8
C27—C26—C31	119.6 (2)	O16—C33—C32	110.0 (4)
C31—C26—C25	121.0 (2)	O16—C33—H33A	109.7
O9—C64—C19	118.04 (19)	O16—C33—H33B	109.7
O10—C64—O9	124.99 (19)	C32—C33—H33A	109.7
O10—C64—C19	116.97 (19)	C32—C33—H33B	109.7
C14—C9—C8	120.40 (18)	H33A—C33—H33B	108.2
C14—C9—C10	119.00 (17)	O18—C34—H34A	109.1
C10—C9—C8	120.60 (18)	O18—C34—H34B	110.4
O3—C5—C6	123.14 (19)	O18—C34—H34C	108.8
O3—C5—C4	117.15 (19)	H34A—C34—H34B	109.5
C6—C5—C4	119.71 (18)	H34A—C34—H34C	109.5
C5—C4—H4	120.2	H34B—C34—H34C	109.5
C3—C4—C5	119.7 (2)		

Cu1^{i}—O5—C8—O4	4.5 (3)	C13—C12—C11—C10	4.1 (3)
Cu1^{i}—O5—C8—C9	−174.02 (13)	C13—C14—C9—C8	−175.80 (17)
Cu1—O1—C1—O2	−0.3 (3)	C13—C14—C9—C10	3.2 (3)
Cu1—O1—C1—C2	179.56 (13)	C8—C9—C10—C11	177.83 (18)
Cu1—O4—C8—O5	−2.8 (3)	C1—C2—C3—C4	−179.21 (19)
Cu1—O4—C8—C9	175.68 (13)	C11—C12—C13—C14	−2.1 (3)
Cu1—O7—C15—C16	−85.9 (2)	C25—C26—C27—C28	173.5 (2)
Cu1—O7—C18—C17	84.9 (2)	C25—C26—C31—C30	−175.7 (2)
Cu1^{i}—O2—C1—O1	−1.6 (3)	C22—C23—C24—C19	−1.7 (3)
Cu1^{i}—O2—C1—C2	178.56 (13)	C6—C7—C2—C1	179.08 (18)
Cu3^{ii}—O10—C64—O9	−7.1 (3)	C6—C7—C2—C3	−1.4 (3)
Cu3—O10—C64—O9	−7.1 (3)	C6—C5—C4—C3	−1.8 (3)
Cu3—O10—C64—C19	172.42 (14)	C14—C9—C10—C11	−1.2 (3)
Cu3^{ii}—O10—C64—C19	172.42 (14)	C23—C22—C21—C20	−0.7 (3)
Cu3—O13—C25—O12	0.3 (4)	C26—C27—C28—C29	2.4 (4)
Cu3^{ii}—O13—C25—O12	0.3 (4)	C26—C31—C30—C29	2.1 (4)
Cu3^{ii}—O13—C25—C26	−177.86 (15)	C64—C19—C24—C23	179.71 (19)

Cu3—O13—C25—C26	−177.86 (15)	C64—C19—C20—C21	−178.2 (2)
Cu2—O9—C64—O10	8.3 (3)	C5—C4—C3—C2	0.3 (3)
Cu2—O9—C64—C19	−171.20 (14)	C27—C26—C31—C30	0.9 (3)
Cu2—O12—C25—O13	−0.4 (4)	C24—C19—C64—O9	−15.8 (3)
Cu2—O12—C25—C26	177.69 (15)	C24—C19—C64—O10	164.7 (2)
Cu2—O15—C32—C33	111.0 (3)	C24—C19—C20—C21	1.8 (3)
O11—C22—C23—C24	−178.86 (19)	C3—C2—C1—O1	4.8 (3)
O11—C22—C21—C20	−179.7 (2)	C3—C2—C1—O2	−175.35 (19)
O6—C12—C13—C14	178.82 (18)	C31—C26—C27—C28	−3.1 (4)
O6—C12—C11—C10	−176.77 (18)	C20—C19—C64—O9	164.2 (2)
O5—C8—C9—C14	166.29 (19)	C20—C19—C64—O10	−15.4 (3)
O5—C8—C9—C10	−12.7 (3)	C20—C19—C24—C23	−0.3 (3)
O4—C8—C9—C14	−12.3 (3)	C39—N1—C37—C38	56.5 (3)
O4—C8—C9—C10	168.72 (19)	C39—N1—C41—C42	−179.3 (2)
O7—C15—C16—O8	−58.8 (3)	C39—N1—C35—C36	68.5 (3)
O3—C5—C4—C3	177.9 (2)	C21—C22—C23—C24	2.2 (3)
O13—C25—C26—C27	15.3 (3)	C30—C29—C28—C27	0.6 (4)
O13—C25—C26—C31	−168.1 (2)	C15—O7—C18—C17	−59.7 (3)
O14—C29—C30—C31	176.0 (2)	C37—N1—C39—C40	50.7 (3)
O14—C29—C28—C27	−178.3 (2)	C37—N1—C41—C42	59.7 (3)
O12—C25—C26—C27	−163.0 (2)	C37—N1—C35—C36	−169.6 (2)
O12—C25—C26—C31	13.6 (3)	C28—C29—C30—C31	−2.8 (4)
O15—C32—C33—O16	58.4 (5)	C41—N1—C39—C40	−67.8 (3)
C12—C13—C14—C9	−1.6 (3)	C41—N1—C37—C38	175.4 (2)
C12—C11—C10—C9	−2.5 (3)	C41—N1—C35—C36	−51.3 (3)
C7—C2—C1—O1	−175.67 (18)	C17—O8—C16—C15	58.4 (3)
C7—C2—C1—O2	4.2 (3)	C35—N1—C39—C40	170.4 (2)
C7—C2—C3—C4	1.3 (3)	C35—N1—C37—C38	−63.9 (3)
C7—C6—C5—O3	−178.0 (2)	C35—N1—C41—C42	−58.9 (3)
C7—C6—C5—C4	1.7 (3)	C18—O7—C15—C16	58.6 (3)
O8—C17—C18—O7	56.1 (3)	C16—O8—C17—C18	−55.9 (3)
C2—C7—C6—C5	−0.1 (3)	C32^{ii}—O15—C32—C33	−59.8 (5)
C19—C20—C21—C22	−1.3 (3)	C33^{ii}—O16—C33—C32	−57.9 (8)

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

Bis(tetraethylammonium) tetrakis(*μ*-4-hydroxybenzoato)bis[aquacopper(II)] dinitrate (compound_6)

Crystal data

$(C_8H_{20}N)_2[Cu_2(C_7H_5O_3)_4(H_2O)_2](NO_3)_2$
M_r = 1096.07
Triclinic, $P\overline{1}$
a = 10.4964 (3) Å
b = 10.6595 (2) Å
c = 12.3226 (3) Å
α = 97.271 (2)°
β = 101.042 (2)°
γ = 108.130 (2)°
V = 1260.15 (6) Å^3

Z = 1
F(000) = 574
D_x = 1.444 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 9558 reflections
θ = 3.7–77.1°
μ = 1.72 mm^{-1}
T = 100 K
Irregular, clear blue
0.16 × 0.13 × 0.09 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.720, T_{max} = 1.000
14549 measured reflections
5136 independent reflections
4648 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 77.8°, θ_{min} = 3.7°
h = −13→12
k = −11→13
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.101
S = 1.08
5136 reflections
408 parameters
263 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.6073P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.60492 (3)	1.09668 (2)	0.06898 (2)	0.01545 (10)	
O5	0.63242 (14)	0.95587 (13)	0.15033 (12)	0.0240 (3)	
O1	0.28109 (14)	0.94966 (13)	0.02841 (12)	0.0228 (3)	
O3	−0.02189 (14)	1.21562 (14)	0.33643 (12)	0.0263 (3)	
H3	0.019917	1.290925	0.379890	0.039*	
O6	0.68959 (15)	0.43690 (13)	0.32703 (11)	0.0242 (3)	
H6	0.753157	0.471250	0.386246	0.036*	
O4	0.45124 (14)	0.79086 (13)	0.03219 (12)	0.0234 (3)	
O7	0.76600 (14)	1.25985 (13)	0.18582 (11)	0.0215 (3)	
H7A	0.818286	1.230265	0.232693	0.032*	
H7B	0.731807	1.303866	0.230401	0.032*	
O2	0.46250 (14)	1.11502 (13)	0.14613 (12)	0.0239 (3)	
O8	0.12980 (15)	0.45992 (14)	0.47304 (12)	0.0266 (3)	
O9	0.01650 (19)	0.51873 (19)	0.33412 (15)	0.0435 (4)	
O10	0.15453 (18)	0.66988 (16)	0.47954 (19)	0.0466 (5)	
N1	0.09987 (18)	0.55237 (17)	0.42740 (16)	0.0283 (4)	
C1	0.33495 (19)	1.04871 (18)	0.11060 (15)	0.0188 (4)	
C8	0.55712 (19)	0.83394 (18)	0.11426 (15)	0.0190 (4)	
C9	0.59538 (19)	0.73170 (18)	0.17232 (16)	0.0201 (4)	
C12	0.6636 (2)	0.53624 (18)	0.27773 (16)	0.0206 (4)	
C11	0.5531 (2)	0.49787 (19)	0.18333 (17)	0.0240 (4)	

H11	0.501073	0.405178	0.154204	0.029*	
C10	0.5193 (2)	0.59497 (19)	0.13213 (17)	0.0228 (4)	
H10	0.442770	0.568296	0.068363	0.027*	
C2	0.24107 (19)	1.09348 (18)	0.17038 (16)	0.0202 (4)	
C3	0.0989 (2)	1.02301 (19)	0.14088 (17)	0.0230 (4)	
H3A	0.061445	0.945147	0.082188	0.028*	
C5	0.0671 (2)	1.17952 (19)	0.28262 (16)	0.0222 (4)	
C6	0.2083 (2)	1.25261 (19)	0.31114 (17)	0.0231 (4)	
H6A	0.245456	1.331688	0.368596	0.028*	
C14	0.7095 (2)	0.76897 (18)	0.26457 (16)	0.0214 (4)	
H14	0.763967	0.861460	0.291399	0.026*	
C13	0.7441 (2)	0.67266 (19)	0.31743 (16)	0.0221 (4)	
H13	0.821883	0.698915	0.380094	0.027*	
C4	0.0116 (2)	1.0653 (2)	0.19626 (18)	0.0256 (4)	
H4	−0.085082	1.016952	0.175532	0.031*	
C7	0.29421 (19)	1.20914 (19)	0.25506 (16)	0.0215 (4)	
H7	0.390566	1.258845	0.274610	0.026*	
N2	−0.0028 (12)	0.4959 (11)	0.0084 (9)	0.0192 (11)	0.5
C17	−0.1172 (4)	0.5224 (4)	0.0560 (3)	0.0236 (8)	0.5
H17A	−0.199133	0.438736	0.033948	0.028*	0.5
H17B	−0.086343	0.542762	0.139361	0.028*	0.5
C18	−0.1611 (6)	0.6357 (5)	0.0190 (5)	0.0298 (12)	0.5
H18A	−0.192215	0.617067	−0.063448	0.045*	0.5
H18B	−0.082545	0.720514	0.044433	0.045*	0.5
H18C	−0.236870	0.643029	0.052121	0.045*	0.5
C15	−0.0459 (4)	0.4598 (4)	−0.1200 (3)	0.0242 (8)	0.5
H15A	−0.063548	0.537132	−0.148426	0.029*	0.5
H15B	0.032565	0.446804	−0.147705	0.029*	0.5
C16	−0.1721 (17)	0.3355 (16)	−0.1701 (11)	0.031 (3)	0.5
H16A	−0.185596	0.315055	−0.252165	0.047*	0.5
H16B	−0.253464	0.351579	−0.152284	0.047*	0.5
H16C	−0.159114	0.259320	−0.138364	0.047*	0.5
C19	0.0264 (4)	0.3808 (4)	0.0570 (3)	0.0241 (8)	0.5
H19A	0.054508	0.407958	0.140285	0.029*	0.5
H19B	−0.060419	0.302142	0.036949	0.029*	0.5
C20	0.1372 (6)	0.3374 (6)	0.0175 (5)	0.0286 (11)	0.5
H20A	0.111914	0.312214	−0.065071	0.043*	0.5
H20B	0.145599	0.259952	0.049559	0.043*	0.5
H20C	0.225811	0.412078	0.042408	0.043*	0.5
C21	0.1260 (4)	0.6212 (4)	0.0365 (3)	0.0235 (8)	0.5
H21A	0.195413	0.600106	0.000420	0.028*	0.5
H21B	0.101573	0.692501	0.002320	0.028*	0.5
C22	0.1922 (17)	0.6771 (15)	0.1607 (11)	0.027 (2)	0.5
H22A	0.272970	0.758069	0.169956	0.041*	0.5
H22B	0.221172	0.609257	0.195169	0.041*	0.5
H22C	0.125245	0.700018	0.197558	0.041*	0.5
N3	0.5007 (12)	1.0017 (12)	0.5000 (11)	0.0263 (5)	0.5
C23	0.4801 (5)	0.8872 (4)	0.4042 (4)	0.0298 (9)	0.5

H23A	0.556981	0.914792	0.366738	0.036*	0.5
H23B	0.393363	0.873525	0.347957	0.036*	0.5
C24	0.4731 (16)	0.7527 (9)	0.4381 (9)	0.032 (2)	0.5
H24A	0.453775	0.684151	0.370408	0.048*	0.5
H24B	0.399225	0.725335	0.477494	0.048*	0.5
H24C	0.561641	0.762451	0.488200	0.048*	0.5
C25	0.3769 (5)	0.9742 (4)	0.5518 (4)	0.0314 (9)	0.5
H25A	0.399375	1.045657	0.619343	0.038*	0.5
H25B	0.361414	0.887484	0.577378	0.038*	0.5
C26	0.2426 (10)	0.9682 (13)	0.4729 (9)	0.035 (2)	0.5
H26A	0.220416	0.899518	0.404664	0.052*	0.5
H26B	0.253938	1.056106	0.452203	0.052*	0.5
H26C	0.167310	0.945044	0.511319	0.052*	0.5
C27	0.5254 (4)	1.1278 (4)	0.4499 (4)	0.0276 (8)	0.5
H27A	0.449016	1.110412	0.382579	0.033*	0.5
H27B	0.612095	1.145372	0.424374	0.033*	0.5
C28	0.5359 (17)	1.2542 (9)	0.5301 (9)	0.032 (2)	0.5
H28A	0.554704	1.330911	0.491972	0.047*	0.5
H28B	0.611245	1.272653	0.597309	0.047*	0.5
H28C	0.448636	1.240223	0.552499	0.047*	0.5
C29	0.6233 (5)	1.0155 (5)	0.5948 (4)	0.0352 (10)	0.5
H29A	0.597152	0.938412	0.633124	0.042*	0.5
H29B	0.642991	1.098612	0.650726	0.042*	0.5
C30	0.7557 (9)	1.0208 (15)	0.5584 (10)	0.043 (3)	0.5
H30A	0.742187	0.933482	0.512399	0.064*	0.5
H30B	0.831779	1.041044	0.625397	0.064*	0.5
H30C	0.778193	1.091153	0.514052	0.064*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.01445 (14)	0.01505 (14)	0.01602 (15)	0.00494 (10)	0.00401 (10)	0.00005 (10)
O5	0.0258 (7)	0.0177 (6)	0.0233 (7)	0.0051 (5)	0.0000 (5)	0.0016 (5)
O1	0.0188 (6)	0.0231 (6)	0.0242 (7)	0.0067 (5)	0.0073 (5)	−0.0049 (5)
O3	0.0211 (7)	0.0299 (7)	0.0261 (7)	0.0108 (6)	0.0061 (6)	−0.0064 (6)
O6	0.0290 (7)	0.0208 (6)	0.0209 (7)	0.0102 (5)	0.0000 (5)	0.0027 (5)
O4	0.0222 (7)	0.0186 (6)	0.0263 (7)	0.0061 (5)	0.0007 (5)	0.0041 (5)
O7	0.0195 (6)	0.0191 (6)	0.0200 (6)	0.0046 (5)	0.0006 (5)	−0.0045 (5)
O2	0.0170 (6)	0.0275 (7)	0.0233 (7)	0.0059 (5)	0.0056 (5)	−0.0054 (5)
O8	0.0292 (7)	0.0251 (7)	0.0248 (7)	0.0126 (6)	0.0030 (6)	−0.0001 (6)
O9	0.0403 (9)	0.0550 (10)	0.0333 (9)	0.0140 (8)	0.0031 (7)	0.0173 (8)
O10	0.0317 (9)	0.0224 (8)	0.0789 (14)	0.0040 (6)	0.0090 (9)	0.0062 (8)
N1	0.0229 (8)	0.0283 (9)	0.0337 (10)	0.0067 (7)	0.0100 (7)	0.0074 (7)
C1	0.0190 (8)	0.0198 (8)	0.0186 (9)	0.0082 (7)	0.0047 (7)	0.0029 (7)
C8	0.0181 (8)	0.0213 (8)	0.0186 (9)	0.0074 (7)	0.0069 (7)	0.0025 (7)
C9	0.0200 (9)	0.0196 (8)	0.0211 (9)	0.0069 (7)	0.0065 (7)	0.0025 (7)
C12	0.0235 (9)	0.0202 (8)	0.0202 (9)	0.0105 (7)	0.0063 (7)	0.0027 (7)
C11	0.0260 (10)	0.0182 (8)	0.0249 (10)	0.0061 (7)	0.0028 (8)	0.0025 (7)

C10	0.0218 (9)	0.0215 (9)	0.0218 (9)	0.0060 (7)	0.0015 (7)	0.0024 (7)
C2	0.0203 (9)	0.0203 (8)	0.0210 (9)	0.0086 (7)	0.0060 (7)	0.0016 (7)
C3	0.0195 (9)	0.0239 (9)	0.0240 (10)	0.0081 (7)	0.0057 (7)	−0.0031 (7)
C5	0.0226 (9)	0.0267 (9)	0.0204 (9)	0.0132 (8)	0.0064 (7)	0.0016 (7)
C6	0.0236 (9)	0.0222 (9)	0.0224 (9)	0.0095 (7)	0.0044 (7)	−0.0025 (7)
C14	0.0219 (9)	0.0183 (8)	0.0221 (9)	0.0060 (7)	0.0044 (7)	0.0010 (7)
C13	0.0217 (9)	0.0223 (9)	0.0200 (9)	0.0080 (7)	0.0013 (7)	0.0007 (7)
C4	0.0180 (9)	0.0271 (9)	0.0281 (10)	0.0067 (7)	0.0050 (8)	−0.0033 (8)
C7	0.0185 (9)	0.0232 (9)	0.0215 (9)	0.0069 (7)	0.0040 (7)	0.0018 (7)
N2	0.0192 (13)	0.0211 (15)	0.016 (3)	0.0034 (11)	0.0052 (15)	0.0045 (16)
C17	0.0194 (18)	0.0242 (18)	0.0229 (19)	0.0016 (14)	0.0069 (15)	0.0015 (15)
C18	0.032 (3)	0.027 (3)	0.031 (3)	0.009 (2)	0.010 (2)	0.007 (2)
C15	0.0253 (19)	0.0273 (19)	0.0151 (17)	0.0035 (15)	0.0042 (14)	0.0027 (14)
C16	0.025 (5)	0.032 (4)	0.021 (3)	−0.008 (3)	0.005 (3)	0.000 (3)
C19	0.0227 (18)	0.0224 (17)	0.0217 (18)	0.0010 (14)	0.0024 (15)	0.0067 (15)
C20	0.027 (3)	0.028 (3)	0.033 (3)	0.011 (2)	0.008 (2)	0.008 (2)
C21	0.0219 (18)	0.0233 (18)	0.0197 (18)	0.0003 (15)	0.0057 (15)	0.0031 (14)
C22	0.023 (5)	0.029 (4)	0.021 (3)	−0.002 (3)	0.005 (3)	0.000 (3)
N3	0.0286 (12)	0.0245 (12)	0.0235 (12)	0.0114 (10)	0.0013 (10)	−0.0012 (10)
C23	0.031 (2)	0.0286 (19)	0.025 (2)	0.0119 (17)	0.0014 (17)	−0.0070 (16)
C24	0.026 (3)	0.027 (3)	0.044 (6)	0.006 (2)	0.018 (5)	0.002 (3)
C25	0.039 (2)	0.029 (2)	0.027 (2)	0.0119 (18)	0.0108 (18)	0.0028 (16)
C26	0.033 (3)	0.019 (3)	0.056 (6)	0.010 (2)	0.014 (3)	0.015 (4)
C27	0.028 (2)	0.0283 (19)	0.025 (2)	0.0102 (16)	0.0019 (16)	0.0030 (16)
C28	0.031 (3)	0.022 (3)	0.041 (5)	0.005 (2)	0.017 (4)	−0.001 (3)
C29	0.042 (2)	0.029 (2)	0.028 (2)	0.0161 (19)	−0.0076 (19)	−0.0029 (17)
C30	0.024 (3)	0.034 (4)	0.061 (6)	0.002 (2)	−0.005 (3)	0.024 (4)

Geometric parameters (Å, º)

Cu1—O5	1.9700 (13)	C18—H18B	0.9800
Cu1—O1^{i}	1.9707 (13)	C18—H18C	0.9800
Cu1—O4^{i}	1.9686 (13)	C15—H15A	0.9900
Cu1—O7	2.1355 (13)	C15—H15B	0.9900
Cu1—O2	1.9636 (13)	C15—C16	1.509 (11)
O5—C8	1.261 (2)	C16—H16A	0.9800
O1—C1	1.264 (2)	C16—H16B	0.9800
O3—H3	0.8400	C16—H16C	0.9800
O3—C5	1.362 (2)	C19—H19A	0.9900
O6—H6	0.8400	C19—H19B	0.9900
O6—C12	1.358 (2)	C19—C20	1.516 (6)
O4—C8	1.266 (2)	C20—H20A	0.9800
O7—H7A	0.8756	C20—H20B	0.9800
O7—H7B	0.8750	C20—H20C	0.9800
O2—C1	1.262 (2)	C21—H21A	0.9900
O8—N1	1.285 (2)	C21—H21B	0.9900
O9—N1	1.237 (3)	C21—C22	1.512 (11)
O10—N1	1.232 (2)	C22—H22A	0.9800

C1—C2	1.491 (2)	C22—H22B	0.9800
C8—C9	1.490 (3)	C22—H22C	0.9800
C9—C10	1.396 (3)	N3—C23	1.518 (12)
C9—C14	1.397 (3)	N3—C25	1.519 (12)
C12—C11	1.391 (3)	N3—C27	1.520 (12)
C12—C13	1.402 (3)	N3—C29	1.518 (12)
C11—H11	0.9500	C23—H23A	0.9900
C11—C10	1.380 (3)	C23—H23B	0.9900
C10—H10	0.9500	C23—C24	1.527 (9)
C2—C3	1.396 (3)	C24—H24A	0.9800
C2—C7	1.394 (3)	C24—H24B	0.9800
C3—H3A	0.9500	C24—H24C	0.9800
C3—C4	1.389 (3)	C25—H25A	0.9900
C5—C6	1.393 (3)	C25—H25B	0.9900
C5—C4	1.395 (3)	C25—C26	1.530 (9)
C6—H6A	0.9500	C26—H26A	0.9800
C6—C7	1.387 (3)	C26—H26B	0.9800
C14—H14	0.9500	C26—H26C	0.9800
C14—C13	1.386 (3)	C27—H27A	0.9900
C13—H13	0.9500	C27—H27B	0.9900
C4—H4	0.9500	C27—C28	1.528 (9)
C7—H7	0.9500	C28—H28A	0.9800
N2—C17	1.518 (11)	C28—H28B	0.9800
N2—C15	1.523 (11)	C28—H28C	0.9800
N2—C19	1.519 (11)	C29—H29A	0.9900
N2—C21	1.520 (11)	C29—H29B	0.9900
C17—H17A	0.9900	C29—C30	1.527 (10)
C17—H17B	0.9900	C30—H30A	0.9800
C17—C18	1.511 (6)	C30—H30B	0.9800
C18—H18A	0.9800	C30—H30C	0.9800

O5—Cu1—O1^{i}	87.67 (6)	C16—C15—H15A	108.4
O5—Cu1—O7	95.42 (5)	C16—C15—H15B	108.4
O1^{i}—Cu1—O7	97.36 (5)	C15—C16—H16A	109.5
O4^{i}—Cu1—O5	169.17 (5)	C15—C16—H16B	109.5
O4^{i}—Cu1—O1^{i}	91.24 (6)	C15—C16—H16C	109.5
O4^{i}—Cu1—O7	95.40 (5)	H16A—C16—H16B	109.5
O2—Cu1—O5	91.45 (6)	H16A—C16—H16C	109.5
O2—Cu1—O1^{i}	169.18 (5)	H16B—C16—H16C	109.5
O2—Cu1—O4^{i}	87.61 (6)	N2—C19—H19A	108.5
O2—Cu1—O7	93.46 (5)	N2—C19—H19B	108.5
C8—O5—Cu1	121.04 (12)	H19A—C19—H19B	107.5
C1—O1—Cu1^{i}	119.98 (12)	C20—C19—N2	115.0 (5)
C5—O3—H3	109.5	C20—C19—H19A	108.5
C12—O6—H6	109.5	C20—C19—H19B	108.5
C8—O4—Cu1^{i}	124.09 (12)	C19—C20—H20A	109.5
Cu1—O7—H7A	111.1	C19—C20—H20B	109.5
Cu1—O7—H7B	110.8	C19—C20—H20C	109.5

H7A—O7—H7B	103.1	H20A—C20—H20B	109.5
C1—O2—Cu1	124.80 (12)	H20A—C20—H20C	109.5
O9—N1—O8	118.39 (17)	H20B—C20—H20C	109.5
O10—N1—O8	118.21 (18)	N2—C21—H21A	108.4
O10—N1—O9	123.40 (19)	N2—C21—H21B	108.4
O1—C1—C2	117.98 (16)	H21A—C21—H21B	107.4
O2—C1—O1	125.49 (17)	C22—C21—N2	115.7 (7)
O2—C1—C2	116.52 (16)	C22—C21—H21A	108.4
O5—C8—O4	125.28 (17)	C22—C21—H21B	108.4
O5—C8—C9	117.71 (16)	C21—C22—H22A	109.5
O4—C8—C9	117.00 (16)	C21—C22—H22B	109.5
C10—C9—C8	119.85 (17)	C21—C22—H22C	109.5
C10—C9—C14	118.64 (17)	H22A—C22—H22B	109.5
C14—C9—C8	121.47 (16)	H22A—C22—H22C	109.5
O6—C12—C11	117.31 (17)	H22B—C22—H22C	109.5
O6—C12—C13	122.66 (17)	C23—N3—C25	111.4 (8)
C11—C12—C13	120.03 (17)	C23—N3—C27	106.1 (8)
C12—C11—H11	120.1	C23—N3—C29	110.2 (7)
C10—C11—C12	119.73 (17)	C25—N3—C27	112.3 (8)
C10—C11—H11	120.1	C29—N3—C25	105.8 (8)
C9—C10—H10	119.4	C29—N3—C27	111.1 (8)
C11—C10—C9	121.20 (18)	N3—C23—H23A	108.4
C11—C10—H10	119.4	N3—C23—H23B	108.4
C3—C2—C1	120.91 (16)	N3—C23—C24	115.5 (6)
C7—C2—C1	120.11 (17)	H23A—C23—H23B	107.5
C7—C2—C3	118.96 (17)	C24—C23—H23A	108.4
C2—C3—H3A	119.6	C24—C23—H23B	108.4
C4—C3—C2	120.81 (17)	C23—C24—H24A	109.5
C4—C3—H3A	119.6	C23—C24—H24B	109.5
O3—C5—C6	122.17 (17)	C23—C24—H24C	109.5
O3—C5—C4	117.46 (17)	H24A—C24—H24B	109.5
C6—C5—C4	120.37 (17)	H24A—C24—H24C	109.5
C5—C6—H6A	120.2	H24B—C24—H24C	109.5
C7—C6—C5	119.54 (17)	N3—C25—H25A	108.6
C7—C6—H6A	120.2	N3—C25—H25B	108.6
C9—C14—H14	119.6	N3—C25—C26	114.8 (6)
C13—C14—C9	120.81 (17)	H25A—C25—H25B	107.5
C13—C14—H14	119.6	C26—C25—H25A	108.6
C12—C13—H13	120.2	C26—C25—H25B	108.6
C14—C13—C12	119.52 (18)	C25—C26—H26A	109.5
C14—C13—H13	120.2	C25—C26—H26B	109.5
C3—C4—C5	119.42 (18)	C25—C26—H26C	109.5
C3—C4—H4	120.3	H26A—C26—H26B	109.5
C5—C4—H4	120.3	H26A—C26—H26C	109.5
C2—C7—H7	119.6	H26B—C26—H26C	109.5
C6—C7—C2	120.87 (18)	N3—C27—H27A	108.6
C6—C7—H7	119.6	N3—C27—H27B	108.6
C17—N2—C15	110.9 (7)	N3—C27—C28	114.9 (6)

C17—N2—C19	107.0 (7)	H27A—C27—H27B	107.5
C17—N2—C21	111.5 (7)	C28—C27—H27A	108.6
C19—N2—C15	110.8 (7)	C28—C27—H27B	108.6
C19—N2—C21	111.7 (7)	C27—C28—H28A	109.5
C21—N2—C15	105.0 (7)	C27—C28—H28B	109.5
N2—C17—H17A	108.4	C27—C28—H28C	109.5
N2—C17—H17B	108.4	H28A—C28—H28B	109.5
H17A—C17—H17B	107.4	H28A—C28—H28C	109.5
C18—C17—N2	115.6 (5)	H28B—C28—H28C	109.5
C18—C17—H17A	108.4	N3—C29—H29A	108.5
C18—C17—H17B	108.4	N3—C29—H29B	108.5
C17—C18—H18A	109.5	N3—C29—C30	115.2 (7)
C17—C18—H18B	109.5	H29A—C29—H29B	107.5
C17—C18—H18C	109.5	C30—C29—H29A	108.5
H18A—C18—H18B	109.5	C30—C29—H29B	108.5
H18A—C18—H18C	109.5	C29—C30—H30A	109.5
H18B—C18—H18C	109.5	C29—C30—H30B	109.5
N2—C15—H15A	108.4	C29—C30—H30C	109.5
N2—C15—H15B	108.4	H30A—C30—H30B	109.5
H15A—C15—H15B	107.5	H30A—C30—H30C	109.5
C16—C15—N2	115.5 (7)	H30B—C30—H30C	109.5

Cu1—O5—C8—O4	−7.3 (3)	C5—C6—C7—C2	0.2 (3)
Cu1—O5—C8—C9	172.57 (12)	C6—C5—C4—C3	1.7 (3)
Cu1^{i}—O1—C1—O2	−8.7 (3)	C14—C9—C10—C11	1.5 (3)
Cu1^{i}—O1—C1—C2	170.35 (12)	C13—C12—C11—C10	−2.9 (3)
Cu1^{i}—O4—C8—O5	8.1 (3)	C4—C5—C6—C7	−1.7 (3)
Cu1^{i}—O4—C8—C9	−171.75 (12)	C7—C2—C3—C4	−1.3 (3)
Cu1—O2—C1—O1	9.2 (3)	C17—N2—C15—C16	−60.3 (13)
Cu1—O2—C1—C2	−169.79 (12)	C17—N2—C19—C20	179.0 (5)
O5—C8—C9—C10	−176.90 (18)	C17—N2—C21—C22	61.0 (12)
O5—C8—C9—C14	0.9 (3)	C15—N2—C17—C18	−59.8 (8)
O1—C1—C2—C3	3.9 (3)	C15—N2—C19—C20	58.0 (8)
O1—C1—C2—C7	−174.45 (17)	C15—N2—C21—C22	−178.8 (10)
O3—C5—C6—C7	178.53 (18)	C19—N2—C17—C18	179.2 (5)
O3—C5—C4—C3	−178.51 (18)	C19—N2—C15—C16	58.4 (13)
O6—C12—C11—C10	177.89 (17)	C19—N2—C21—C22	−58.7 (12)
O6—C12—C13—C14	−178.41 (17)	C21—N2—C17—C18	56.8 (8)
O4—C8—C9—C10	3.0 (3)	C21—N2—C15—C16	179.1 (11)
O4—C8—C9—C14	−179.28 (17)	C21—N2—C19—C20	−58.7 (8)
O2—C1—C2—C3	−177.03 (18)	C23—N3—C25—C26	−65.0 (10)
O2—C1—C2—C7	4.7 (3)	C23—N3—C27—C28	174.7 (8)
C1—C2—C3—C4	−179.64 (18)	C23—N3—C29—C30	49.9 (11)
C1—C2—C7—C6	179.67 (18)	C25—N3—C23—C24	−65.6 (11)
C8—C9—C10—C11	179.29 (18)	C25—N3—C27—C28	52.8 (11)
C8—C9—C14—C13	−179.73 (17)	C25—N3—C29—C30	170.5 (8)
C9—C14—C13—C12	0.0 (3)	C27—N3—C23—C24	171.9 (8)
C12—C11—C10—C9	1.0 (3)	C27—N3—C25—C26	53.8 (10)

C11—C12—C13—C14	2.5 (3)	C27—N3—C29—C30	−67.4 (10)
C10—C9—C14—C13	−1.9 (3)	C29—N3—C23—C24	51.5 (11)
C2—C3—C4—C5	−0.2 (3)	C29—N3—C25—C26	175.2 (7)
C3—C2—C7—C6	1.3 (3)	C29—N3—C27—C28	−65.5 (11)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*.

Bis[8-(dimethylamino)-*N*,*N*-dimethylnaphthalen-1-aminium] bis(*μ*-4-hydroxybenzoato)bis(*μ*-4-oxidobenzoato)bis[aquacopper(II)] dioxane trisolvate monohydrate (compound_7)

Crystal data

$(C_{14}H_{19}N_2)_2[Cu_2(C_7H_5O_3)_2(C_7H_4O_3)_2(H_2O)_2]\cdot 3(C_4H_8O_2)\cdot H_2O$
M_r = 1420.47
Triclinic, $P\overline{1}$
a = 10.2155 (2) Å
b = 12.0897 (3) Å
c = 15.7111 (4) Å
α = 69.581 (3)°
β = 75.988 (2)°
γ = 79.651 (2)°
V = 1754.64 (8) Å^3
Z = 1
$F(000)$ = 746
D_x = 1.344 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 13870 reflections
θ = 3.9–76.4°
μ = 1.38 mm^{-1}
T = 100 K
Irregular, dark green
0.19 × 0.16 × 0.12 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2021)
T_{min} = 0.890, T_{max} = 1.000
21601 measured reflections
6618 independent reflections
5772 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 70.1°, θ_{min} = 3.9°
h = −11→12
k = −14→14
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.189
S = 1.05
6618 reflections
361 parameters
3 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.104P)^2 + 1.3001P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.05 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 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. H atoms on the non-coordinated water molecule have not been modelled.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.41243 (3)	1.03328 (4)	0.56430 (3)	0.05295 (19)	
O2	0.28374 (18)	1.0623 (2)	0.48196 (14)	0.0533 (5)	
O5	0.53049 (19)	0.8085 (2)	0.50342 (15)	0.0606 (6)	
O1	0.43470 (19)	1.0072 (2)	0.37024 (15)	0.0642 (6)	
O4	0.3805 (2)	0.8663 (2)	0.61457 (15)	0.0624 (6)	
O7	0.2650 (2)	1.0729 (3)	0.67983 (15)	0.0729 (7)	
H7A	0.243652	1.006377	0.724847	0.109*	
H7B	0.294519	1.114115	0.707290	0.109*	
N1	0.1882 (2)	0.4847 (3)	0.26458 (17)	0.0543 (6)	
O3	−0.0933 (2)	1.1176 (3)	0.20193 (17)	0.0803 (8)	
N2	0.1811 (3)	0.7077 (3)	0.23467 (18)	0.0642 (7)	
C19	0.1764 (2)	0.5416 (3)	0.48681 (19)	0.0466 (6)	
C7	0.2451 (3)	1.0837 (3)	0.2541 (2)	0.0472 (6)	
H7	0.337867	1.076527	0.225115	0.057*	
C2	0.2116 (2)	1.0756 (2)	0.34692 (19)	0.0437 (6)	
C1	0.3175 (3)	1.0479 (3)	0.4042 (2)	0.0488 (7)	
C18	0.1704 (3)	0.4236 (3)	0.5491 (2)	0.0506 (7)	
H18	0.167908	0.409167	0.612828	0.061*	
C16	0.1727 (3)	0.3519 (3)	0.4252 (2)	0.0517 (7)	
H16	0.172479	0.287161	0.404197	0.062*	
C6	0.1453 (3)	1.1021 (3)	0.2031 (2)	0.0518 (7)	
H6	0.169295	1.110413	0.138981	0.062*	
C20	0.1786 (3)	0.5633 (3)	0.39178 (19)	0.0466 (6)	
C3	0.0745 (3)	1.0892 (3)	0.38825 (19)	0.0473 (6)	
H3	0.050180	1.088584	0.450726	0.057*	
C9	0.4164 (3)	0.6634 (3)	0.6303 (2)	0.0582 (8)	
C21	0.1819 (3)	0.6831 (3)	0.3323 (2)	0.0538 (7)	
C15	0.1778 (3)	0.4643 (3)	0.3627 (2)	0.0490 (7)	
C8	0.4452 (3)	0.7875 (3)	0.5789 (2)	0.0578 (8)	
C17	0.1680 (3)	0.3309 (3)	0.5196 (2)	0.0519 (7)	
H17	0.163160	0.252830	0.562284	0.062*	
C24	0.1809 (3)	0.6368 (3)	0.5184 (2)	0.0518 (7)	
H24	0.180192	0.621831	0.582038	0.062*	
O6	0.3297 (4)	0.3177 (3)	0.7702 (3)	0.1148 (13)	
H6A	0.371348	0.246340	0.764438	0.172*	
C4	−0.0256 (3)	1.1036 (3)	0.3387 (2)	0.0560 (8)	
H4	−0.118384	1.110321	0.367757	0.067*	
C22	0.1854 (3)	0.7737 (3)	0.3652 (2)	0.0585 (8)	
H22	0.187458	0.852888	0.324297	0.070*	
C5	0.0092 (3)	1.1083 (3)	0.2468 (2)	0.0578 (8)	
C23	0.1861 (3)	0.7495 (3)	0.4588 (2)	0.0577 (7)	
H23	0.190141	0.812351	0.480905	0.069*	
C10	0.4835 (3)	0.5708 (4)	0.5974 (3)	0.0669 (10)	
H10	0.549054	0.587890	0.541371	0.080*	
C14	0.3211 (3)	0.6365 (4)	0.7131 (2)	0.0663 (10)	

H14	0.276047	0.698248	0.737315	0.080*	
C12	0.3582 (4)	0.4321 (4)	0.7257 (3)	0.0813 (12)	
C13	0.2922 (3)	0.5228 (4)	0.7595 (3)	0.0752 (11)	
H13	0.226383	0.505690	0.815325	0.090*	
C11	0.4570 (3)	0.4568 (4)	0.6437 (3)	0.0734 (10)	
H11	0.504593	0.394417	0.621063	0.088*	
C25	0.3216 (4)	0.4303 (4)	0.2238 (3)	0.0750 (11)	
H25A	0.395746	0.462606	0.234019	0.113*	
H25B	0.327582	0.448714	0.157179	0.113*	
H25C	0.328733	0.344044	0.253574	0.113*	
C26	0.0748 (4)	0.4427 (4)	0.2452 (3)	0.0825 (12)	
H26A	0.081251	0.355873	0.269691	0.124*	
H26B	0.079407	0.468610	0.178199	0.124*	
H26C	−0.011534	0.475856	0.274947	0.124*	
C28	0.0512 (5)	0.7777 (4)	0.2101 (3)	0.0926 (14)	
H28A	−0.025759	0.738063	0.253488	0.139*	
H28B	0.047433	0.783342	0.146975	0.139*	
H28C	0.046878	0.857623	0.213779	0.139*	
C27	0.3000 (5)	0.7669 (5)	0.1724 (3)	0.0955 (14)	
H27A	0.299074	0.843744	0.180619	0.143*	
H27B	0.295990	0.779193	0.107959	0.143*	
H27C	0.383474	0.716950	0.187273	0.143*	
H1	0.211 (5)	0.598 (5)	0.228 (4)	0.114 (17)*	
O8	0.5441 (8)	0.8767 (9)	0.2438 (7)	0.132 (3)	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0186 (2)	0.0960 (4)	0.0415 (3)	0.00493 (19)	−0.00648 (16)	−0.0234 (2)
O2	0.0224 (8)	0.0920 (15)	0.0451 (11)	0.0053 (9)	−0.0099 (7)	−0.0247 (10)
O5	0.0272 (9)	0.1013 (17)	0.0495 (12)	−0.0037 (10)	−0.0006 (8)	−0.0257 (11)
O1	0.0225 (9)	0.120 (2)	0.0487 (11)	0.0102 (10)	−0.0099 (8)	−0.0325 (12)
O4	0.0299 (10)	0.0950 (17)	0.0505 (12)	0.0046 (10)	−0.0019 (8)	−0.0180 (11)
O7	0.0354 (11)	0.131 (2)	0.0458 (12)	0.0177 (12)	−0.0058 (9)	−0.0338 (13)
N1	0.0398 (13)	0.0727 (16)	0.0472 (13)	0.0024 (11)	−0.0078 (10)	−0.0196 (12)
O3	0.0341 (11)	0.146 (3)	0.0556 (14)	−0.0047 (13)	−0.0214 (10)	−0.0195 (14)
N2	0.0664 (18)	0.0680 (17)	0.0434 (14)	0.0039 (13)	−0.0080 (12)	−0.0068 (12)
C19	0.0216 (11)	0.0658 (17)	0.0450 (14)	0.0058 (11)	−0.0058 (10)	−0.0142 (12)
C7	0.0256 (12)	0.0633 (17)	0.0459 (15)	−0.0017 (11)	−0.0062 (10)	−0.0113 (12)
C2	0.0241 (12)	0.0573 (15)	0.0449 (14)	−0.0002 (10)	−0.0088 (10)	−0.0111 (11)
C1	0.0221 (12)	0.0710 (18)	0.0481 (15)	0.0000 (11)	−0.0072 (11)	−0.0149 (13)
C18	0.0249 (12)	0.0723 (19)	0.0436 (15)	0.0066 (12)	−0.0073 (10)	−0.0103 (13)
C16	0.0313 (13)	0.0606 (17)	0.0581 (17)	0.0025 (12)	−0.0051 (12)	−0.0187 (14)
C6	0.0346 (14)	0.0733 (19)	0.0403 (14)	−0.0025 (12)	−0.0089 (11)	−0.0097 (13)
C20	0.0265 (12)	0.0584 (16)	0.0468 (15)	0.0060 (11)	−0.0055 (10)	−0.0129 (12)
C3	0.0262 (12)	0.0704 (18)	0.0411 (14)	−0.0011 (11)	−0.0069 (10)	−0.0143 (12)
C9	0.0220 (12)	0.096 (2)	0.0504 (16)	0.0020 (13)	−0.0126 (11)	−0.0159 (16)
C21	0.0378 (14)	0.0630 (18)	0.0478 (16)	0.0057 (12)	−0.0059 (12)	−0.0090 (13)

C15	0.0285 (12)	0.0656 (17)	0.0463 (15)	0.0045 (11)	−0.0061 (11)	−0.0152 (13)
C8	0.0215 (12)	0.102 (2)	0.0467 (16)	0.0049 (13)	−0.0125 (11)	−0.0212 (16)
C17	0.0279 (13)	0.0606 (17)	0.0529 (16)	0.0043 (11)	−0.0045 (11)	−0.0073 (13)
C24	0.0279 (13)	0.0738 (19)	0.0514 (16)	0.0046 (12)	−0.0078 (11)	−0.0219 (14)
O6	0.083 (2)	0.109 (3)	0.124 (3)	−0.0259 (19)	−0.030 (2)	0.010 (2)
C4	0.0252 (13)	0.083 (2)	0.0523 (17)	0.0019 (13)	−0.0088 (11)	−0.0155 (15)
C22	0.0465 (16)	0.0547 (17)	0.0626 (19)	0.0037 (13)	−0.0057 (14)	−0.0119 (14)
C5	0.0289 (13)	0.091 (2)	0.0475 (16)	−0.0019 (13)	−0.0143 (11)	−0.0123 (15)
C23	0.0366 (15)	0.0676 (19)	0.0652 (19)	0.0024 (13)	−0.0060 (13)	−0.0231 (15)
C10	0.0266 (14)	0.101 (3)	0.063 (2)	0.0046 (15)	−0.0135 (13)	−0.0154 (18)
C14	0.0295 (14)	0.113 (3)	0.0489 (17)	−0.0024 (16)	−0.0140 (12)	−0.0146 (18)
C12	0.053 (2)	0.099 (3)	0.080 (3)	−0.011 (2)	−0.0307 (19)	−0.001 (2)
C13	0.0372 (16)	0.112 (3)	0.060 (2)	−0.0057 (18)	−0.0115 (14)	−0.007 (2)
C11	0.0379 (17)	0.093 (3)	0.080 (3)	0.0020 (16)	−0.0188 (16)	−0.016 (2)
C25	0.060 (2)	0.101 (3)	0.056 (2)	0.0127 (19)	−0.0041 (16)	−0.0306 (19)
C26	0.066 (2)	0.119 (3)	0.064 (2)	−0.014 (2)	−0.0213 (18)	−0.025 (2)
C28	0.099 (3)	0.100 (3)	0.063 (2)	0.027 (3)	−0.034 (2)	−0.013 (2)
C27	0.100 (3)	0.107 (3)	0.055 (2)	−0.020 (3)	0.012 (2)	−0.010 (2)
O8	0.084 (5)	0.199 (9)	0.164 (7)	0.039 (5)	−0.057 (5)	−0.126 (7)

Geometric parameters (Å, º)

Cu1—O2	1.9661 (19)	C3—H3	0.9500
Cu1—O5^{i}	1.952 (3)	C3—C4	1.381 (4)
Cu1—O1^{i}	1.972 (2)	C9—C8	1.480 (5)
Cu1—O4	1.951 (3)	C9—C10	1.396 (5)
Cu1—O7	2.195 (2)	C9—C14	1.397 (4)
O2—C1	1.251 (4)	C21—C22	1.373 (5)
O5—C8	1.265 (4)	C17—H17	0.9500
O1—C1	1.273 (3)	C24—H24	0.9500
O4—C8	1.274 (4)	C24—C23	1.360 (5)
O7—H7A	0.8868	O6—H6A	0.9151
O7—H7B	0.8899	O6—C12	1.363 (6)
N1—C15	1.454 (4)	C4—H4	0.9500
N1—C25	1.500 (4)	C4—C5	1.384 (4)
N1—C26	1.477 (5)	C22—H22	0.9500
N1—H1	1.33 (5)	C22—C23	1.396 (5)
O3—C5	1.366 (4)	C23—H23	0.9500
N2—C21	1.458 (4)	C10—H10	0.9500
N2—C28	1.498 (5)	C10—C11	1.357 (6)
N2—C27	1.481 (5)	C14—H14	0.9500
N2—H1	1.34 (5)	C14—C13	1.361 (6)
C19—C18	1.423 (4)	C12—C13	1.381 (7)
C19—C20	1.418 (4)	C12—C11	1.406 (6)
C19—C24	1.415 (4)	C13—H13	0.9500
C7—H7	0.9500	C11—H11	0.9500
C7—C2	1.387 (4)	C25—H25A	0.9800
C7—C6	1.387 (4)	C25—H25B	0.9800

C2—C1	1.491 (4)	C25—H25C	0.9800
C2—C3	1.400 (4)	C26—H26A	0.9800
C18—H18	0.9500	C26—H26B	0.9800
C18—C17	1.360 (5)	C26—H26C	0.9800
C16—H16	0.9500	C28—H28A	0.9800
C16—C15	1.372 (4)	C28—H28B	0.9800
C16—C17	1.405 (4)	C28—H28C	0.9800
C6—H6	0.9500	C27—H27A	0.9800
C6—C5	1.395 (4)	C27—H27B	0.9800
C20—C21	1.425 (4)	C27—H27C	0.9800
C20—C15	1.425 (4)		

O2—Cu1—O1^{i}	169.41 (8)	C16—C15—C20	120.9 (3)
O2—Cu1—O7	96.76 (8)	C20—C15—N1	118.3 (3)
O5^{i}—Cu1—O2	89.56 (9)	O5—C8—O4	124.6 (3)
O5^{i}—Cu1—O1^{i}	90.35 (10)	O5—C8—C9	118.6 (3)
O5^{i}—Cu1—O7	99.17 (11)	O4—C8—C9	116.8 (3)
O1^{i}—Cu1—O7	93.70 (8)	C18—C17—C16	119.2 (3)
O4—Cu1—O2	89.80 (10)	C18—C17—H17	120.4
O4—Cu1—O5^{i}	169.45 (9)	C16—C17—H17	120.4
O4—Cu1—O1^{i}	88.35 (11)	C19—C24—H24	119.6
O4—Cu1—O7	91.37 (11)	C23—C24—C19	120.7 (3)
C1—O2—Cu1	122.97 (16)	C23—C24—H24	119.6
C8—O5—Cu1^{i}	121.9 (2)	C12—O6—H6A	133.4
C1—O1—Cu1^{i}	121.97 (19)	C3—C4—H4	119.9
C8—O4—Cu1	123.8 (2)	C3—C4—C5	120.1 (3)
Cu1—O7—H7A	110.5	C5—C4—H4	119.9
Cu1—O7—H7B	114.9	C21—C22—H22	119.9
H7A—O7—H7B	104.2	C21—C22—C23	120.2 (3)
C15—N1—C25	111.3 (2)	C23—C22—H22	119.9
C15—N1—C26	112.7 (3)	O3—C5—C6	122.0 (3)
C15—N1—H1	102 (2)	O3—C5—C4	117.9 (3)
C25—N1—H1	99 (2)	C4—C5—C6	120.2 (3)
C26—N1—C25	110.5 (3)	C24—C23—C22	120.5 (3)
C26—N1—H1	120 (2)	C24—C23—H23	119.7
C21—N2—C28	111.0 (3)	C22—C23—H23	119.7
C21—N2—C27	112.7 (3)	C9—C10—H10	119.3
C21—N2—H1	101 (2)	C11—C10—C9	121.3 (3)
C28—N2—H1	119 (2)	C11—C10—H10	119.3
C27—N2—C28	111.1 (3)	C9—C14—H14	119.7
C27—N2—H1	102 (2)	C13—C14—C9	120.6 (4)
C20—C19—C18	119.2 (3)	C13—C14—H14	119.7
C24—C19—C18	120.9 (3)	O6—C12—C13	121.3 (4)
C24—C19—C20	119.9 (3)	O6—C12—C11	118.6 (5)
C2—C7—H7	119.5	C13—C12—C11	120.1 (4)
C2—C7—C6	121.0 (2)	C14—C13—C12	120.3 (4)
C6—C7—H7	119.5	C14—C13—H13	119.9
C7—C2—C1	121.5 (2)	C12—C13—H13	119.9

C7—C2—C3	118.9 (2)	C10—C11—C12	119.1 (4)
C3—C2—C1	119.5 (2)	C10—C11—H11	120.4
O2—C1—O1	125.5 (2)	C12—C11—H11	120.4
O2—C1—C2	118.1 (2)	N1—C25—H25A	109.5
O1—C1—C2	116.3 (3)	N1—C25—H25B	109.5
C19—C18—H18	119.2	N1—C25—H25C	109.5
C17—C18—C19	121.7 (3)	H25A—C25—H25B	109.5
C17—C18—H18	119.2	H25A—C25—H25C	109.5
C15—C16—H16	119.4	H25B—C25—H25C	109.5
C15—C16—C17	121.2 (3)	N1—C26—H26A	109.5
C17—C16—H16	119.4	N1—C26—H26B	109.5
C7—C6—H6	120.4	N1—C26—H26C	109.5
C7—C6—C5	119.3 (3)	H26A—C26—H26B	109.5
C5—C6—H6	120.4	H26A—C26—H26C	109.5
C19—C20—C21	117.3 (3)	H26B—C26—H26C	109.5
C19—C20—C15	117.9 (3)	N2—C28—H28A	109.5
C15—C20—C21	124.8 (3)	N2—C28—H28B	109.5
C2—C3—H3	119.8	N2—C28—H28C	109.5
C4—C3—C2	120.4 (3)	H28A—C28—H28B	109.5
C4—C3—H3	119.8	H28A—C28—H28C	109.5
C10—C9—C8	120.9 (3)	H28B—C28—H28C	109.5
C10—C9—C14	118.6 (4)	N2—C27—H27A	109.5
C14—C9—C8	120.5 (3)	N2—C27—H27B	109.5
C20—C21—N2	118.2 (3)	N2—C27—H27C	109.5
C22—C21—N2	120.4 (3)	H27A—C27—H27B	109.5
C22—C21—C20	121.4 (3)	H27A—C27—H27C	109.5
C16—C15—N1	120.8 (3)	H27B—C27—H27C	109.5

Cu1—O2—C1—O1	4.0 (5)	C3—C4—C5—C6	−1.9 (5)
Cu1—O2—C1—C2	−178.53 (19)	C9—C10—C11—C12	1.1 (5)
Cu1^{i}—O5—C8—O4	5.8 (4)	C9—C14—C13—C12	0.8 (5)
Cu1^{i}—O5—C8—C9	−174.54 (18)	C21—C20—C15—N1	3.4 (4)
Cu1^{i}—O1—C1—O2	−3.0 (5)	C21—C20—C15—C16	−179.1 (3)
Cu1^{i}—O1—C1—C2	179.5 (2)	C21—C22—C23—C24	−1.1 (5)
Cu1—O4—C8—O5	−5.4 (4)	C15—C16—C17—C18	−0.9 (4)
Cu1—O4—C8—C9	174.92 (18)	C15—C20—C21—N2	1.5 (4)
N2—C21—C22—C23	179.9 (3)	C15—C20—C21—C22	−178.6 (3)
C19—C18—C17—C16	0.5 (4)	C8—C9—C10—C11	−179.5 (3)
C19—C20—C21—N2	−178.6 (2)	C8—C9—C14—C13	178.5 (3)
C19—C20—C21—C22	1.2 (4)	C17—C16—C15—N1	177.5 (2)
C19—C20—C15—N1	−176.4 (2)	C17—C16—C15—C20	0.1 (4)
C19—C20—C15—C16	1.0 (4)	C24—C19—C18—C17	−179.1 (2)
C19—C24—C23—C22	0.8 (4)	C24—C19—C20—C21	−1.5 (4)
C7—C2—C1—O2	167.6 (3)	C24—C19—C20—C15	178.3 (2)
C7—C2—C1—O1	−14.7 (4)	O6—C12—C13—C14	−179.1 (3)
C7—C2—C3—C4	3.7 (5)	O6—C12—C11—C10	178.2 (3)
C7—C6—C5—O3	−175.1 (3)	C10—C9—C8—O5	1.1 (4)
C7—C6—C5—C4	4.1 (5)	C10—C9—C8—O4	−179.2 (3)

C2—C7—C6—C5	−2.3 (5)	C10—C9—C14—C13	−1.5 (5)
C2—C3—C4—C5	−2.0 (5)	C14—C9—C8—O5	−178.9 (3)
C1—C2—C3—C4	−173.8 (3)	C14—C9—C8—O4	0.9 (4)
C18—C19—C20—C21	178.7 (2)	C14—C9—C10—C11	0.5 (5)
C18—C19—C20—C15	−1.4 (4)	C13—C12—C11—C10	−1.8 (6)
C18—C19—C24—C23	−179.7 (2)	C11—C12—C13—C14	0.8 (6)
C6—C7—C2—C1	175.9 (3)	C25—N1—C15—C16	−66.3 (4)
C6—C7—C2—C3	−1.5 (5)	C25—N1—C15—C20	111.1 (3)
C20—C19—C18—C17	0.7 (4)	C26—N1—C15—C16	58.5 (4)
C20—C19—C24—C23	0.6 (4)	C26—N1—C15—C20	−124.1 (3)
C20—C21—C22—C23	0.1 (5)	C28—N2—C21—C20	112.3 (4)
C3—C2—C1—O2	−15.0 (4)	C28—N2—C21—C22	−67.5 (4)
C3—C2—C1—O1	162.7 (3)	C27—N2—C21—C20	−122.3 (3)
C3—C4—C5—O3	177.3 (3)	C27—N2—C21—C22	57.8 (4)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.

catena-Poly[[tetrakis(*μ*-4-hydroxybenzoato)dicopper(II)]-*μ*-(4,4-bipyridine *N*,*N*′-dioxide)] (compound_8)

Crystal data

$[Cu_2(C_7H_5O_3)_4(C_{10}H_8N_2O_2)]\cdot H_2O$
M_r = 881.72
Monoclinic, *C*2/*c*
a = 11.9851 (8) Å
b = 17.7472 (11) Å
c = 17.1294 (10) Å
β = 92.637 (5)°
V = 3639.6 (4) Å^3
Z = 4
F(000) = 1800
D_x = 1.609 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 3614 reflections
θ = 4.5–75.7°
μ = 2.12 mm^{-1}
T = 130 K
Block, dark green
0.32 × 0.06 × 0.05 mm

Data collection

Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.2273 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.898, T_{max} = 1.000
6868 measured reflections
3663 independent reflections
3386 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 75.9°, θ_{min} = 4.5°
h = −13→14
k = −22→19
l = −14→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
S = 1.05
3663 reflections
263 parameters
2 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 1.6515P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.54139 (2)	0.54327 (2)	0.55445 (2)	0.01706 (11)
O1	0.40892 (10)	0.39073 (7)	0.53016 (7)	0.0225 (3)
O2	0.47249 (11)	0.47017 (7)	0.62379 (8)	0.0237 (3)
O3	0.32641 (10)	0.52033 (8)	0.45993 (8)	0.0243 (3)
O4	0.39850 (10)	0.59718 (7)	0.55335 (8)	0.0241 (3)
O5	0.500000	0.68052 (11)	0.750000	0.0259 (4)
H5	0.467094	0.653598	0.782900	0.039*
O6	0.61601 (10)	0.60211 (8)	0.65226 (8)	0.0252 (3)
O7	−0.08298 (11)	0.73890 (8)	0.49096 (9)	0.0279 (3)
H7	−0.073715	0.782010	0.510444	0.042*
O8	0.14206 (12)	0.27908 (9)	0.81889 (8)	0.0315 (3)
H8	0.098782	0.247191	0.797536	0.047*
N1	0.72175 (12)	0.59467 (8)	0.67856 (9)	0.0217 (3)
C1	0.41454 (14)	0.41455 (10)	0.60065 (10)	0.0197 (3)
C2	0.34645 (14)	0.37614 (10)	0.65823 (10)	0.0209 (3)
C12	0.01382 (14)	0.69945 (10)	0.49891 (11)	0.0223 (3)
C8	0.31973 (14)	0.57449 (10)	0.50733 (10)	0.0209 (3)
C9	0.21109 (14)	0.61541 (10)	0.50826 (10)	0.0206 (3)
C7	0.26800 (15)	0.32172 (10)	0.63378 (10)	0.0225 (3)
H7A	0.262365	0.307233	0.580351	0.027*
C6	0.19869 (15)	0.28877 (10)	0.68605 (11)	0.0249 (4)
H6	0.145326	0.252219	0.668495	0.030*
C11	0.10430 (15)	0.72402 (10)	0.54651 (11)	0.0235 (3)
H11	0.098850	0.769221	0.575802	0.028*
C17	0.94118 (15)	0.58594 (10)	0.73482 (11)	0.0236 (4)
C3	0.35321 (15)	0.39701 (11)	0.73707 (11)	0.0248 (4)
H3	0.405288	0.434477	0.754374	0.030*
C13	0.01999 (15)	0.63124 (11)	0.45921 (11)	0.0254 (4)
H13	−0.043056	0.612977	0.429322	0.030*
C10	0.20231 (14)	0.68216 (10)	0.55087 (10)	0.0227 (3)
H10	0.263924	0.699033	0.583112	0.027*
C5	0.20708 (15)	0.30921 (11)	0.76486 (11)	0.0246 (4)
C18	0.85292 (15)	0.57140 (11)	0.78293 (11)	0.0269 (4)
H18	0.867997	0.558339	0.836152	0.032*
C16	0.91425 (15)	0.60349 (11)	0.65689 (11)	0.0269 (4)
H16	0.972354	0.612354	0.622120	0.032*
C14	0.11820 (15)	0.59009 (10)	0.46338 (11)	0.0245 (4)
H14	0.122520	0.544045	0.435389	0.029*
C15	0.80470 (16)	0.60814 (11)	0.62966 (11)	0.0269 (4)

H15	0.787426	0.620798	0.576581	0.032*
C19	0.74381 (15)	0.57585 (11)	0.75379 (11)	0.0263 (4)
H19	0.684199	0.565602	0.786902	0.032*
C4	0.28504 (16)	0.36375 (11)	0.79002 (11)	0.0275 (4)
H4	0.291054	0.377890	0.843540	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.01606 (16)	0.01854 (16)	0.01641 (16)	0.00039 (8)	−0.00081 (10)	0.00009 (8)
O1	0.0262 (6)	0.0211 (6)	0.0203 (6)	−0.0031 (5)	0.0032 (5)	0.0003 (5)
O2	0.0245 (6)	0.0263 (6)	0.0201 (6)	−0.0066 (5)	−0.0015 (5)	0.0028 (5)
O3	0.0196 (6)	0.0263 (6)	0.0266 (7)	0.0057 (5)	−0.0016 (5)	−0.0041 (5)
O4	0.0190 (6)	0.0270 (6)	0.0260 (6)	0.0040 (5)	−0.0015 (5)	−0.0034 (5)
O5	0.0234 (9)	0.0268 (9)	0.0281 (10)	0.000	0.0077 (7)	0.000
O6	0.0160 (6)	0.0326 (7)	0.0266 (7)	0.0023 (5)	−0.0046 (5)	−0.0098 (5)
O7	0.0205 (6)	0.0258 (6)	0.0372 (8)	0.0041 (5)	−0.0025 (5)	−0.0067 (5)
O8	0.0331 (7)	0.0392 (8)	0.0226 (7)	−0.0107 (6)	0.0046 (5)	0.0008 (6)
N1	0.0182 (7)	0.0230 (7)	0.0235 (7)	−0.0011 (5)	−0.0031 (5)	−0.0039 (6)
C1	0.0170 (7)	0.0219 (8)	0.0199 (8)	0.0027 (6)	−0.0008 (6)	0.0016 (6)
C2	0.0184 (8)	0.0238 (8)	0.0203 (8)	0.0009 (6)	0.0003 (6)	0.0003 (6)
C12	0.0182 (8)	0.0240 (8)	0.0248 (9)	0.0015 (6)	0.0023 (6)	0.0025 (6)
C8	0.0189 (8)	0.0225 (8)	0.0213 (8)	0.0016 (6)	0.0018 (6)	0.0051 (6)
C9	0.0187 (8)	0.0223 (8)	0.0208 (8)	0.0012 (6)	0.0014 (6)	0.0027 (6)
C7	0.0228 (8)	0.0250 (8)	0.0199 (8)	−0.0006 (7)	0.0011 (6)	−0.0016 (6)
C6	0.0243 (8)	0.0262 (8)	0.0241 (9)	−0.0031 (7)	0.0001 (7)	−0.0003 (7)
C11	0.0225 (8)	0.0217 (8)	0.0263 (9)	0.0004 (6)	0.0021 (7)	−0.0025 (7)
C17	0.0202 (9)	0.0267 (8)	0.0239 (9)	−0.0012 (6)	−0.0009 (7)	−0.0011 (7)
C3	0.0230 (8)	0.0268 (8)	0.0243 (9)	−0.0044 (7)	−0.0017 (7)	−0.0014 (7)
C13	0.0189 (8)	0.0290 (9)	0.0280 (9)	0.0004 (7)	−0.0023 (6)	−0.0027 (7)
C10	0.0188 (8)	0.0266 (8)	0.0225 (8)	0.0000 (6)	−0.0007 (6)	0.0002 (7)
C5	0.0238 (8)	0.0280 (9)	0.0222 (9)	−0.0009 (7)	0.0025 (7)	0.0021 (7)
C18	0.0235 (9)	0.0364 (10)	0.0208 (9)	0.0019 (7)	−0.0005 (7)	0.0033 (7)
C16	0.0216 (8)	0.0368 (10)	0.0221 (9)	−0.0047 (7)	−0.0002 (7)	0.0018 (7)
C14	0.0224 (8)	0.0245 (8)	0.0264 (9)	0.0016 (6)	0.0007 (7)	−0.0029 (7)
C15	0.0231 (8)	0.0353 (10)	0.0220 (9)	−0.0022 (7)	−0.0007 (7)	0.0017 (7)
C19	0.0228 (8)	0.0312 (9)	0.0250 (9)	−0.0003 (7)	0.0030 (7)	0.0000 (7)
C4	0.0305 (9)	0.0336 (10)	0.0182 (8)	−0.0041 (8)	0.0000 (7)	−0.0025 (7)

Geometric parameters (Å, º)

Cu1—O1^{i}	1.9765 (12)	C9—C14	1.398 (3)
Cu1—O2	1.9663 (13)	C7—H7A	0.9500
Cu1—O3^{i}	1.9701 (12)	C7—C6	1.379 (2)
Cu1—O4	1.9610 (12)	C6—H6	0.9500
Cu1—O6	2.1352 (13)	C6—C5	1.397 (3)
O1—C1	1.278 (2)	C11—H11	0.9500
O2—C1	1.260 (2)	C11—C10	1.389 (3)

O3—C8	1.263 (2)	C17—C17^{iii}	1.480 (4)
O4—C8	1.267 (2)	C17—C18	1.395 (3)
O5—H5^{ii}	0.85 (3)	C17—C16	1.394 (3)
O5—H5	0.8497	C3—H3	0.9500
O6—N1	1.3320 (19)	C3—C4	1.381 (3)
O7—H7	0.8400	C13—H13	0.9500
O7—C12	1.357 (2)	C13—C14	1.384 (3)
O8—H8	0.8400	C10—H10	0.9500
O8—C5	1.348 (2)	C5—C4	1.399 (3)
N1—C15	1.350 (2)	C18—H18	0.9500
N1—C19	1.346 (3)	C18—C19	1.380 (3)
C1—C2	1.476 (2)	C16—H16	0.9500
C2—C7	1.399 (2)	C16—C15	1.376 (3)
C2—C3	1.399 (3)	C14—H14	0.9500
C12—C11	1.396 (3)	C15—H15	0.9500
C12—C13	1.392 (3)	C19—H19	0.9500
C8—C9	1.492 (2)	C4—H4	0.9500
C9—C10	1.398 (2)		

O1^{i}—Cu1—O6	98.98 (5)	C7—C6—H6	120.1
O2—Cu1—O1^{i}	169.44 (5)	C7—C6—C5	119.90 (17)
O2—Cu1—O3^{i}	93.44 (6)	C5—C6—H6	120.1
O2—Cu1—O6	91.21 (5)	C12—C11—H11	120.1
O3^{i}—Cu1—O1^{i}	88.66 (6)	C10—C11—C12	119.77 (17)
O3^{i}—Cu1—O6	93.91 (5)	C10—C11—H11	120.1
O4—Cu1—O1^{i}	89.79 (5)	C18—C17—C17^{iii}	121.9 (2)
O4—Cu1—O2	86.38 (6)	C16—C17—C17^{iii}	120.7 (2)
O4—Cu1—O3^{i}	170.18 (5)	C16—C17—C18	117.30 (17)
O4—Cu1—O6	95.91 (5)	C2—C3—H3	119.6
C1—O1—Cu1^{i}	119.48 (11)	C4—C3—C2	120.74 (17)
C1—O2—Cu1	124.57 (12)	C4—C3—H3	119.6
C8—O3—Cu1^{i}	126.26 (12)	C12—C13—H13	120.1
C8—O4—Cu1	118.44 (11)	C14—C13—C12	119.85 (17)
H5—O5—H5^{ii}	111.6	C14—C13—H13	120.1
N1—O6—Cu1	125.07 (10)	C9—C10—H10	119.7
C12—O7—H7	109.5	C11—C10—C9	120.59 (17)
C5—O8—H8	109.5	C11—C10—H10	119.7
O6—N1—C15	119.26 (16)	O8—C5—C6	122.84 (17)
O6—N1—C19	119.37 (15)	O8—C5—C4	117.51 (17)
C19—N1—C15	121.34 (16)	C6—C5—C4	119.64 (17)
O1—C1—C2	118.16 (15)	C17—C18—H18	119.7
O2—C1—O1	124.09 (16)	C19—C18—C17	120.50 (18)
O2—C1—C2	117.73 (16)	C19—C18—H18	119.7
C7—C2—C1	120.24 (16)	C17—C16—H16	119.6
C7—C2—C3	118.70 (16)	C15—C16—C17	120.89 (17)
C3—C2—C1	120.91 (16)	C15—C16—H16	119.6
O7—C12—C11	122.35 (16)	C9—C14—H14	119.6
O7—C12—C13	117.66 (16)	C13—C14—C9	120.83 (17)

C13—C12—C11	119.97 (16)	C13—C14—H14	119.6
O3—C8—O4	125.04 (16)	N1—C15—C16	119.83 (18)
O3—C8—C9	117.29 (16)	N1—C15—H15	120.1
O4—C8—C9	117.67 (16)	C16—C15—H15	120.1
C10—C9—C8	120.32 (16)	N1—C19—C18	120.10 (17)
C14—C9—C8	120.74 (16)	N1—C19—H19	119.9
C14—C9—C10	118.85 (16)	C18—C19—H19	119.9
C2—C7—H7A	119.5	C3—C4—C5	120.01 (17)
C6—C7—C2	121.00 (17)	C3—C4—H4	120.0
C6—C7—H7A	119.5	C5—C4—H4	120.0

Cu1^{i}—O1—C1—O2	−17.0 (2)	C1—C2—C3—C4	176.48 (17)
Cu1^{i}—O1—C1—C2	161.12 (12)	C2—C7—C6—C5	−0.6 (3)
Cu1—O2—C1—O1	12.8 (2)	C2—C3—C4—C5	−0.8 (3)
Cu1—O2—C1—C2	−165.33 (11)	C12—C11—C10—C9	−0.3 (3)
Cu1^{i}—O3—C8—O4	0.6 (3)	C12—C13—C14—C9	1.2 (3)
Cu1^{i}—O3—C8—C9	179.74 (11)	C8—C9—C10—C11	174.30 (16)
Cu1—O4—C8—O3	−1.9 (2)	C8—C9—C14—C13	−174.76 (17)
Cu1—O4—C8—C9	179.02 (11)	C7—C2—C3—C4	1.0 (3)
Cu1—O6—N1—C15	57.1 (2)	C7—C6—C5—O8	−179.88 (17)
Cu1—O6—N1—C19	−124.59 (15)	C7—C6—C5—C4	0.8 (3)
O1—C1—C2—C7	−7.9 (2)	C6—C5—C4—C3	0.0 (3)
O1—C1—C2—C3	176.70 (16)	C11—C12—C13—C14	−3.8 (3)
O2—C1—C2—C7	170.33 (16)	C17^{iii}—C17—C18—C19	176.46 (14)
O2—C1—C2—C3	−5.1 (2)	C17^{iii}—C17—C16—C15	−175.92 (15)
O3—C8—C9—C10	−170.92 (16)	C17—C18—C19—N1	−0.3 (3)
O3—C8—C9—C14	5.6 (2)	C17—C16—C15—N1	−0.8 (3)
O4—C8—C9—C10	8.3 (2)	C3—C2—C7—C6	−0.3 (3)
O4—C8—C9—C14	−175.21 (16)	C13—C12—C11—C10	3.3 (3)
O6—N1—C15—C16	177.51 (17)	C10—C9—C14—C13	1.8 (3)
O6—N1—C19—C18	−176.93 (17)	C18—C17—C16—C15	1.7 (3)
O7—C12—C11—C10	−178.44 (16)	C16—C17—C18—C19	−1.1 (3)
O7—C12—C13—C14	177.89 (17)	C14—C9—C10—C11	−2.3 (3)
O8—C5—C4—C3	−179.45 (18)	C15—N1—C19—C18	1.3 (3)
C1—C2—C7—C6	−175.80 (16)	C19—N1—C15—C16	−0.7 (3)

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

Tetrakis(*μ*-4-hydroxybenzoato)bis[(4,4′-bipyridine *N*,*N*′-dioxide)copper(II)] dioxane disolvate (compound_9)

Crystal data

$[Cu_2(C_7H_5O_3)_4(C_{10}H_8N_2O_2)_2]\cdot 2C_4H_8O_2$
M_r = 1228.09
Orthorhombic, *Pbca*
a = 17.5993 (3) Å
b = 16.4583 (2) Å
c = 19.7389 (2) Å
V = 5717.46 (13) Å^3
Z = 4
F(000) = 2536

D_x = 1.427 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 10015 reflections
θ = 4.3–76.7°
μ = 1.59 mm^{-1}
T = 100 K
Irregular, dark green
0.09 × 0.07 × 0.05 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.344, T_{max} = 1.000
23256 measured reflections
5876 independent reflections
4731 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 77.9°, θ_{min} = 4.3°
h = −21→18
k = −20→16
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 1.06
5876 reflections
427 parameters
110 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 2.8715P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.55547 (2)	0.53111 (2)	0.53308 (2)	0.01823 (12)	
O5	0.50248 (10)	0.36700 (10)	0.46401 (8)	0.0252 (4)	
O7	0.65573 (9)	0.57946 (10)	0.57670 (7)	0.0241 (3)	
O1	0.59137 (11)	0.56075 (11)	0.44259 (8)	0.0291 (4)	
O8	0.61763 (11)	0.68383 (10)	1.05831 (8)	0.0273 (4)	
O2	0.49637 (11)	0.50447 (11)	0.38364 (8)	0.0280 (4)	
O4	0.59873 (10)	0.42265 (10)	0.52209 (9)	0.0298 (4)	
O3	0.71353 (11)	0.63150 (11)	0.14972 (8)	0.0321 (4)	
H3	0.683225	0.640715	0.117760	0.048*	
O6	0.72255 (11)	0.06580 (10)	0.49628 (10)	0.0354 (4)	
H6	0.761058	0.068772	0.521300	0.053*	
N1	0.65256 (11)	0.59407 (11)	0.64321 (9)	0.0206 (4)	
N2	0.62549 (12)	0.67097 (11)	0.99246 (9)	0.0230 (4)	
O11	0.3089 (3)	0.7105 (2)	0.7347 (2)	0.0404 (8)	0.810 (7)
C8	0.56697 (13)	0.36401 (13)	0.49230 (11)	0.0211 (4)	
C9	0.60821 (14)	0.28525 (14)	0.49182 (11)	0.0226 (5)	
C2	0.59825 (14)	0.56527 (13)	0.32415 (11)	0.0234 (5)	
C1	0.55878 (14)	0.54118 (13)	0.38817 (11)	0.0220 (5)	
C15	0.63041 (15)	0.66824 (14)	0.66426 (11)	0.0252 (5)	
H15	0.617842	0.709079	0.632146	0.030*	

C17	0.64367 (14)	0.62556 (13)	0.78060 (11)	0.0227 (5)	
C10	0.68067 (14)	0.28099 (14)	0.52013 (12)	0.0244 (5)	
H10	0.703137	0.328469	0.538773	0.029*	
C16	0.62607 (15)	0.68455 (14)	0.73252 (11)	0.0251 (5)	
H16	0.610782	0.737066	0.747134	0.030*	
C21	0.60508 (14)	0.71260 (14)	0.87942 (11)	0.0245 (5)	
H21	0.585371	0.751688	0.848675	0.029*	
C20	0.63854 (14)	0.64219 (13)	0.85414 (11)	0.0226 (5)	
C5	0.67379 (15)	0.60924 (14)	0.20579 (11)	0.0258 (5)	
C3	0.67212 (14)	0.59578 (14)	0.32695 (11)	0.0237 (5)	
H3A	0.696648	0.601726	0.369532	0.028*	
C12	0.68680 (15)	0.13871 (14)	0.49508 (13)	0.0266 (5)	
C23	0.65919 (16)	0.60197 (15)	0.97036 (12)	0.0281 (5)	
H23	0.677647	0.563450	1.002205	0.034*	
O9	0.45652 (16)	0.6662 (2)	0.77770 (13)	0.0493 (10)	0.810 (7)
C11	0.72015 (14)	0.20845 (14)	0.52137 (11)	0.0245 (5)	
H11	0.769830	0.206259	0.540094	0.029*	
C4	0.71015 (14)	0.61755 (14)	0.26802 (12)	0.0255 (5)	
H4	0.760583	0.637948	0.270175	0.031*	
C22	0.59993 (14)	0.72685 (14)	0.94821 (11)	0.0241 (5)	
H22	0.578336	0.776103	0.964300	0.029*	
C7	0.56233 (15)	0.55780 (16)	0.26172 (12)	0.0297 (5)	
H7	0.511831	0.537584	0.259521	0.036*	
C19	0.66950 (17)	0.53507 (14)	0.68720 (12)	0.0294 (5)	
H19	0.684302	0.483013	0.671134	0.035*	
C24	0.66691 (16)	0.58720 (14)	0.90203 (12)	0.0278 (5)	
H24	0.691776	0.539147	0.887204	0.033*	
C14	0.57563 (15)	0.21559 (15)	0.46452 (12)	0.0265 (5)	
H14	0.526630	0.218244	0.444465	0.032*	
C18	0.66551 (17)	0.54966 (15)	0.75576 (12)	0.0311 (6)	
H18	0.677809	0.507415	0.786663	0.037*	
C6	0.59989 (16)	0.57974 (16)	0.20242 (12)	0.0313 (5)	
H6A	0.575141	0.574575	0.159865	0.038*	
C13	0.61441 (16)	0.14216 (15)	0.46646 (14)	0.0321 (6)	
H13	0.591747	0.094479	0.448363	0.039*	
C25	0.3282 (2)	0.7026 (3)	0.80400 (19)	0.0388 (9)	0.810 (7)
H25A	0.315819	0.646817	0.819254	0.047*	0.810 (7)
H25B	0.297075	0.740911	0.830851	0.047*	0.810 (7)
C26	0.4093 (3)	0.7187 (3)	0.8173 (2)	0.0438 (11)	0.810 (7)
H26A	0.421075	0.775979	0.806207	0.053*	0.810 (7)
H26B	0.420166	0.710148	0.866016	0.053*	0.810 (7)
C28	0.3552 (2)	0.6565 (3)	0.6955 (2)	0.0434 (10)	0.810 (7)
H28A	0.343313	0.663150	0.646780	0.052*	0.810 (7)
H28B	0.343961	0.599572	0.708289	0.052*	0.810 (7)
C27	0.4373 (2)	0.6736 (3)	0.7071 (2)	0.0509 (12)	0.810 (7)
H27A	0.468400	0.635153	0.680357	0.061*	0.810 (7)
H27B	0.449168	0.729354	0.691464	0.061*	0.810 (7)
O10	0.4610 (8)	0.7313 (12)	0.7713 (7)	0.064 (4)	0.190 (7)

C29	0.405 (2)	0.753 (2)	0.8167 (18)	0.071 (5)	0.190 (7)
H29A	0.380285	0.804734	0.803985	0.085*	0.190 (7)
H29B	0.424871	0.756919	0.863503	0.085*	0.190 (7)
C32	0.4311 (12)	0.7477 (15)	0.7046 (9)	0.059 (4)	0.190 (7)
H32A	0.408561	0.802772	0.703074	0.071*	0.190 (7)
H32B	0.472222	0.744782	0.670468	0.071*	0.190 (7)
C30	0.341 (2)	0.666 (2)	0.8067 (17)	0.085 (5)	0.190 (7)
H30A	0.367741	0.613827	0.808990	0.101*	0.190 (7)
H30B	0.298857	0.667275	0.840290	0.101*	0.190 (7)
C31	0.3752 (19)	0.689 (2)	0.6905 (16)	0.076 (5)	0.190 (7)
H31A	0.399871	0.634867	0.690215	0.091*	0.190 (7)
H31B	0.355471	0.698959	0.644293	0.091*	0.190 (7)
O12	0.313 (2)	0.6859 (18)	0.7354 (18)	0.087 (6)	0.190 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02456 (19)	0.01616 (18)	0.01396 (18)	−0.00172 (12)	0.00016 (12)	0.00019 (11)
O5	0.0280 (9)	0.0203 (8)	0.0273 (8)	0.0012 (7)	−0.0046 (7)	−0.0030 (6)
O7	0.0285 (8)	0.0306 (8)	0.0131 (7)	−0.0072 (7)	0.0008 (6)	−0.0039 (6)
O1	0.0347 (10)	0.0364 (9)	0.0161 (8)	−0.0099 (8)	0.0034 (7)	0.0002 (7)
O8	0.0405 (10)	0.0279 (8)	0.0137 (7)	0.0021 (7)	0.0000 (7)	−0.0025 (6)
O2	0.0354 (10)	0.0334 (9)	0.0151 (7)	−0.0093 (8)	0.0011 (7)	0.0020 (6)
O4	0.0308 (9)	0.0206 (8)	0.0380 (10)	0.0002 (7)	−0.0082 (8)	−0.0057 (7)
O3	0.0359 (10)	0.0420 (10)	0.0183 (8)	0.0017 (8)	0.0057 (7)	0.0073 (7)
O6	0.0327 (10)	0.0212 (8)	0.0524 (12)	0.0044 (7)	−0.0152 (9)	−0.0072 (8)
N1	0.0252 (9)	0.0233 (9)	0.0132 (8)	−0.0024 (8)	−0.0019 (7)	−0.0024 (7)
N2	0.0334 (11)	0.0211 (9)	0.0146 (9)	0.0000 (8)	−0.0009 (8)	−0.0029 (7)
O11	0.0389 (15)	0.049 (2)	0.0337 (14)	0.0094 (15)	0.0003 (11)	−0.0053 (15)
C8	0.0264 (11)	0.0194 (10)	0.0175 (10)	−0.0011 (9)	0.0010 (8)	0.0016 (8)
C9	0.0260 (11)	0.0232 (11)	0.0187 (10)	−0.0007 (9)	0.0005 (9)	0.0002 (8)
C2	0.0336 (13)	0.0187 (10)	0.0180 (10)	0.0003 (9)	0.0032 (9)	0.0016 (8)
C1	0.0299 (12)	0.0197 (10)	0.0165 (10)	0.0009 (9)	0.0010 (9)	0.0006 (8)
C15	0.0343 (13)	0.0218 (11)	0.0195 (11)	−0.0008 (10)	−0.0017 (9)	0.0029 (9)
C17	0.0299 (12)	0.0197 (10)	0.0184 (10)	0.0005 (9)	−0.0021 (9)	−0.0009 (8)
C10	0.0302 (12)	0.0180 (10)	0.0251 (11)	0.0000 (9)	−0.0019 (9)	−0.0003 (9)
C16	0.0366 (13)	0.0192 (10)	0.0196 (11)	0.0026 (9)	0.0009 (9)	−0.0014 (8)
C21	0.0348 (13)	0.0203 (10)	0.0184 (10)	0.0047 (9)	−0.0035 (9)	0.0014 (8)
C20	0.0317 (12)	0.0195 (10)	0.0165 (10)	−0.0004 (9)	−0.0020 (9)	−0.0012 (8)
C5	0.0338 (12)	0.0257 (11)	0.0180 (11)	0.0022 (10)	0.0073 (9)	0.0036 (9)
C3	0.0315 (12)	0.0224 (11)	0.0172 (10)	0.0026 (10)	0.0020 (9)	0.0015 (8)
C12	0.0294 (12)	0.0198 (11)	0.0305 (12)	0.0025 (9)	−0.0037 (10)	−0.0025 (9)
C23	0.0440 (15)	0.0215 (11)	0.0186 (11)	0.0065 (11)	−0.0039 (10)	0.0010 (9)
O9	0.0365 (15)	0.077 (2)	0.0349 (14)	0.0123 (14)	−0.0016 (11)	−0.0141 (14)
C11	0.0272 (12)	0.0227 (11)	0.0236 (11)	−0.0014 (9)	−0.0032 (9)	−0.0003 (9)
C4	0.0276 (12)	0.0262 (11)	0.0225 (11)	0.0026 (10)	0.0023 (9)	0.0031 (9)
C22	0.0325 (13)	0.0184 (10)	0.0214 (10)	0.0054 (9)	0.0004 (9)	−0.0004 (9)
C7	0.0336 (13)	0.0351 (13)	0.0205 (12)	−0.0066 (11)	0.0006 (10)	0.0024 (10)

C19	0.0449 (15)	0.0229 (11)	0.0203 (11)	0.0068 (11)	−0.0021 (10)	−0.0033 (9)
C24	0.0430 (14)	0.0194 (11)	0.0211 (11)	0.0070 (10)	−0.0027 (10)	−0.0025 (9)
C14	0.0277 (12)	0.0227 (12)	0.0292 (12)	0.0019 (10)	−0.0045 (9)	−0.0036 (9)
C18	0.0490 (16)	0.0240 (11)	0.0204 (11)	0.0091 (11)	−0.0052 (11)	−0.0018 (9)
C6	0.0396 (14)	0.0370 (13)	0.0172 (11)	−0.0034 (11)	0.0007 (10)	0.0032 (10)
C13	0.0336 (14)	0.0231 (12)	0.0396 (14)	−0.0009 (10)	−0.0079 (11)	−0.0062 (10)
C25	0.039 (2)	0.050 (2)	0.0271 (17)	0.0065 (18)	0.0047 (14)	−0.0004 (17)
C26	0.043 (2)	0.057 (3)	0.0308 (19)	−0.001 (2)	0.0031 (16)	−0.011 (2)
C28	0.035 (2)	0.058 (3)	0.037 (2)	0.0044 (19)	0.0039 (17)	−0.013 (2)
C27	0.041 (2)	0.076 (3)	0.035 (2)	0.001 (2)	0.0008 (16)	−0.021 (2)
O10	0.053 (5)	0.092 (7)	0.046 (5)	0.003 (5)	0.001 (4)	0.003 (5)
C29	0.063 (7)	0.094 (8)	0.057 (7)	−0.002 (6)	−0.001 (6)	−0.001 (6)
C32	0.050 (6)	0.086 (7)	0.041 (6)	−0.003 (6)	0.002 (5)	0.006 (5)
C30	0.079 (8)	0.096 (8)	0.078 (7)	−0.004 (6)	−0.001 (6)	0.000 (6)
C31	0.072 (7)	0.092 (8)	0.064 (7)	−0.003 (6)	0.006 (6)	−0.001 (6)
O12	0.082 (8)	0.096 (8)	0.081 (7)	−0.005 (6)	0.001 (6)	−0.003 (6)

Geometric parameters (Å, º)

Cu1—O5^{i}	1.9637 (17)	C12—C11	1.390 (3)
Cu1—O7	2.1185 (16)	C12—C13	1.395 (4)
Cu1—O1	1.9564 (16)	C23—H23	0.9500
Cu1—O2^{i}	1.9692 (16)	C23—C24	1.377 (3)
Cu1—O4	1.9527 (17)	O9—C26	1.431 (5)
O5—C8	1.266 (3)	O9—C27	1.439 (5)
O7—N1	1.336 (2)	C11—H11	0.9500
O1—C1	1.260 (3)	C4—H4	0.9500
O8—N2	1.324 (2)	C22—H22	0.9500
O2—C1	1.257 (3)	C7—H7	0.9500
O4—C8	1.261 (3)	C7—C6	1.392 (3)
O3—H3	0.8400	C19—H19	0.9500
O3—C5	1.360 (3)	C19—C18	1.376 (3)
O6—H6	0.8400	C24—H24	0.9500
O6—C12	1.355 (3)	C14—H14	0.9500
N1—C15	1.347 (3)	C14—C13	1.388 (4)
N1—C19	1.336 (3)	C18—H18	0.9500
N2—C23	1.353 (3)	C6—H6A	0.9500
N2—C22	1.346 (3)	C13—H13	0.9500
O11—C25	1.416 (6)	C25—H25A	0.9900
O11—C28	1.433 (6)	C25—H25B	0.9900
C8—C9	1.486 (3)	C25—C26	1.475 (7)
C9—C10	1.394 (3)	C26—H26A	0.9900
C9—C14	1.390 (3)	C26—H26B	0.9900
C2—C1	1.496 (3)	C28—H28A	0.9900
C2—C3	1.395 (4)	C28—H28B	0.9900
C2—C7	1.390 (3)	C28—C27	1.489 (6)
C15—H15	0.9500	C27—H27A	0.9900
C15—C16	1.376 (3)	C27—H27B	0.9900

C17—C16	1.392 (3)	O10—C29	1.38 (4)
C17—C20	1.480 (3)	O10—C32	1.44 (2)
C17—C18	1.396 (3)	C29—H29A	0.9900
C10—H10	0.9500	C29—H29B	0.9900
C10—C11	1.381 (3)	C29—C30	1.83 (5)
C16—H16	0.9500	C32—H32A	0.9900
C21—H21	0.9500	C32—H32B	0.9900
C21—C20	1.392 (3)	C32—C31	1.41 (4)
C21—C22	1.381 (3)	C30—H30A	0.9900
C20—C24	1.401 (3)	C30—H30B	0.9900
C5—C4	1.392 (3)	C30—O12	1.52 (4)
C5—C6	1.390 (4)	C31—H31A	0.9900
C3—H3A	0.9500	C31—H31B	0.9900
C3—C4	1.389 (3)	C31—O12	1.41 (5)

O5^{i}—Cu1—O7	95.73 (7)	N2—C22—H22	120.0
O5^{i}—Cu1—O2^{i}	89.36 (7)	C21—C22—H22	120.0
O1—Cu1—O5^{i}	88.94 (7)	C2—C7—H7	119.8
O1—Cu1—O7	90.49 (7)	C2—C7—C6	120.4 (2)
O1—Cu1—O2^{i}	170.38 (7)	C6—C7—H7	119.8
O2^{i}—Cu1—O7	99.10 (7)	N1—C19—H19	120.0
O4—Cu1—O5^{i}	170.56 (7)	N1—C19—C18	120.1 (2)
O4—Cu1—O7	93.66 (7)	C18—C19—H19	120.0
O4—Cu1—O1	90.04 (8)	C20—C24—H24	119.6
O4—Cu1—O2^{i}	90.09 (8)	C23—C24—C20	120.8 (2)
C8—O5—Cu1^{i}	119.08 (15)	C23—C24—H24	119.6
N1—O7—Cu1	115.61 (13)	C9—C14—H14	119.9
C1—O1—Cu1	124.60 (16)	C13—C14—C9	120.3 (2)
C1—O2—Cu1^{i}	119.24 (15)	C13—C14—H14	119.9
C8—O4—Cu1	125.36 (16)	C17—C18—H18	119.5
C5—O3—H3	109.5	C19—C18—C17	121.0 (2)
C12—O6—H6	109.5	C19—C18—H18	119.5
O7—N1—C15	118.58 (18)	C5—C6—C7	119.7 (2)
O7—N1—C19	119.90 (19)	C5—C6—H6A	120.2
C19—N1—C15	121.50 (19)	C7—C6—H6A	120.2
O8—N2—C23	119.76 (19)	C12—C13—H13	120.1
O8—N2—C22	119.54 (19)	C14—C13—C12	119.7 (2)
C22—N2—C23	120.71 (19)	C14—C13—H13	120.1
C25—O11—C28	109.2 (4)	O11—C25—H25A	109.0
O5—C8—C9	118.0 (2)	O11—C25—H25B	109.0
O4—C8—O5	125.0 (2)	O11—C25—C26	112.8 (4)
O4—C8—C9	117.0 (2)	H25A—C25—H25B	107.8
C10—C9—C8	119.2 (2)	C26—C25—H25A	109.0
C14—C9—C8	121.4 (2)	C26—C25—H25B	109.0
C14—C9—C10	119.4 (2)	O9—C26—C25	110.8 (4)
C3—C2—C1	119.7 (2)	O9—C26—H26A	109.5
C7—C2—C1	120.9 (2)	O9—C26—H26B	109.5
C7—C2—C3	119.4 (2)	C25—C26—H26A	109.5

O1—C1—C2	116.2 (2)	C25—C26—H26B	109.5
O2—C1—O1	125.6 (2)	H26A—C26—H26B	108.1
O2—C1—C2	118.2 (2)	O11—C28—H28A	109.5
N1—C15—H15	120.2	O11—C28—H28B	109.5
N1—C15—C16	119.7 (2)	O11—C28—C27	110.5 (4)
C16—C15—H15	120.2	H28A—C28—H28B	108.1
C16—C17—C20	121.7 (2)	C27—C28—H28A	109.5
C16—C17—C18	116.5 (2)	C27—C28—H28B	109.5
C18—C17—C20	121.8 (2)	O9—C27—C28	111.2 (4)
C9—C10—H10	119.6	O9—C27—H27A	109.4
C11—C10—C9	120.7 (2)	O9—C27—H27B	109.4
C11—C10—H10	119.6	C28—C27—H27A	109.4
C15—C16—C17	121.3 (2)	C28—C27—H27B	109.4
C15—C16—H16	119.4	H27A—C27—H27B	108.0
C17—C16—H16	119.4	C29—O10—C32	106 (2)
C20—C21—H21	119.3	O10—C29—H29A	111.8
C22—C21—H21	119.3	O10—C29—H29B	111.8
C22—C21—C20	121.4 (2)	O10—C29—C30	100 (2)
C21—C20—C17	122.1 (2)	H29A—C29—H29B	109.5
C21—C20—C24	116.5 (2)	C30—C29—H29A	111.8
C24—C20—C17	121.4 (2)	C30—C29—H29B	111.8
O3—C5—C4	117.1 (2)	O10—C32—H32A	110.1
O3—C5—C6	122.5 (2)	O10—C32—H32B	110.1
C6—C5—C4	120.5 (2)	H32A—C32—H32B	108.4
C2—C3—H3A	119.7	C31—C32—O10	108 (2)
C4—C3—C2	120.6 (2)	C31—C32—H32A	110.1
C4—C3—H3A	119.7	C31—C32—H32B	110.1
O6—C12—C11	121.9 (2)	C29—C30—H30A	112.2
O6—C12—C13	117.9 (2)	C29—C30—H30B	112.2
C11—C12—C13	120.2 (2)	H30A—C30—H30B	109.8
N2—C23—H23	119.8	O12—C30—C29	98 (2)
N2—C23—C24	120.5 (2)	O12—C30—H30A	112.2
C24—C23—H23	119.8	O12—C30—H30B	112.2
C26—O9—C27	109.9 (3)	C32—C31—H31A	108.2
C10—C11—C12	119.7 (2)	C32—C31—H31B	108.2
C10—C11—H11	120.2	C32—C31—O12	116 (3)
C12—C11—H11	120.2	H31A—C31—H31B	107.4
C5—C4—H4	120.3	O12—C31—H31A	108.2
C3—C4—C5	119.5 (2)	O12—C31—H31B	108.2
C3—C4—H4	120.3	C31—O12—C30	110 (3)
N2—C22—C21	120.0 (2)		

Cu1^{i}—O5—C8—O4	−1.2 (3)	C10—C9—C14—C13	1.1 (4)
Cu1^{i}—O5—C8—C9	177.52 (15)	C16—C17—C20—C21	10.1 (4)
Cu1—O7—N1—C15	−91.3 (2)	C16—C17—C20—C24	−170.3 (2)
Cu1—O7—N1—C19	87.2 (2)	C16—C17—C18—C19	0.3 (4)
Cu1—O1—C1—O2	4.7 (3)	C21—C20—C24—C23	2.1 (4)
Cu1—O1—C1—C2	−176.11 (15)	C20—C17—C16—C15	−179.4 (2)

Cu1^{i}—O2—C1—O1	−2.8 (3)	C20—C17—C18—C19	179.6 (3)
Cu1^{i}—O2—C1—C2	178.11 (15)	C20—C21—C22—N2	−2.0 (4)
Cu1—O4—C8—O5	1.2 (3)	C3—C2—C1—O1	9.7 (3)
Cu1—O4—C8—C9	−177.61 (15)	C3—C2—C1—O2	−171.1 (2)
O5—C8—C9—C10	177.4 (2)	C3—C2—C7—C6	0.6 (4)
O5—C8—C9—C14	−3.5 (3)	C23—N2—C22—C21	2.7 (4)
O7—N1—C15—C16	179.5 (2)	C11—C12—C13—C14	−0.2 (4)
O7—N1—C19—C18	−179.3 (2)	C4—C5—C6—C7	−0.5 (4)
O8—N2—C23—C24	179.2 (2)	C22—N2—C23—C24	−1.0 (4)
O8—N2—C22—C21	−177.4 (2)	C22—C21—C20—C17	179.2 (2)
O4—C8—C9—C10	−3.8 (3)	C22—C21—C20—C24	−0.4 (4)
O4—C8—C9—C14	175.4 (2)	C7—C2—C1—O1	−170.3 (2)
O3—C5—C4—C3	179.9 (2)	C7—C2—C1—O2	8.9 (3)
O3—C5—C6—C7	179.8 (2)	C7—C2—C3—C4	−0.9 (3)
O6—C12—C11—C10	−178.7 (2)	C19—N1—C15—C16	1.0 (4)
O6—C12—C13—C14	179.7 (2)	C14—C9—C10—C11	−0.1 (4)
N1—C15—C16—C17	−0.5 (4)	C18—C17—C16—C15	−0.2 (4)
N1—C19—C18—C17	0.2 (4)	C18—C17—C20—C21	−169.2 (3)
N2—C23—C24—C20	−1.5 (4)	C18—C17—C20—C24	10.4 (4)
O11—C25—C26—O9	−56.8 (5)	C6—C5—C4—C3	0.3 (4)
O11—C28—C27—O9	58.0 (6)	C13—C12—C11—C10	1.2 (4)
C8—C9—C10—C11	179.1 (2)	C25—O11—C28—C27	−57.2 (5)
C8—C9—C14—C13	−178.1 (2)	C26—O9—C27—C28	−56.3 (5)
C9—C10—C11—C12	−1.0 (4)	C28—O11—C25—C26	57.3 (5)
C9—C14—C13—C12	−0.9 (4)	C27—O9—C26—C25	54.9 (5)
C2—C3—C4—C5	0.4 (3)	O10—C29—C30—O12	−69 (3)
C2—C7—C6—C5	0.0 (4)	O10—C32—C31—O12	60 (4)
C1—C2—C3—C4	179.2 (2)	C29—O10—C32—C31	−72 (3)
C1—C2—C7—C6	−179.4 (2)	C29—C30—O12—C31	59 (3)
C15—N1—C19—C18	−0.9 (4)	C32—O10—C29—C30	76 (2)
C17—C20—C24—C23	−177.5 (2)	C32—C31—O12—C30	−61 (4)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

(*μ*-Acetato)(dioxane)tris(*μ*-4-hydroxybenzoato)dicopper(II) dioxane 3.5-solvate (compound_10)

Crystal data

$[Cu_2(C_7H_5O_3)_3(C_2H_3O_2)(C_4H_8O_2)]\cdot 3.5C_4H_8O_2$
M_r = 993.92
Monoclinic, *I*2/*a*
a = 19.4801 (2) Å
b = 13.0774 (2) Å
c = 34.5443 (5) Å
β = 97.702 (1)°
V = 8720.7 (2) Å^3
Z = 8

F(000) = 4144
D_x = 1.514 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 12029 reflections
θ = 2.6–77.0°
μ = 1.90 mm^{-1}
T = 100 K
Irregular, clear light blue
0.26 × 0.09 × 0.06 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.764, T_{max} = 1.000
37963 measured reflections
9032 independent reflections
6906 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 78.1°, θ_{min} = 4.1°
h = −24→24
k = −16→16
l = −28→43

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.191
S = 1.09
9032 reflections
436 parameters
16 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0941P)^2 + 18.8122P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.12 e Å^{-3}
$\Delta\rho_{min}$ = −1.24 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cu2	0.44041 (2)	0.51623 (4)	0.35763 (2)	0.02947 (15)
Cu1	0.30843 (2)	0.50941 (4)	0.35715 (2)	0.02987 (15)
O13	0.05315 (11)	0.4778 (2)	0.35721 (7)	0.0337 (5)
O2	0.30466 (11)	0.5698 (2)	0.30561 (7)	0.0392 (6)
O8	0.44644 (12)	0.4646 (2)	0.41110 (7)	0.0407 (6)
O1	0.41904 (11)	0.5676 (2)	0.30416 (7)	0.0391 (6)
O10	0.43263 (12)	0.6561 (2)	0.37857 (8)	0.0404 (6)
O12	0.19732 (11)	0.5053 (2)	0.35894 (7)	0.0369 (6)
O5	0.31312 (12)	0.3742 (2)	0.33394 (7)	0.0393 (6)
O4	0.42855 (12)	0.3756 (2)	0.33901 (8)	0.0414 (6)
O7	0.33150 (11)	0.4503 (2)	0.40920 (7)	0.0406 (6)
O11	0.31846 (12)	0.6492 (2)	0.37953 (8)	0.0431 (6)
O6	0.37646 (14)	−0.0615 (2)	0.26699 (9)	0.0566 (8)
H6	0.411533	−0.095216	0.273652	0.085*
O14	0.49812 (14)	0.3538 (2)	0.21595 (10)	0.0573 (8)
O3	0.30985 (16)	0.7474 (3)	0.13673 (9)	0.0608 (9)
H3	0.269905	0.772437	0.131840	0.091*
C9	0.37065 (16)	0.2276 (3)	0.31508 (10)	0.0347 (7)
C1	0.35749 (16)	0.5857 (3)	0.28908 (10)	0.0334 (7)
C8	0.37070 (16)	0.3329 (3)	0.33044 (9)	0.0334 (7)
C2	0.34527 (17)	0.6291 (3)	0.24884 (10)	0.0366 (7)

C15	0.39327 (16)	0.4382 (3)	0.42566 (10)	0.0336 (7)
C22	0.37622 (19)	0.6950 (3)	0.38545 (11)	0.0398 (8)
C16	0.40368 (19)	0.3911 (3)	0.46479 (11)	0.0411 (8)
O15	0.3630 (2)	0.3028 (4)	0.18432 (12)	0.0898 (13)
C3	0.27752 (19)	0.6499 (3)	0.23152 (11)	0.0427 (9)
H3A	0.239946	0.636583	0.245707	0.051*
C7	0.3989 (2)	0.6463 (4)	0.22722 (13)	0.0539 (11)
H7	0.445139	0.631333	0.238242	0.065*
C12	0.37601 (19)	0.0322 (3)	0.28385 (12)	0.0445 (9)
C14	0.43073 (18)	0.1694 (3)	0.32141 (12)	0.0449 (9)
H14	0.470813	0.197199	0.336437	0.054*
C10	0.3124 (2)	0.1862 (4)	0.29364 (13)	0.0512 (10)
H10	0.270403	0.223955	0.289909	0.061*
C4	0.2644 (2)	0.6894 (3)	0.19413 (12)	0.0473 (9)
H4	0.218118	0.703503	0.182926	0.057*
C26	0.08145 (18)	0.5156 (4)	0.32423 (12)	0.0515 (11)
H26A	0.081698	0.591269	0.324855	0.062*
H26B	0.051977	0.493612	0.300078	0.062*
C27	0.15447 (18)	0.4770 (4)	0.32362 (12)	0.0534 (12)
H27A	0.153967	0.401680	0.320873	0.064*
H27B	0.173591	0.506501	0.300920	0.064*
C13	0.43340 (18)	0.0727 (3)	0.30644 (12)	0.0444 (9)
H13	0.474695	0.033561	0.311605	0.053*
C11	0.3151 (2)	0.0892 (4)	0.27750 (14)	0.0577 (12)
H11	0.275376	0.061757	0.262081	0.069*
O9	0.4332 (3)	0.2645 (6)	0.57554 (15)	0.158 (3)
H9	0.475453	0.265261	0.584575	0.237*
C5	0.3185 (2)	0.7083 (4)	0.17307 (12)	0.0493 (10)
C17	0.3487 (2)	0.3545 (4)	0.48191 (13)	0.0581 (12)
H17	0.302917	0.359420	0.468479	0.070*
C25	0.09698 (19)	0.5089 (5)	0.39178 (12)	0.0679 (16)
H25A	0.077723	0.483734	0.415122	0.081*
H25B	0.098910	0.584502	0.393076	0.081*
C6	0.3860 (2)	0.6851 (4)	0.18967 (13)	0.0611 (13)
H6A	0.423384	0.695983	0.175093	0.073*
C24	0.16922 (19)	0.4665 (5)	0.39156 (12)	0.0666 (15)
H24A	0.198972	0.486336	0.415911	0.080*
H24B	0.167409	0.390914	0.390270	0.080*
C28	0.4463 (3)	0.3372 (6)	0.23929 (15)	0.0812 (18)
H28A	0.454840	0.381743	0.262597	0.097*
H28B	0.448857	0.265315	0.248406	0.097*
C18	0.3600 (3)	0.3100 (5)	0.51906 (16)	0.0782 (17)
H18	0.322004	0.283295	0.530427	0.094*
C21	0.4693 (3)	0.3848 (5)	0.48548 (13)	0.0758 (17)
H21	0.507538	0.410986	0.474207	0.091*
C23	0.3785 (3)	0.8010 (4)	0.40150 (16)	0.0635 (12)
H23A	0.426723	0.820561	0.409991	0.095*
H23B	0.352366	0.803686	0.423833	0.095*

H23C	0.357871	0.848395	0.381235	0.095*
C29	0.3787 (3)	0.3565 (5)	0.21985 (17)	0.0744 (15)
H29A	0.344515	0.337519	0.237407	0.089*
H29B	0.373770	0.430747	0.214539	0.089*
C19	0.4246 (4)	0.3046 (7)	0.53894 (17)	0.116 (3)
C20	0.4805 (4)	0.3411 (8)	0.52241 (17)	0.124 (4)
H20	0.526012	0.336326	0.536121	0.149*
C30	0.4107 (5)	0.3217 (7)	0.16096 (17)	0.116 (3)
H30A	0.408630	0.394801	0.153427	0.140*
H30B	0.400532	0.280473	0.136838	0.140*
C31	0.4837 (4)	0.2964 (6)	0.1809 (2)	0.111 (2)
H31A	0.487082	0.222395	0.186889	0.133*
H31B	0.518025	0.313050	0.163161	0.133*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu2	0.0139 (2)	0.0439 (3)	0.0306 (3)	0.00001 (17)	0.00304 (18)	0.00413 (19)
Cu1	0.0141 (2)	0.0458 (3)	0.0299 (3)	0.00123 (17)	0.00361 (18)	0.00215 (19)
O13	0.0140 (10)	0.0547 (15)	0.0323 (12)	−0.0001 (9)	0.0024 (9)	−0.0018 (10)
O2	0.0174 (10)	0.0651 (17)	0.0350 (13)	0.0040 (10)	0.0032 (9)	0.0106 (11)
O8	0.0192 (11)	0.0681 (17)	0.0344 (13)	0.0011 (11)	0.0019 (9)	0.0134 (12)
O1	0.0179 (10)	0.0665 (17)	0.0333 (12)	0.0046 (10)	0.0044 (9)	0.0117 (11)
O10	0.0242 (11)	0.0463 (14)	0.0505 (15)	−0.0023 (10)	0.0043 (10)	−0.0021 (11)
O12	0.0115 (10)	0.0643 (17)	0.0348 (13)	−0.0018 (9)	0.0026 (9)	−0.0039 (11)
O5	0.0208 (11)	0.0492 (15)	0.0481 (14)	−0.0002 (10)	0.0055 (10)	−0.0067 (11)
O4	0.0197 (11)	0.0505 (15)	0.0542 (16)	−0.0018 (10)	0.0058 (10)	−0.0065 (12)
O7	0.0195 (11)	0.0685 (17)	0.0341 (13)	−0.0011 (11)	0.0049 (9)	0.0095 (12)
O11	0.0251 (12)	0.0549 (16)	0.0491 (15)	0.0043 (11)	0.0045 (10)	−0.0068 (12)
O6	0.0317 (13)	0.0620 (19)	0.074 (2)	0.0072 (12)	0.0001 (13)	−0.0295 (15)
O14	0.0350 (15)	0.0607 (19)	0.078 (2)	−0.0055 (13)	0.0151 (14)	−0.0013 (15)
O3	0.0489 (17)	0.088 (2)	0.0479 (16)	0.0206 (16)	0.0153 (13)	0.0219 (16)
C9	0.0215 (14)	0.050 (2)	0.0336 (17)	0.0012 (13)	0.0068 (13)	−0.0020 (15)
C1	0.0244 (15)	0.0438 (19)	0.0319 (17)	0.0028 (13)	0.0035 (13)	0.0018 (14)
C8	0.0191 (14)	0.051 (2)	0.0298 (17)	0.0008 (13)	0.0029 (12)	0.0024 (14)
C2	0.0286 (16)	0.046 (2)	0.0368 (18)	0.0066 (14)	0.0082 (14)	0.0028 (15)
C15	0.0218 (14)	0.048 (2)	0.0305 (17)	−0.0002 (13)	0.0024 (12)	−0.0007 (14)
C22	0.0325 (17)	0.050 (2)	0.0359 (19)	0.0023 (15)	0.0022 (14)	0.0023 (15)
C16	0.0352 (18)	0.054 (2)	0.0364 (19)	0.0087 (16)	0.0139 (15)	0.0085 (16)
O15	0.073 (3)	0.113 (3)	0.078 (3)	−0.029 (2)	−0.010 (2)	−0.027 (2)
C3	0.0306 (17)	0.059 (2)	0.040 (2)	0.0040 (16)	0.0095 (15)	0.0056 (17)
C7	0.034 (2)	0.079 (3)	0.050 (2)	0.0111 (19)	0.0100 (17)	0.020 (2)
C12	0.0282 (17)	0.058 (2)	0.046 (2)	0.0032 (16)	0.0027 (15)	−0.0163 (18)
C14	0.0229 (16)	0.054 (2)	0.057 (2)	−0.0002 (15)	−0.0006 (15)	−0.0024 (18)
C10	0.0293 (18)	0.062 (3)	0.060 (3)	0.0056 (17)	−0.0023 (17)	−0.016 (2)
C4	0.0341 (19)	0.063 (3)	0.046 (2)	0.0114 (17)	0.0087 (16)	0.0089 (18)
C26	0.0169 (16)	0.091 (3)	0.047 (2)	−0.0021 (17)	0.0028 (15)	0.021 (2)
C27	0.0153 (16)	0.109 (4)	0.036 (2)	−0.0032 (18)	0.0046 (14)	−0.004 (2)

C13	0.0215 (16)	0.053 (2)	0.058 (2)	0.0058 (14)	0.0001 (15)	−0.0053 (18)
C11	0.0289 (18)	0.068 (3)	0.072 (3)	0.0014 (18)	−0.0059 (18)	−0.030 (2)
O9	0.147 (4)	0.254 (7)	0.091 (3)	0.129 (5)	0.079 (3)	0.122 (4)
C5	0.042 (2)	0.062 (3)	0.045 (2)	0.0126 (18)	0.0121 (17)	0.0139 (19)
C17	0.050 (2)	0.078 (3)	0.051 (2)	0.006 (2)	0.025 (2)	0.017 (2)
C25	0.0136 (16)	0.153 (5)	0.038 (2)	−0.001 (2)	0.0074 (15)	−0.019 (3)
C6	0.039 (2)	0.094 (4)	0.054 (3)	0.017 (2)	0.0193 (19)	0.029 (2)
C24	0.0168 (17)	0.143 (5)	0.040 (2)	0.008 (2)	0.0041 (15)	0.020 (3)
C28	0.056 (3)	0.134 (5)	0.057 (3)	−0.042 (3)	0.021 (2)	−0.011 (3)
C18	0.100 (4)	0.080 (4)	0.066 (3)	0.015 (3)	0.054 (3)	0.028 (3)
C21	0.047 (3)	0.139 (5)	0.041 (2)	0.026 (3)	0.004 (2)	0.032 (3)
C23	0.052 (3)	0.055 (3)	0.084 (3)	0.002 (2)	0.010 (2)	−0.016 (2)
C29	0.046 (3)	0.098 (4)	0.081 (4)	−0.006 (3)	0.015 (2)	−0.024 (3)
C19	0.132 (6)	0.166 (7)	0.061 (3)	0.103 (5)	0.049 (4)	0.063 (4)
C20	0.091 (5)	0.234 (10)	0.051 (3)	0.072 (6)	0.024 (3)	0.061 (5)
C30	0.172 (6)	0.132 (5)	0.046 (3)	−0.072 (5)	0.020 (4)	−0.036 (3)
C31	0.117 (5)	0.095 (4)	0.139 (5)	−0.027 (4)	0.090 (4)	−0.061 (4)

Geometric parameters (Å, º)

Cu2—O13^{i}	2.199 (2)	C12—C11	1.393 (5)
Cu2—O8	1.955 (2)	C14—H14	0.9500
Cu2—O1	1.957 (2)	C14—C13	1.370 (6)
Cu2—O10	1.981 (3)	C10—H10	0.9500
Cu2—O4	1.951 (3)	C10—C11	1.388 (6)
Cu1—O2	1.940 (2)	C4—H4	0.9500
Cu1—O12	2.174 (2)	C4—C5	1.382 (5)
Cu1—O5	1.948 (3)	C26—H26A	0.9900
Cu1—O7	1.953 (2)	C26—H26B	0.9900
Cu1—O11	1.984 (3)	C26—C27	1.512 (5)
O13—C26	1.419 (4)	C27—H27A	0.9900
O13—C25	1.431 (5)	C27—H27B	0.9900
O2—C1	1.259 (4)	C13—H13	0.9500
O8—C15	1.259 (4)	C11—H11	0.9500
O1—C1	1.264 (4)	O9—H9	0.8400
O10—C22	1.261 (4)	O9—C19	1.358 (6)
O12—C27	1.432 (4)	C5—C6	1.395 (6)
O12—C24	1.412 (5)	C17—H17	0.9500
O5—C8	1.266 (4)	C17—C18	1.399 (7)
O4—C8	1.257 (4)	C25—H25A	0.9900
O7—C15	1.270 (4)	C25—H25B	0.9900
O11—C22	1.267 (4)	C25—C24	1.514 (5)
O6—H6	0.8199	C6—H6A	0.9500
O6—C12	1.356 (5)	C24—H24A	0.9900
O14—C28	1.391 (5)	C24—H24B	0.9900
O14—C31	1.421 (7)	C28—H28A	0.9900
O3—H3	0.8400	C28—H28B	0.9900
O3—C5	1.345 (5)	C28—C29	1.419 (8)

C9—C8	1.475 (5)	C18—H18	0.9500
C9—C14	1.389 (5)	C18—C19	1.352 (10)
C9—C10	1.380 (5)	C21—H21	0.9500
C1—C2	1.491 (5)	C21—C20	1.389 (7)
C2—C3	1.401 (5)	C23—H23A	0.9800
C2—C7	1.382 (5)	C23—H23B	0.9800
C15—C16	1.474 (5)	C23—H23C	0.9800
C22—C23	1.492 (6)	C29—H29A	0.9900
C16—C17	1.378 (5)	C29—H29B	0.9900
C16—C21	1.380 (6)	C19—C20	1.380 (11)
O15—C29	1.412 (6)	C20—H20	0.9500
O15—C30	1.334 (8)	C30—H30A	0.9900
C3—H3A	0.9500	C30—H30B	0.9900
C3—C4	1.383 (5)	C30—C31	1.530 (12)
C7—H7	0.9500	C31—H31A	0.9900
C7—C6	1.384 (6)	C31—H31B	0.9900
C12—C13	1.381 (5)		

O8—Cu2—O13^{i}	94.87 (9)	H26A—C26—H26B	108.0
O8—Cu2—O1	171.23 (9)	C27—C26—H26A	109.4
O8—Cu2—O10	88.38 (12)	C27—C26—H26B	109.4
O1—Cu2—O13^{i}	93.90 (9)	O12—C27—C26	110.1 (3)
O1—Cu2—O10	90.61 (12)	O12—C27—H27A	109.6
O10—Cu2—O13^{i}	95.46 (10)	O12—C27—H27B	109.6
O4—Cu2—O13^{i}	96.05 (10)	C26—C27—H27A	109.6
O4—Cu2—O8	88.61 (12)	C26—C27—H27B	109.6
O4—Cu2—O1	90.65 (12)	H27A—C27—H27B	108.2
O4—Cu2—O10	168.31 (10)	C12—C13—H13	120.0
O2—Cu1—O12	96.93 (9)	C14—C13—C12	119.9 (3)
O2—Cu1—O5	89.40 (11)	C14—C13—H13	120.0
O2—Cu1—O7	168.97 (9)	C12—C11—H11	119.9
O2—Cu1—O11	88.41 (12)	C10—C11—C12	120.2 (4)
O5—Cu1—O12	95.24 (10)	C10—C11—H11	119.9
O5—Cu1—O7	90.13 (12)	C19—O9—H9	109.5
O5—Cu1—O11	171.36 (10)	O3—C5—C4	123.4 (4)
O7—Cu1—O12	94.09 (9)	O3—C5—C6	117.4 (4)
O7—Cu1—O11	90.42 (12)	C4—C5—C6	119.2 (4)
O11—Cu1—O12	93.33 (10)	C16—C17—H17	119.9
C26—O13—Cu2^{ii}	119.2 (2)	C16—C17—C18	120.1 (5)
C26—O13—C25	108.6 (3)	C18—C17—H17	119.9
C25—O13—Cu2^{ii}	118.0 (2)	O13—C25—H25A	109.7
C1—O2—Cu1	123.4 (2)	O13—C25—H25B	109.7
C15—O8—Cu2	121.6 (2)	O13—C25—C24	110.0 (4)
C1—O1—Cu2	121.6 (2)	H25A—C25—H25B	108.2
C22—O10—Cu2	123.5 (2)	C24—C25—H25A	109.7
C27—O12—Cu1	116.8 (2)	C24—C25—H25B	109.7
C24—O12—Cu1	121.7 (2)	C7—C6—C5	120.5 (4)
C24—O12—C27	110.1 (3)	C7—C6—H6A	119.7

C8—O5—Cu1	121.2 (2)	C5—C6—H6A	119.7
C8—O4—Cu2	124.0 (2)	O12—C24—C25	109.4 (4)
C15—O7—Cu1	123.3 (2)	O12—C24—H24A	109.8
C22—O11—Cu1	122.5 (2)	O12—C24—H24B	109.8
C12—O6—H6	114.7	C25—C24—H24A	109.8
C28—O14—C31	109.6 (4)	C25—C24—H24B	109.8
C5—O3—H3	109.5	H24A—C24—H24B	108.2
C14—C9—C8	119.7 (3)	O14—C28—H28A	108.9
C10—C9—C8	121.4 (3)	O14—C28—H28B	108.9
C10—C9—C14	118.9 (4)	O14—C28—C29	113.4 (5)
O2—C1—O1	124.8 (3)	H28A—C28—H28B	107.7
O2—C1—C2	116.6 (3)	C29—C28—H28A	108.9
O1—C1—C2	118.7 (3)	C29—C28—H28B	108.9
O5—C8—C9	118.4 (3)	C17—C18—H18	119.7
O4—C8—O5	124.4 (3)	C19—C18—C17	120.6 (5)
O4—C8—C9	117.2 (3)	C19—C18—H18	119.7
C3—C2—C1	119.7 (3)	C16—C21—H21	119.3
C7—C2—C1	121.9 (3)	C16—C21—C20	121.4 (5)
C7—C2—C3	118.4 (3)	C20—C21—H21	119.3
O8—C15—O7	124.6 (3)	C22—C23—H23A	109.5
O8—C15—C16	117.5 (3)	C22—C23—H23B	109.5
O7—C15—C16	117.9 (3)	C22—C23—H23C	109.5
O10—C22—O11	123.8 (4)	H23A—C23—H23B	109.5
O10—C22—C23	117.4 (4)	H23A—C23—H23C	109.5
O11—C22—C23	118.7 (4)	H23B—C23—H23C	109.5
C17—C16—C15	121.3 (4)	O15—C29—C28	113.7 (5)
C17—C16—C21	118.4 (4)	O15—C29—H29A	108.8
C21—C16—C15	120.2 (3)	O15—C29—H29B	108.8
C30—O15—C29	110.3 (4)	C28—C29—H29A	108.8
C2—C3—H3A	119.4	C28—C29—H29B	108.8
C4—C3—C2	121.2 (3)	H29A—C29—H29B	107.7
C4—C3—H3A	119.4	O9—C19—C20	120.9 (7)
C2—C7—H7	119.6	C18—C19—O9	118.9 (7)
C2—C7—C6	120.7 (4)	C18—C19—C20	120.2 (5)
C6—C7—H7	119.6	C21—C20—H20	120.4
O6—C12—C13	122.5 (3)	C19—C20—C21	119.1 (6)
O6—C12—C11	118.1 (3)	C19—C20—H20	120.4
C13—C12—C11	119.4 (4)	O15—C30—H30A	109.3
C9—C14—H14	119.3	O15—C30—H30B	109.3
C13—C14—C9	121.4 (3)	O15—C30—C31	111.6 (6)
C13—C14—H14	119.3	H30A—C30—H30B	108.0
C9—C10—H10	120.0	C31—C30—H30A	109.3
C9—C10—C11	120.1 (4)	C31—C30—H30B	109.3
C11—C10—H10	120.0	O14—C31—C30	109.6 (5)
C3—C4—H4	120.0	O14—C31—H31A	109.8
C5—C4—C3	120.0 (4)	O14—C31—H31B	109.8
C5—C4—H4	120.0	C30—C31—H31A	109.8
O13—C26—H26A	109.4	C30—C31—H31B	109.8

O13—C26—H26B	109.4	H31A—C31—H31B	108.2
O13—C26—C27	111.3 (3)		
Cu2^{ii}—O13—C26—C27	−162.8 (3)	C1—C2—C7—C6	178.9 (4)
Cu2^{ii}—O13—C25—C24	160.5 (3)	C8—C9—C14—C13	−177.7 (4)
Cu2—O8—C15—O7	−6.6 (5)	C8—C9—C10—C11	176.0 (4)
Cu2—O8—C15—C16	174.3 (3)	C2—C3—C4—C5	0.5 (7)
Cu2—O1—C1—O2	−2.6 (5)	C2—C7—C6—C5	0.5 (8)
Cu2—O1—C1—C2	177.3 (2)	C15—C16—C17—C18	179.9 (5)
Cu2—O10—C22—O11	−0.4 (5)	C15—C16—C21—C20	179.9 (6)
Cu2—O10—C22—C23	179.5 (3)	C16—C17—C18—C19	1.6 (9)
Cu2—O4—C8—O5	−2.3 (5)	C16—C21—C20—C19	−1.0 (12)
Cu2—O4—C8—C9	177.3 (2)	O15—C30—C31—O14	−57.8 (8)
Cu1—O2—C1—O1	−2.3 (5)	C3—C2—C7—C6	1.3 (7)
Cu1—O2—C1—C2	177.8 (2)	C3—C4—C5—O3	−179.4 (4)
Cu1—O12—C27—C26	−158.4 (3)	C3—C4—C5—C6	1.3 (7)
Cu1—O12—C24—C25	158.4 (3)	C7—C2—C3—C4	−1.8 (7)
Cu1—O5—C8—O4	−4.5 (5)	C14—C9—C8—O5	−161.0 (3)
Cu1—O5—C8—C9	175.9 (2)	C14—C9—C8—O4	19.3 (5)
Cu1—O7—C15—O8	1.9 (5)	C14—C9—C10—C11	−2.5 (6)
Cu1—O7—C15—C16	−179.1 (3)	C10—C9—C8—O5	20.5 (5)
Cu1—O11—C22—O10	−1.5 (5)	C10—C9—C8—O4	−159.1 (4)
Cu1—O11—C22—C23	178.5 (3)	C10—C9—C14—C13	0.8 (6)
O13—C26—C27—O12	−57.2 (5)	C4—C5—C6—C7	−1.8 (8)
O13—C25—C24—O12	61.2 (6)	C26—O13—C25—C24	−59.8 (5)
O2—C1—C2—C3	−0.1 (5)	C27—O12—C24—C25	−59.4 (5)
O2—C1—C2—C7	−177.6 (4)	C13—C12—C11—C10	0.3 (7)
O8—C15—C16—C17	−176.1 (4)	C11—C12—C13—C14	−1.9 (7)
O8—C15—C16—C21	5.2 (6)	O9—C19—C20—C21	−177.8 (8)
O1—C1—C2—C3	−180.0 (4)	C17—C16—C21—C20	1.2 (9)
O1—C1—C2—C7	2.5 (6)	C17—C18—C19—O9	177.5 (7)
O7—C15—C16—C17	4.7 (6)	C17—C18—C19—C20	−1.4 (12)
O7—C15—C16—C21	−174.0 (4)	C25—O13—C26—C27	58.1 (5)
O6—C12—C13—C14	177.3 (4)	C24—O12—C27—C26	57.4 (5)
O6—C12—C11—C10	−179.0 (4)	C28—O14—C31—C30	54.1 (8)
O14—C28—C29—O15	53.8 (8)	C18—C19—C20—C21	1.1 (13)
O3—C5—C6—C7	178.8 (5)	C21—C16—C17—C18	−1.4 (8)
C9—C14—C13—C12	1.4 (6)	C29—O15—C30—C31	55.7 (8)
C9—C10—C11—C12	1.9 (7)	C30—O15—C29—C28	−54.5 (9)
C1—C2—C3—C4	−179.4 (4)	C31—O14—C28—C29	−54.0 (8)

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

Bis(μ-acetato)bis(μ-4-hydroxybenzoato)bis[(1,4-diazabicyclo[2.2.2]octane)\ copper(II)] dioxane decasolvate (compound_11)

Crystal data

$[Cu_2(C_7H_5O_3)_2(C_2H_3O_2)_2(C_6H_{12}N_2)_2]\cdot 10C_4H_8O_2$
M_r = 1624.77
Monoclinic, *Pc*
a = 9.7404 (3) Å
b = 20.3001 (4) Å
c = 22.9088 (7) Å
β = 118.494 (4)°
V = 3981.1 (2) Å^3
Z = 2
F(000) = 1732
D_x = 1.355 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 12586 reflections
θ = 4.4–76.2°
μ = 1.36 mm^{-1}
T = 100 K
Irregular, clear green
0.34 × 0.25 × 0.14 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2023)
T_{min} = 0.533, T_{max} = 1.000
27595 measured reflections
12037 independent reflections
9752 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 78.3°, θ_{min} = 3.1°
h = −11→12
k = −25→21
l = −28→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.098
$wR(F^2)$ = 0.287
S = 1.21
12037 reflections
985 parameters
352 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.46 (6)

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. Twin: Twin law (-1.0, 0.0, 0.0, 0.0, -1.0, 0.0, 0.0, 0.0, -1.0), BASF [0.46 (6)]. One of the 1,4-diazabicyclo[2.2.2]octane (DABCO) ligands clearly showed disorder. This ligand was modelled in two components, in Parts 1 and 2. Atoms in component 2 have the lower occupancy and were allowed to remain isotropic. There was evidence ofor some disorder in the second DABCO. Attempts to model the second DABCO ligand over two sites were unsuccessful.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.85466 (11)	0.73020 (5)	0.69458 (5)	0.0479 (3)	
Cu2	1.09154 (12)	0.77234 (5)	0.80581 (5)	0.0510 (4)	

O1	0.7337 (8)	0.7533 (3)	0.7410 (3)	0.0545 (14)
O2	0.9351 (9)	0.7849 (4)	0.8363 (4)	0.0649 (19)
O3	1.0129 (8)	0.7148 (4)	0.6664 (3)	0.0566 (15)
O4	1.2119 (8)	0.7550 (4)	0.7585 (4)	0.0627 (18)
O5	0.8421 (9)	0.8227 (3)	0.6666 (3)	0.068 (2)
O6	1.0355 (8)	0.8600 (3)	0.7626 (3)	0.0628 (18)
O7	0.8164 (8)	1.1244 (3)	0.5991 (4)	0.0627 (18)
H7	0.735797	1.125104	0.561829	0.094*
O8	0.9025 (8)	0.6434 (3)	0.7376 (3)	0.0556 (16)
O9	1.1103 (9)	0.6788 (3)	0.8302 (3)	0.066 (2)
O10	1.1514 (8)	0.3737 (3)	0.8890 (3)	0.0580 (16)
H10	1.242643	0.370206	0.920603	0.087*
N1	0.6535 (9)	0.6937 (3)	0.6026 (3)	0.0513 (18)
N2	0.4233 (9)	0.6476 (3)	0.4961 (4)	0.0501 (17)
N3	1.2970 (9)	0.8068 (3)	0.8954 (4)	0.0494 (17)
N4	1.5416 (9)	0.8509 (3)	0.9987 (4)	0.0504 (17)
C1	0.7935 (13)	0.7739 (4)	0.7989 (5)	0.055 (2)
C2	0.6851 (14)	0.7869 (5)	0.8273 (5)	0.065 (3)
H2A	0.577999	0.791468	0.790999	0.098*
H2B	0.716445	0.827648	0.853370	0.098*
H2C	0.690263	0.750096	0.855914	0.098*
C3	1.1553 (12)	0.7302 (3)	0.7004 (5)	0.050 (2)
C4	1.2659 (13)	0.7193 (5)	0.6735 (5)	0.061 (2)
H4A	1.329110	0.680081	0.694197	0.092*
H4B	1.334402	0.757743	0.683438	0.092*
H4C	1.206838	0.713027	0.625350	0.092*
C23	0.9304 (11)	0.8674 (4)	0.7040 (5)	0.053 (2)
C24	0.9011 (10)	0.9353 (4)	0.6761 (4)	0.0465 (18)
C25	0.9800 (11)	0.9889 (4)	0.7165 (5)	0.057 (2)
H25	1.052472	0.982761	0.762134	0.068*
C26	0.9494 (12)	1.0524 (4)	0.6878 (5)	0.061 (3)
H26	1.004959	1.088933	0.714558	0.074*
C27	0.8426 (10)	1.0628 (4)	0.6229 (5)	0.0482 (18)
C28	0.7625 (11)	1.0083 (4)	0.5820 (5)	0.055 (2)
H28	0.690983	1.014453	0.536306	0.066*
C29	0.7902 (11)	0.9457 (4)	0.6100 (4)	0.055 (2)
H29	0.732614	0.909320	0.583570	0.066*
C30	1.0211 (10)	0.6345 (4)	0.7946 (4)	0.0476 (18)
C31	1.0515 (10)	0.5660 (4)	0.8206 (4)	0.0482 (19)
C32	1.1735 (11)	0.5534 (4)	0.8840 (4)	0.0524 (19)
H32	1.231937	0.589170	0.911329	0.063*
C33	1.2111 (10)	0.4893 (4)	0.9079 (4)	0.0514 (19)
H33	1.295430	0.481419	0.951050	0.062*
C34	1.1242 (10)	0.4367 (4)	0.8682 (4)	0.0489 (19)
C35	0.9965 (10)	0.4496 (4)	0.8065 (4)	0.0441 (17)
H35	0.932200	0.414425	0.780473	0.053*
C36	0.9632 (10)	0.5127 (4)	0.7831 (4)	0.0489 (19)
H36	0.877725	0.520475	0.740214	0.059*

C5	0.5817 (13)	0.7470 (4)	0.5539 (4)	0.071 (2)	
H5A	0.661791	0.768011	0.545101	0.086*	
H5B	0.539214	0.780894	0.571925	0.086*	
C6	0.4459 (14)	0.7184 (4)	0.4869 (5)	0.071 (2)	
H6A	0.348008	0.742948	0.474581	0.085*	
H6B	0.473148	0.723614	0.450713	0.085*	
C7	0.5318 (11)	0.6656 (5)	0.6174 (5)	0.0652 (18)	
H7A	0.497202	0.699641	0.638520	0.078*	
H7B	0.576483	0.628375	0.648895	0.078*	
C8	0.3893 (12)	0.6411 (6)	0.5523 (5)	0.0649 (18)	
H8A	0.367328	0.594432	0.557268	0.078*	
H8B	0.295776	0.667446	0.543298	0.078*	
C9	0.7044 (12)	0.6412 (5)	0.5746 (5)	0.067 (2)	
H9A	0.758000	0.606883	0.608765	0.081*	
H9B	0.780430	0.658960	0.561494	0.081*	
C10	0.5667 (12)	0.6099 (5)	0.5137 (5)	0.067 (2)	
H10A	0.588724	0.609772	0.475705	0.081*	
H10B	0.552632	0.563742	0.523670	0.081*	
C11	1.3898 (15)	0.7540 (6)	0.9385 (7)	0.0551 (16)	0.718 (10)
H11A	1.432578	0.726717	0.915159	0.066*	0.718 (10)
H11B	1.322873	0.725593	0.949579	0.066*	0.718 (10)
C12	1.5237 (16)	0.7814 (6)	1.0023 (6)	0.060 (4)	0.718 (10)
H12A	1.503952	0.771593	1.039974	0.072*	0.718 (10)
H12B	1.622080	0.759205	1.010895	0.072*	0.718 (10)
C15	1.2510 (15)	0.8512 (6)	0.9375 (6)	0.0545 (16)	0.718 (10)
H15A	1.194310	0.825070	0.955746	0.065*	0.718 (10)
H15B	1.181069	0.886781	0.909577	0.065*	0.718 (10)
C16	1.4002 (14)	0.8813 (6)	0.9950 (6)	0.056 (3)	0.718 (10)
H16A	1.402012	0.929361	0.988013	0.067*	0.718 (10)
H16B	1.398941	0.874346	1.037537	0.067*	0.718 (10)
C19	1.3896 (15)	0.8494 (6)	0.8764 (6)	0.0548 (16)	0.718 (10)
H19A	1.331843	0.890677	0.856814	0.066*	0.718 (10)
H19B	1.408878	0.827023	0.842540	0.066*	0.718 (10)
C20	1.5472 (15)	0.8653 (7)	0.9384 (6)	0.058 (3)	0.718 (10)
H20A	1.631272	0.838998	0.937081	0.070*	0.718 (10)
H20B	1.572144	0.912470	0.937805	0.070*	0.718 (10)
C13	1.271 (3)	0.8015 (14)	0.9559 (13)	0.0545 (16)*	0.282 (10)
H13A	1.235087	0.756741	0.958949	0.065*	0.282 (10)
H13B	1.191994	0.833793	0.952728	0.065*	0.282 (10)
C14	1.432 (3)	0.816 (2)	1.0179 (11)	0.080 (10)*	0.282 (10)
H14A	1.415467	0.844130	1.049389	0.096*	0.282 (10)
H14B	1.480252	0.774396	1.040558	0.096*	0.282 (10)
C17	1.327 (3)	0.8756 (10)	0.8885 (11)	0.0543 (16)*	0.282 (10)
H17A	1.233185	0.901935	0.879702	0.065*	0.282 (10)
H17B	1.346134	0.880739	0.850136	0.065*	0.282 (10)
C18	1.469 (3)	0.9017 (10)	0.9517 (11)	0.048 (6)*	0.282 (10)
H18A	1.544868	0.921148	0.939613	0.058*	0.282 (10)
H18B	1.433958	0.936897	0.971671	0.058*	0.282 (10)

C21	1.428 (2)	0.7627 (12)	0.9128 (14)	0.0545 (16)*	0.282 (10)
H21A	1.442221	0.754562	0.873300	0.065*	0.282 (10)
H21B	1.408343	0.720003	0.928145	0.065*	0.282 (10)
C22	1.578 (2)	0.7947 (14)	0.9686 (14)	0.062 (8)*	0.282 (10)
H22A	1.636052	0.761343	1.003274	0.074*	0.282 (10)
H22B	1.645886	0.809399	0.949975	0.074*	0.282 (10)
O11	0.6469 (17)	1.2339 (5)	0.6830 (6)	0.116 (4)	
O12	0.9058 (11)	1.1719 (4)	0.7875 (5)	0.088 (2)	
C37	0.805 (3)	1.2511 (7)	0.7007 (9)	0.108 (5)	
H37A	0.810676	1.298416	0.691462	0.130*	
H37B	0.839709	1.225312	0.673301	0.130*	
C38	0.914 (2)	1.2374 (6)	0.7737 (8)	0.093 (4)	
H38A	1.022609	1.248661	0.784663	0.112*	
H38B	0.884026	1.265241	0.801290	0.112*	
C39	0.7412 (18)	1.1545 (9)	0.7692 (9)	0.098 (4)	
H39A	0.703883	1.181747	0.794602	0.118*	
H39B	0.733860	1.107483	0.778986	0.118*	
C40	0.6454 (17)	1.1674 (9)	0.6963 (9)	0.100 (4)	
H40A	0.687781	1.141840	0.671755	0.120*	
H40B	0.536496	1.152935	0.681097	0.120*	
O13	1.355 (2)	1.1690 (8)	0.8567 (11)	0.165 (5)	
O14	1.445 (3)	1.0405 (9)	0.9130 (13)	0.180 (6)	
C41	1.275 (3)	1.1275 (10)	0.8797 (15)	0.149 (5)	
H41A	1.200759	1.100376	0.841917	0.179*	
H41B	1.213476	1.155389	0.893987	0.179*	
C42	1.372 (4)	1.0832 (12)	0.9345 (16)	0.170 (6)	
H42A	1.450083	1.108938	0.972612	0.204*	
H42B	1.305914	1.058761	0.949108	0.204*	
C43	1.536 (4)	1.0739 (11)	0.8949 (17)	0.162 (6)	
H43A	1.574782	1.042848	0.872779	0.194*	
H43B	1.627666	1.089920	0.935585	0.194*	
C44	1.465 (4)	1.1308 (12)	0.8501 (17)	0.171 (6)	
H44A	1.551154	1.160420	0.855505	0.206*	
H44B	1.415657	1.114120	0.803947	0.206*	
O16	1.4098 (13)	0.8818 (4)	0.6857 (5)	0.094 (3)	
O15	1.4720 (14)	1.0174 (5)	0.7067 (8)	0.113 (4)	
C45	1.3353 (18)	0.9927 (8)	0.7042 (10)	0.099 (4)	
H45A	1.247286	0.997265	0.658592	0.119*	
H45B	1.309755	1.018077	0.734664	0.119*	
C46	1.359 (2)	0.9209 (7)	0.7242 (7)	0.090 (4)	
H46A	1.436835	0.917540	0.771757	0.108*	
H46B	1.258860	0.902753	0.718892	0.108*	
C47	1.5375 (19)	0.9108 (6)	0.6829 (10)	0.088 (4)	
H47A	1.557816	0.886458	0.650303	0.105*	
H47B	1.631343	0.906628	0.726872	0.105*	
C48	1.511 (2)	0.9830 (6)	0.6634 (9)	0.094 (4)	
H48A	1.607434	1.001937	0.665651	0.113*	
H48B	1.425969	0.987324	0.617141	0.113*	

O17	0.7898 (13)	0.9441 (4)	0.9096 (5)	0.096 (3)
O18	1.0942 (14)	0.9954 (5)	0.9541 (6)	0.106 (3)
C49	0.9221 (18)	0.9168 (7)	0.9667 (8)	0.094 (4)
H49A	0.964045	0.878968	0.952838	0.113*
H49B	0.888839	0.900485	0.998762	0.113*
C50	1.046 (2)	0.9674 (10)	0.9992 (10)	0.111 (5)
H50A	1.006810	1.003012	1.016842	0.133*
H50B	1.137750	0.947185	1.037082	0.133*
C51	0.955 (3)	1.0224 (8)	0.8978 (10)	0.112 (5)
H51A	0.908601	1.056936	0.913464	0.135*
H51B	0.983877	1.042620	0.865879	0.135*
C52	0.843 (3)	0.9707 (8)	0.8657 (8)	0.115 (6)
H52A	0.891924	0.935246	0.852127	0.139*
H52B	0.753256	0.988256	0.825293	0.139*
O19	1.1865 (17)	0.7493 (9)	1.0421 (7)	0.141 (6)
O20	0.9620 (16)	0.7060 (7)	1.0781 (10)	0.130 (5)
C53	1.226 (2)	0.7344 (13)	1.1086 (10)	0.127 (7)
H53A	1.310960	0.764189	1.138406	0.153*
H53B	1.266262	0.688800	1.118443	0.153*
C54	1.093 (2)	0.7405 (13)	1.1245 (12)	0.132 (8)
H54A	1.126336	0.722839	1.169497	0.159*
H54B	1.066390	0.787569	1.124147	0.159*
C55	0.918 (2)	0.7239 (8)	1.0126 (11)	0.112 (6)
H55A	0.886360	0.770870	1.005777	0.135*
H55B	0.826590	0.697206	0.981839	0.135*
C56	1.055 (2)	0.7129 (11)	0.9976 (11)	0.114 (5)
H56A	1.081314	0.665438	1.001740	0.137*
H56B	1.023119	0.726698	0.951508	0.137*
O21	1.5083 (12)	0.5874 (10)	1.0261 (5)	0.144 (6)
O22	1.6773 (10)	0.5843 (4)	1.1660 (4)	0.083 (2)
C57	1.6700 (16)	0.6025 (7)	1.0601 (7)	0.084 (3)
H57A	1.684826	0.650769	1.065923	0.101*
H57B	1.720335	0.586978	1.033936	0.101*
C58	1.7448 (15)	0.5686 (5)	1.1280 (6)	0.086 (4)
H58A	1.738670	0.520347	1.121272	0.104*
H58B	1.856714	0.580821	1.152327	0.104*
C59	1.5175 (17)	0.5698 (9)	1.1285 (8)	0.096 (4)
H59A	1.464938	0.581742	1.154818	0.115*
H59B	1.505338	0.521745	1.120472	0.115*
C60	1.4398 (14)	0.6033 (10)	1.0653 (6)	0.096 (5)
H60A	1.328134	0.590622	1.041981	0.116*
H60B	1.445787	0.651535	1.072703	0.116*
O23	1.3926 (18)	0.3381 (8)	0.7451 (7)	0.137 (4)
O24	1.5903 (12)	0.3973 (7)	0.8647 (5)	0.104 (3)
C61	1.555 (3)	0.3247 (13)	0.7834 (12)	0.138 (5)
H61A	1.596662	0.310842	0.753329	0.165*
H61B	1.570681	0.287809	0.814182	0.165*
C62	1.643 (2)	0.3825 (11)	0.8219 (9)	0.112 (4)

H62A	1.756423	0.372354	0.846300	0.134*
H62B	1.627309	0.420182	0.791877	0.134*
C63	1.424 (2)	0.4104 (11)	0.8305 (10)	0.113 (4)
H63A	1.402252	0.450066	0.802338	0.136*
H63B	1.387164	0.418847	0.863335	0.136*
C64	1.340 (2)	0.3539 (11)	0.7892 (10)	0.112 (4)
H64A	1.353896	0.315461	0.818036	0.134*
H64B	1.227279	0.364176	0.764380	0.134*
O25	0.9100 (12)	0.5321 (9)	0.5691 (7)	0.127 (5)
O26	1.1814 (14)	0.4963 (16)	0.5693 (7)	0.207 (12)
C65	0.9256 (19)	0.5368 (10)	0.5116 (10)	0.117 (6)
H65A	0.972828	0.579644	0.510557	0.140*
H65B	0.821868	0.533594	0.471701	0.140*
C66	1.028 (2)	0.4819 (14)	0.5123 (9)	0.147 (10)
H66A	0.987350	0.438982	0.518121	0.176*
H66B	1.033983	0.480881	0.470468	0.176*
C67	1.166 (2)	0.4967 (10)	0.6256 (9)	0.122 (7)
H67A	1.269889	0.504509	0.664504	0.146*
H67B	1.129061	0.452963	0.631201	0.146*
C68	1.060 (2)	0.5458 (9)	0.6241 (11)	0.114 (6)
H68A	1.096392	0.589957	0.619002	0.137*
H68B	1.054527	0.545099	0.666103	0.137*
O27	0.8625 (18)	0.8288 (6)	0.4919 (7)	0.124 (4)
O28	1.1376 (14)	0.7734 (5)	0.5050 (5)	0.097 (3)
C69	0.865 (3)	0.7660 (8)	0.4695 (11)	0.139 (10)
H69A	0.762695	0.755737	0.431005	0.167*
H69B	0.885279	0.733758	0.505057	0.167*
C70	0.996 (3)	0.7604 (11)	0.4486 (9)	0.127 (7)
H70A	0.996343	0.715533	0.431740	0.153*
H70B	0.976981	0.792525	0.413111	0.153*
C71	1.143 (2)	0.8375 (7)	0.5315 (8)	0.103 (5)
H71A	1.134290	0.871621	0.498989	0.123*
H71B	1.243293	0.843948	0.572668	0.123*
C72	1.002 (2)	0.8431 (7)	0.5469 (8)	0.100 (4)
H72A	1.018736	0.812357	0.583215	0.120*
H72B	0.997980	0.888406	0.562096	0.120*
O29	1.4761 (9)	0.5705 (4)	0.7695 (4)	0.0687 (17)
O30	1.6818 (10)	0.6123 (5)	0.9001 (4)	0.085 (2)
C73	1.6225 (15)	0.5983 (6)	0.7883 (6)	0.074 (3)
H73A	1.608943	0.646166	0.779051	0.088*
H73B	1.664376	0.579168	0.760209	0.088*
C74	1.7380 (16)	0.5888 (8)	0.8580 (6)	0.081 (3)
H74A	1.762869	0.541337	0.866595	0.097*
H74B	1.835408	0.612406	0.867488	0.097*
C75	1.5335 (15)	0.5819 (7)	0.8839 (6)	0.077 (3)
H75A	1.490328	0.601291	0.911364	0.093*
H75B	1.547628	0.534028	0.892954	0.093*
C76	1.4250 (14)	0.5935 (6)	0.8125 (5)	0.068 (3)

H76A	1.323859	0.572301	0.801178	0.082*
H76B	1.406049	0.641511	0.805425	0.082*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0440 (7)	0.0355 (6)	0.0306 (6)	0.0017 (5)	−0.0095 (5)	−0.0007 (4)
Cu2	0.0467 (7)	0.0319 (5)	0.0319 (6)	0.0010 (4)	−0.0156 (5)	0.0007 (4)
O1	0.047 (3)	0.057 (3)	0.033 (3)	0.001 (3)	−0.004 (3)	−0.010 (3)
O2	0.055 (4)	0.059 (3)	0.040 (4)	0.000 (3)	−0.009 (3)	−0.007 (3)
O3	0.046 (4)	0.067 (3)	0.032 (3)	−0.006 (3)	−0.002 (3)	−0.008 (3)
O4	0.048 (3)	0.055 (3)	0.047 (4)	0.000 (3)	−0.008 (3)	−0.014 (3)
O5	0.070 (4)	0.034 (3)	0.042 (3)	0.002 (3)	−0.020 (3)	0.006 (2)
O6	0.060 (4)	0.035 (3)	0.040 (3)	0.006 (3)	−0.019 (3)	0.006 (2)
O7	0.053 (3)	0.043 (3)	0.048 (4)	0.000 (2)	−0.012 (3)	0.004 (2)
O8	0.048 (3)	0.037 (3)	0.035 (3)	−0.003 (2)	−0.017 (3)	0.000 (2)
O9	0.062 (4)	0.035 (3)	0.039 (3)	−0.001 (3)	−0.026 (3)	0.004 (2)
O10	0.051 (3)	0.037 (3)	0.047 (3)	0.000 (2)	−0.009 (3)	0.012 (2)
N1	0.046 (4)	0.037 (3)	0.033 (4)	0.005 (3)	−0.012 (3)	−0.002 (3)
N2	0.050 (4)	0.035 (3)	0.036 (3)	−0.001 (3)	−0.004 (3)	−0.002 (3)
N3	0.044 (4)	0.038 (3)	0.033 (4)	−0.002 (3)	−0.007 (3)	−0.001 (3)
N4	0.043 (4)	0.045 (3)	0.034 (3)	0.002 (3)	−0.005 (3)	−0.007 (3)
C1	0.055 (6)	0.042 (4)	0.045 (5)	−0.005 (3)	0.005 (5)	0.001 (3)
C2	0.064 (6)	0.048 (4)	0.048 (5)	0.004 (4)	−0.002 (5)	−0.008 (4)
C3	0.048 (5)	0.036 (4)	0.033 (4)	0.002 (3)	−0.008 (4)	−0.001 (3)
C4	0.051 (5)	0.061 (5)	0.043 (5)	0.000 (4)	−0.001 (4)	0.005 (4)
C23	0.045 (4)	0.040 (4)	0.039 (4)	0.003 (3)	−0.010 (4)	0.006 (3)
C24	0.037 (4)	0.041 (4)	0.031 (3)	0.007 (3)	−0.008 (3)	0.005 (3)
C25	0.045 (5)	0.038 (4)	0.045 (5)	−0.001 (3)	−0.013 (4)	0.001 (3)
C26	0.051 (5)	0.036 (4)	0.055 (5)	0.002 (4)	−0.008 (4)	0.007 (4)
C27	0.041 (4)	0.038 (3)	0.046 (4)	0.001 (3)	0.005 (4)	0.003 (3)
C28	0.052 (5)	0.037 (3)	0.038 (4)	0.008 (3)	−0.010 (4)	0.007 (2)
C29	0.049 (4)	0.033 (3)	0.035 (3)	0.001 (3)	−0.019 (3)	0.004 (3)
C30	0.040 (4)	0.040 (4)	0.036 (4)	0.001 (3)	−0.003 (3)	0.003 (3)
C31	0.040 (4)	0.041 (4)	0.036 (4)	0.000 (3)	−0.005 (3)	0.002 (3)
C32	0.046 (4)	0.049 (4)	0.036 (4)	−0.001 (3)	−0.002 (4)	0.000 (3)
C33	0.044 (4)	0.045 (4)	0.032 (4)	−0.004 (3)	−0.009 (3)	0.003 (3)
C34	0.040 (4)	0.038 (3)	0.038 (4)	−0.003 (3)	−0.006 (3)	0.007 (3)
C35	0.037 (4)	0.039 (3)	0.031 (4)	−0.003 (3)	−0.004 (3)	−0.003 (3)
C36	0.041 (4)	0.043 (4)	0.030 (4)	0.000 (3)	−0.010 (3)	0.005 (3)
C5	0.070 (4)	0.041 (3)	0.034 (3)	−0.006 (3)	−0.031 (3)	0.000 (2)
C6	0.071 (4)	0.041 (3)	0.034 (3)	−0.007 (3)	−0.030 (3)	0.000 (2)
C7	0.053 (4)	0.066 (4)	0.042 (3)	−0.009 (3)	−0.006 (3)	−0.014 (3)
C8	0.053 (4)	0.066 (4)	0.042 (3)	−0.008 (3)	−0.006 (3)	−0.014 (3)
C9	0.050 (3)	0.054 (3)	0.050 (4)	0.003 (3)	−0.016 (3)	−0.021 (3)
C10	0.050 (3)	0.054 (3)	0.050 (4)	0.004 (3)	−0.015 (3)	−0.021 (3)
C11	0.0548 (18)	0.0544 (18)	0.0547 (18)	0.0000 (9)	0.0251 (12)	0.0001 (9)
C12	0.048 (6)	0.050 (6)	0.041 (6)	0.005 (5)	−0.013 (5)	−0.001 (5)

C15	0.0545 (17)	0.0544 (17)	0.0542 (17)	0.0001 (9)	0.0255 (12)	−0.0003 (9)
C16	0.048 (6)	0.053 (6)	0.040 (6)	0.007 (5)	−0.002 (5)	−0.008 (5)
C19	0.0546 (17)	0.0548 (17)	0.0543 (17)	−0.0002 (9)	0.0255 (12)	−0.0003 (9)
C20	0.056 (7)	0.060 (6)	0.038 (6)	0.000 (5)	0.007 (5)	0.000 (5)
O11	0.117 (9)	0.095 (7)	0.091 (8)	0.037 (6)	0.013 (7)	0.009 (5)
O12	0.083 (5)	0.072 (4)	0.075 (5)	0.012 (4)	0.011 (5)	0.015 (4)
C37	0.150 (17)	0.075 (8)	0.086 (10)	0.009 (9)	0.046 (11)	0.017 (7)
C38	0.106 (11)	0.073 (6)	0.088 (9)	−0.005 (7)	0.036 (9)	0.000 (6)
C39	0.077 (8)	0.115 (10)	0.105 (11)	0.012 (7)	0.044 (8)	0.029 (8)
C40	0.065 (7)	0.122 (11)	0.103 (11)	0.005 (7)	0.033 (8)	0.002 (9)
O13	0.190 (12)	0.116 (8)	0.222 (13)	0.017 (7)	0.127 (11)	0.027 (8)
O14	0.192 (12)	0.119 (8)	0.242 (14)	0.021 (8)	0.114 (12)	0.005 (8)
C41	0.163 (12)	0.106 (8)	0.221 (14)	0.026 (8)	0.126 (11)	0.030 (9)
C42	0.188 (13)	0.116 (9)	0.227 (14)	0.029 (9)	0.115 (12)	0.017 (9)
C43	0.185 (13)	0.110 (9)	0.227 (14)	0.026 (9)	0.127 (12)	0.010 (9)
C44	0.197 (13)	0.129 (9)	0.222 (14)	0.015 (9)	0.127 (12)	0.014 (9)
O16	0.098 (6)	0.072 (5)	0.089 (6)	−0.012 (5)	0.027 (6)	−0.007 (4)
O15	0.105 (8)	0.086 (6)	0.163 (11)	−0.012 (5)	0.076 (8)	−0.030 (6)
C45	0.075 (8)	0.105 (9)	0.134 (13)	−0.008 (7)	0.064 (9)	−0.016 (9)
C46	0.100 (10)	0.088 (8)	0.067 (7)	0.004 (7)	0.028 (8)	0.012 (6)
C47	0.081 (8)	0.067 (6)	0.119 (12)	−0.005 (6)	0.050 (9)	−0.002 (7)
C48	0.102 (10)	0.077 (7)	0.121 (12)	0.000 (7)	0.067 (10)	0.008 (7)
O17	0.089 (6)	0.062 (4)	0.095 (7)	−0.006 (4)	0.009 (5)	−0.013 (4)
O18	0.094 (7)	0.092 (6)	0.106 (8)	−0.026 (5)	0.028 (7)	−0.010 (6)
C49	0.084 (8)	0.087 (8)	0.079 (8)	−0.011 (7)	0.013 (7)	−0.014 (6)
C50	0.109 (12)	0.118 (12)	0.089 (11)	−0.032 (10)	0.034 (10)	−0.038 (9)
C51	0.124 (14)	0.088 (8)	0.103 (11)	−0.017 (9)	0.036 (11)	−0.006 (8)
C52	0.162 (18)	0.079 (8)	0.060 (7)	−0.014 (9)	0.016 (9)	0.003 (6)
O19	0.097 (8)	0.190 (13)	0.073 (7)	−0.043 (9)	−0.011 (6)	0.041 (8)
O20	0.087 (7)	0.130 (9)	0.174 (14)	−0.019 (7)	0.063 (9)	−0.021 (10)
C53	0.086 (11)	0.18 (2)	0.076 (11)	0.012 (11)	0.009 (9)	0.008 (11)
C54	0.075 (9)	0.173 (19)	0.114 (15)	−0.007 (11)	0.016 (10)	−0.057 (14)
C55	0.082 (10)	0.085 (9)	0.109 (13)	0.001 (7)	−0.004 (9)	0.044 (8)
C56	0.080 (10)	0.130 (13)	0.104 (13)	−0.011 (9)	0.021 (10)	0.013 (11)
O21	0.062 (5)	0.283 (19)	0.042 (4)	0.030 (8)	−0.013 (4)	−0.001 (7)
O22	0.062 (4)	0.085 (5)	0.057 (4)	−0.004 (4)	−0.009 (4)	0.006 (4)
C57	0.068 (7)	0.097 (8)	0.068 (7)	0.000 (6)	0.017 (6)	0.004 (6)
C58	0.073 (7)	0.059 (5)	0.064 (6)	0.008 (5)	−0.018 (6)	−0.002 (5)
C59	0.065 (7)	0.111 (10)	0.080 (8)	0.001 (7)	0.009 (7)	−0.009 (7)
C60	0.048 (5)	0.161 (14)	0.056 (6)	0.027 (7)	0.005 (5)	0.001 (7)
O23	0.111 (7)	0.151 (8)	0.105 (7)	0.010 (7)	0.016 (6)	−0.027 (6)
O24	0.076 (5)	0.156 (8)	0.070 (5)	−0.020 (5)	0.027 (4)	−0.017 (5)
C61	0.108 (8)	0.157 (10)	0.105 (8)	0.008 (8)	0.015 (7)	−0.027 (8)
C62	0.087 (7)	0.163 (9)	0.079 (6)	−0.012 (7)	0.035 (6)	−0.028 (7)
C63	0.087 (7)	0.150 (9)	0.090 (7)	−0.003 (7)	0.032 (6)	−0.012 (7)
C64	0.092 (7)	0.137 (9)	0.096 (7)	0.005 (7)	0.036 (6)	−0.022 (7)
O25	0.055 (5)	0.205 (14)	0.096 (8)	0.004 (6)	0.015 (5)	−0.045 (9)
O26	0.059 (6)	0.46 (4)	0.079 (8)	0.035 (12)	0.014 (6)	0.010 (14)

C65	0.064 (8)	0.146 (15)	0.098 (12)	0.014 (9)	0.004 (8)	0.046 (11)
C66	0.072 (9)	0.25 (3)	0.071 (9)	0.012 (13)	−0.004 (8)	−0.025 (13)
C67	0.104 (11)	0.120 (12)	0.077 (9)	0.042 (10)	−0.008 (8)	−0.020 (8)
C68	0.097 (11)	0.105 (11)	0.106 (12)	0.000 (8)	0.020 (9)	−0.041 (9)
O27	0.126 (9)	0.111 (8)	0.113 (9)	0.049 (7)	0.039 (8)	0.035 (7)
O28	0.097 (7)	0.098 (6)	0.065 (5)	0.032 (5)	0.014 (5)	0.008 (4)
C69	0.152 (19)	0.075 (9)	0.093 (12)	−0.007 (9)	−0.021 (13)	−0.001 (7)
C70	0.138 (17)	0.134 (15)	0.065 (9)	0.025 (13)	0.012 (10)	−0.031 (9)
C71	0.107 (11)	0.076 (7)	0.089 (9)	0.020 (7)	0.017 (9)	0.003 (6)
C72	0.118 (12)	0.079 (7)	0.086 (9)	0.034 (8)	0.035 (10)	0.024 (7)
O29	0.065 (4)	0.075 (4)	0.049 (4)	0.010 (3)	0.014 (3)	0.002 (3)
O30	0.070 (5)	0.118 (6)	0.050 (4)	0.000 (5)	0.016 (4)	0.003 (4)
C73	0.066 (6)	0.083 (7)	0.060 (6)	0.011 (5)	0.020 (5)	−0.002 (5)
C74	0.067 (6)	0.113 (9)	0.055 (6)	0.022 (6)	0.022 (5)	0.019 (6)
C75	0.065 (6)	0.091 (7)	0.061 (6)	0.012 (5)	0.017 (5)	0.021 (5)
C76	0.060 (6)	0.083 (7)	0.047 (5)	0.006 (5)	0.014 (5)	0.006 (5)

Geometric parameters (Å, º)

Cu1—O1	1.984 (7)	C40—H40B	0.9900
Cu1—O3	1.959 (7)	O13—C41	1.405 (19)
Cu1—O5	1.970 (6)	O13—C44	1.389 (19)
Cu1—O8	1.963 (6)	O14—C42	1.355 (19)
Cu1—N1	2.211 (7)	O14—C43	1.33 (2)
Cu2—O2	1.973 (9)	C41—H41A	0.9900
Cu2—O4	1.971 (8)	C41—H41B	0.9900
Cu2—O6	1.982 (6)	C41—C42	1.46 (2)
Cu2—O9	1.963 (6)	C42—H42A	0.9900
Cu2—N3	2.189 (7)	C42—H42B	0.9900
O1—C1	1.240 (12)	C43—H43A	0.9900
O2—C1	1.248 (13)	C43—H43B	0.9900
O3—C3	1.264 (12)	C43—C44	1.48 (2)
O4—C3	1.277 (12)	C44—H44A	0.9900
O5—C23	1.262 (11)	C44—H44B	0.9900
O6—C23	1.250 (10)	O16—C46	1.442 (19)
O7—H7	0.8400	O16—C47	1.404 (18)
O7—C27	1.341 (10)	O15—C45	1.398 (18)
O8—C30	1.279 (10)	O15—C48	1.409 (19)
O9—C30	1.245 (10)	C45—H45A	0.9900
O10—H10	0.8400	C45—H45B	0.9900
O10—C34	1.346 (10)	C45—C46	1.51 (2)
N1—C5	1.470 (11)	C46—H46A	0.9900
N1—C7	1.493 (14)	C46—H46B	0.9900
N1—C9	1.448 (12)	C47—H47A	0.9900
N2—C6	1.485 (11)	C47—H47B	0.9900
N2—C8	1.478 (15)	C47—C48	1.518 (19)
N2—C10	1.471 (13)	C48—H48A	0.9900
N3—C11	1.444 (15)	C48—H48B	0.9900

N3—C15	1.535 (15)	O17—C49	1.439 (19)
N3—C19	1.458 (14)	O17—C52	1.44 (2)
N3—C13	1.52 (3)	O18—C50	1.44 (2)
N3—C17	1.45 (2)	O18—C51	1.46 (2)
N3—C21	1.45 (2)	C49—H49A	0.9900
N4—C12	1.429 (15)	C49—H49B	0.9900
N4—C16	1.474 (15)	C49—C50	1.49 (2)
N4—C20	1.439 (15)	C50—H50A	0.9900
N4—C14	1.51 (3)	C50—H50B	0.9900
N4—C18	1.41 (2)	C51—H51A	0.9900
N4—C22	1.46 (2)	C51—H51B	0.9900
C1—C2	1.503 (17)	C51—C52	1.43 (2)
C2—H2A	0.9800	C52—H52A	0.9900
C2—H2B	0.9800	C52—H52B	0.9900
C2—H2C	0.9800	O19—C53	1.41 (2)
C3—C4	1.489 (17)	O19—C56	1.41 (2)
C4—H4A	0.9800	O20—C54	1.40 (2)
C4—H4B	0.9800	O20—C55	1.40 (3)
C4—H4C	0.9800	C53—H53A	0.9900
C23—C24	1.489 (11)	C53—H53B	0.9900
C24—C25	1.396 (12)	C53—C54	1.50 (3)
C24—C29	1.394 (10)	C54—H54A	0.9900
C25—H25	0.9500	C54—H54B	0.9900
C25—C26	1.412 (12)	C55—H55A	0.9900
C26—H26	0.9500	C55—H55B	0.9900
C26—C27	1.365 (13)	C55—C56	1.55 (3)
C27—C28	1.419 (12)	C56—H56A	0.9900
C28—H28	0.9500	C56—H56B	0.9900
C28—C29	1.390 (11)	O21—C57	1.419 (18)
C29—H29	0.9500	O21—C60	1.388 (18)
C30—C31	1.485 (12)	O22—C58	1.355 (19)
C31—C32	1.393 (12)	O22—C59	1.404 (16)
C31—C36	1.394 (12)	C57—H57A	0.9900
C32—H32	0.9500	C57—H57B	0.9900
C32—C33	1.391 (13)	C57—C58	1.530 (18)
C33—H33	0.9500	C58—H58A	0.9900
C33—C34	1.398 (12)	C58—H58B	0.9900
C34—C35	1.391 (11)	C59—H59A	0.9900
C35—H35	0.9500	C59—H59B	0.9900
C35—C36	1.367 (12)	C59—C60	1.44 (2)
C36—H36	0.9500	C60—H60A	0.9900
C5—H5A	0.9900	C60—H60B	0.9900
C5—H5B	0.9900	O23—C61	1.42 (3)
C5—C6	1.582 (11)	O23—C64	1.37 (2)
C6—H6A	0.9900	O24—C62	1.343 (19)
C6—H6B	0.9900	O24—C63	1.45 (2)
C7—H7A	0.9900	C61—H61A	0.9900
C7—H7B	0.9900	C61—H61B	0.9900

C7—C8	1.557 (12)	C61—C62	1.47 (3)
C8—H8A	0.9900	C62—H62A	0.9900
C8—H8B	0.9900	C62—H62B	0.9900
C9—H9A	0.9900	C63—H63A	0.9900
C9—H9B	0.9900	C63—H63B	0.9900
C9—C10	1.538 (11)	C63—C64	1.46 (3)
C10—H10A	0.9900	C64—H64A	0.9900
C10—H10B	0.9900	C64—H64B	0.9900
C11—H11A	0.9900	O25—C65	1.40 (2)
C11—H11B	0.9900	O25—C68	1.43 (2)
C11—C12	1.526 (15)	O26—C66	1.47 (2)
C12—H12A	0.9900	O26—C67	1.37 (2)
C12—H12B	0.9900	C65—H65A	0.9900
C15—H15A	0.9900	C65—H65B	0.9900
C15—H15B	0.9900	C65—C66	1.49 (2)
C15—C16	1.547 (15)	C66—H66A	0.9900
C16—H16A	0.9900	C66—H66B	0.9900
C16—H16B	0.9900	C67—H67A	0.9900
C19—H19A	0.9900	C67—H67B	0.9900
C19—H19B	0.9900	C67—C68	1.422 (17)
C19—C20	1.549 (15)	C68—H68A	0.9900
C20—H20A	0.9900	C68—H68B	0.9900
C20—H20B	0.9900	O27—C69	1.38 (2)
C13—H13A	0.9900	O27—C72	1.37 (2)
C13—H13B	0.9900	O28—C70	1.40 (2)
C13—C14	1.56 (2)	O28—C71	1.426 (18)
C14—H14A	0.9900	C69—H69A	0.9900
C14—H14B	0.9900	C69—H69B	0.9900
C17—H17A	0.9900	C69—C70	1.56 (4)
C17—H17B	0.9900	C70—H70A	0.9900
C17—C18	1.54 (2)	C70—H70B	0.9900
C18—H18A	0.9900	C71—H71A	0.9900
C18—H18B	0.9900	C71—H71B	0.9900
C21—H21A	0.9900	C71—C72	1.58 (3)
C21—H21B	0.9900	C72—H72A	0.9900
C21—C22	1.55 (2)	C72—H72B	0.9900
C22—H22A	0.9900	O29—C73	1.397 (16)
C22—H22B	0.9900	O29—C76	1.382 (14)
O11—C37	1.44 (3)	O30—C74	1.401 (15)
O11—C40	1.38 (2)	O30—C75	1.446 (16)
O12—C38	1.379 (16)	C73—H73A	0.9900
O12—C39	1.493 (18)	C73—H73B	0.9900
C37—H37A	0.9900	C73—C74	1.463 (17)
C37—H37B	0.9900	C74—H74A	0.9900
C37—C38	1.52 (2)	C74—H74B	0.9900
C38—H38A	0.9900	C75—H75A	0.9900
C38—H38B	0.9900	C75—H75B	0.9900
C39—H39A	0.9900	C75—C76	1.482 (15)

C39—H39B	0.9900	C76—H76A	0.9900
C39—C40	1.50 (2)	C76—H76B	0.9900
C40—H40A	0.9900		

O1—Cu1—N1	96.1 (3)	C40—C39—H39A	110.4
O3—Cu1—O1	167.5 (3)	C40—C39—H39B	110.4
O3—Cu1—O5	88.9 (3)	O11—C40—C39	110.6 (15)
O3—Cu1—O8	89.2 (3)	O11—C40—H40A	109.5
O3—Cu1—N1	96.4 (3)	O11—C40—H40B	109.5
O5—Cu1—O1	89.4 (3)	C39—C40—H40A	109.5
O5—Cu1—N1	96.9 (3)	C39—C40—H40B	109.5
O8—Cu1—O1	90.1 (3)	H40A—C40—H40B	108.1
O8—Cu1—O5	169.2 (3)	C44—O13—C41	107.4 (18)
O8—Cu1—N1	93.9 (2)	C43—O14—C42	109 (2)
O2—Cu2—O6	89.2 (3)	O13—C41—H41A	108.2
O2—Cu2—N3	98.2 (3)	O13—C41—H41B	108.2
O4—Cu2—O2	168.6 (3)	O13—C41—C42	116 (2)
O4—Cu2—O6	88.8 (3)	H41A—C41—H41B	107.3
O4—Cu2—N3	93.2 (3)	C42—C41—H41A	108.2
O6—Cu2—N3	95.3 (3)	C42—C41—H41B	108.2
O9—Cu2—O2	90.4 (4)	O14—C42—C41	108 (2)
O9—Cu2—O4	89.1 (4)	O14—C42—H42A	110.0
O9—Cu2—O6	167.6 (3)	O14—C42—H42B	110.0
O9—Cu2—N3	97.1 (3)	C41—C42—H42A	110.0
C1—O1—Cu1	123.9 (7)	C41—C42—H42B	110.0
C1—O2—Cu2	121.4 (7)	H42A—C42—H42B	108.4
C3—O3—Cu1	124.2 (6)	O14—C43—H43A	108.0
C3—O4—Cu2	124.8 (7)	O14—C43—H43B	108.0
C23—O5—Cu1	123.2 (5)	O14—C43—C44	117 (2)
C23—O6—Cu2	122.3 (5)	H43A—C43—H43B	107.2
C27—O7—H7	109.5	C44—C43—H43A	108.0
C30—O8—Cu1	121.5 (5)	C44—C43—H43B	108.0
C30—O9—Cu2	124.8 (5)	O13—C44—C43	119 (2)
C34—O10—H10	109.5	O13—C44—H44A	107.5
C5—N1—Cu1	111.2 (5)	O13—C44—H44B	107.5
C5—N1—C7	108.2 (8)	C43—C44—H44A	107.5
C7—N1—Cu1	110.5 (5)	C43—C44—H44B	107.5
C9—N1—Cu1	109.5 (5)	H44A—C44—H44B	107.0
C9—N1—C5	110.1 (8)	C47—O16—C46	111.7 (11)
C9—N1—C7	107.3 (8)	C45—O15—C48	111.5 (12)
C8—N2—C6	108.7 (8)	O15—C45—H45A	109.8
C10—N2—C6	110.9 (9)	O15—C45—H45B	109.8
C10—N2—C8	107.1 (8)	O15—C45—C46	109.4 (13)
C11—N3—Cu2	113.5 (6)	H45A—C45—H45B	108.2
C11—N3—C15	106.3 (9)	C46—C45—H45A	109.8
C11—N3—C19	111.7 (9)	C46—C45—H45B	109.8
C15—N3—Cu2	111.5 (6)	O16—C46—C45	113.0 (12)
C19—N3—Cu2	109.1 (6)	O16—C46—H46A	109.0

C19—N3—C15	104.4 (8)	O16—C46—H46B	109.0
C13—N3—Cu2	110.9 (10)	C45—C46—H46A	109.0
C17—N3—Cu2	110.2 (10)	C45—C46—H46B	109.0
C17—N3—C13	107.1 (15)	H46A—C46—H46B	107.8
C17—N3—C21	115.3 (16)	O16—C47—H47A	109.0
C21—N3—Cu2	110.6 (10)	O16—C47—H47B	109.0
C21—N3—C13	102.3 (16)	O16—C47—C48	112.9 (12)
C12—N4—C16	106.3 (10)	H47A—C47—H47B	107.8
C12—N4—C20	108.4 (9)	C48—C47—H47A	109.0
C20—N4—C16	108.8 (9)	C48—C47—H47B	109.0
C18—N4—C14	113.2 (19)	O15—C48—C47	109.6 (13)
C18—N4—C22	110.9 (18)	O15—C48—H48A	109.7
C22—N4—C14	98 (2)	O15—C48—H48B	109.7
O1—C1—O2	126.5 (11)	C47—C48—H48A	109.7
O1—C1—C2	117.0 (9)	C47—C48—H48B	109.7
O2—C1—C2	116.5 (9)	H48A—C48—H48B	108.2
C1—C2—H2A	109.5	C49—O17—C52	108.3 (14)
C1—C2—H2B	109.5	C50—O18—C51	107.4 (15)
C1—C2—H2C	109.5	O17—C49—H49A	109.6
H2A—C2—H2B	109.5	O17—C49—H49B	109.6
H2A—C2—H2C	109.5	O17—C49—C50	110.4 (14)
H2B—C2—H2C	109.5	H49A—C49—H49B	108.1
O3—C3—O4	122.7 (10)	C50—C49—H49A	109.6
O3—C3—C4	120.4 (8)	C50—C49—H49B	109.6
O4—C3—C4	116.9 (9)	O18—C50—C49	112.3 (14)
C3—C4—H4A	109.5	O18—C50—H50A	109.2
C3—C4—H4B	109.5	O18—C50—H50B	109.2
C3—C4—H4C	109.5	C49—C50—H50A	109.2
H4A—C4—H4B	109.5	C49—C50—H50B	109.2
H4A—C4—H4C	109.5	H50A—C50—H50B	107.9
H4B—C4—H4C	109.5	O18—C51—H51A	109.8
O5—C23—C24	116.4 (7)	O18—C51—H51B	109.8
O6—C23—O5	125.9 (8)	H51A—C51—H51B	108.2
O6—C23—C24	117.6 (7)	C52—C51—O18	109.6 (14)
C25—C24—C23	120.3 (7)	C52—C51—H51A	109.8
C29—C24—C23	119.8 (7)	C52—C51—H51B	109.8
C29—C24—C25	119.9 (7)	O17—C52—H52A	109.5
C24—C25—H25	120.8	O17—C52—H52B	109.5
C24—C25—C26	118.4 (8)	C51—C52—O17	110.8 (14)
C26—C25—H25	120.8	C51—C52—H52A	109.5
C25—C26—H26	119.0	C51—C52—H52B	109.5
C27—C26—C25	122.0 (8)	H52A—C52—H52B	108.1
C27—C26—H26	119.0	C56—O19—C53	110.6 (16)
O7—C27—C26	119.1 (8)	C55—O20—C54	112.8 (17)
O7—C27—C28	121.4 (8)	O19—C53—H53A	108.5
C26—C27—C28	119.5 (7)	O19—C53—H53B	108.5
C27—C28—H28	120.5	O19—C53—C54	114.9 (18)
C29—C28—C27	118.9 (8)	H53A—C53—H53B	107.5

C29—C28—H28	120.5	C54—C53—H53A	108.5
C24—C29—H29	119.4	C54—C53—H53B	108.5
C28—C29—C24	121.2 (7)	O20—C54—C53	110.8 (17)
C28—C29—H29	119.4	O20—C54—H54A	109.5
O8—C30—C31	116.8 (7)	O20—C54—H54B	109.5
O9—C30—O8	125.1 (7)	C53—C54—H54A	109.5
O9—C30—C31	118.0 (7)	C53—C54—H54B	109.5
C32—C31—C30	119.8 (7)	H54A—C54—H54B	108.1
C32—C31—C36	118.0 (7)	O20—C55—H55A	109.7
C36—C31—C30	122.2 (7)	O20—C55—H55B	109.7
C31—C32—H32	119.5	O20—C55—C56	110.0 (14)
C33—C32—C31	121.0 (8)	H55A—C55—H55B	108.2
C33—C32—H32	119.5	C56—C55—H55A	109.7
C32—C33—H33	120.2	C56—C55—H55B	109.7
C32—C33—C34	119.7 (7)	O19—C56—C55	110.4 (19)
C34—C33—H33	120.2	O19—C56—H56A	109.6
O10—C34—C33	122.7 (7)	O19—C56—H56B	109.6
O10—C34—C35	118.0 (7)	C55—C56—H56A	109.6
C35—C34—C33	119.1 (7)	C55—C56—H56B	109.6
C34—C35—H35	119.8	H56A—C56—H56B	108.1
C36—C35—C34	120.4 (7)	C60—O21—C57	110.2 (11)
C36—C35—H35	119.8	C58—O22—C59	107.0 (10)
C31—C36—H36	119.2	O21—C57—H57A	109.9
C35—C36—C31	121.6 (7)	O21—C57—H57B	109.9
C35—C36—H36	119.2	O21—C57—C58	109.1 (13)
N1—C5—H5A	109.7	H57A—C57—H57B	108.3
N1—C5—H5B	109.7	C58—C57—H57A	109.9
N1—C5—C6	110.0 (7)	C58—C57—H57B	109.9
H5A—C5—H5B	108.2	O22—C58—C57	113.9 (10)
C6—C5—H5A	109.7	O22—C58—H58A	108.8
C6—C5—H5B	109.7	O22—C58—H58B	108.8
N2—C6—C5	109.2 (7)	C57—C58—H58A	108.8
N2—C6—H6A	109.8	C57—C58—H58B	108.8
N2—C6—H6B	109.8	H58A—C58—H58B	107.7
C5—C6—H6A	109.8	O22—C59—H59A	108.7
C5—C6—H6B	109.8	O22—C59—H59B	108.7
H6A—C6—H6B	108.3	O22—C59—C60	114.4 (14)
N1—C7—H7A	109.6	H59A—C59—H59B	107.6
N1—C7—H7B	109.6	C60—C59—H59A	108.7
N1—C7—C8	110.2 (8)	C60—C59—H59B	108.7
H7A—C7—H7B	108.1	O21—C60—C59	110.8 (12)
C8—C7—H7A	109.6	O21—C60—H60A	109.5
C8—C7—H7B	109.6	O21—C60—H60B	109.5
N2—C8—C7	109.8 (9)	C59—C60—H60A	109.5
N2—C8—H8A	109.7	C59—C60—H60B	109.5
N2—C8—H8B	109.7	H60A—C60—H60B	108.1
C7—C8—H8A	109.7	C64—O23—C61	106.7 (17)
C7—C8—H8B	109.7	C62—O24—C63	111.5 (13)

H8A—C8—H8B	108.2	O23—C61—H61A	109.2
N1—C9—H9A	109.3	O23—C61—H61B	109.2
N1—C9—H9B	109.3	O23—C61—C62	112.0 (19)
N1—C9—C10	111.8 (8)	H61A—C61—H61B	107.9
H9A—C9—H9B	107.9	C62—C61—H61A	109.2
C10—C9—H9A	109.3	C62—C61—H61B	109.2
C10—C9—H9B	109.3	O24—C62—C61	107.4 (18)
N2—C10—C9	109.7 (8)	O24—C62—H62A	110.2
N2—C10—H10A	109.7	O24—C62—H62B	110.2
N2—C10—H10B	109.7	C61—C62—H62A	110.2
C9—C10—H10A	109.7	C61—C62—H62B	110.2
C9—C10—H10B	109.7	H62A—C62—H62B	108.5
H10A—C10—H10B	108.2	O24—C63—H63A	109.7
N3—C11—H11A	109.5	O24—C63—H63B	109.7
N3—C11—H11B	109.5	O24—C63—C64	109.9 (16)
N3—C11—C12	110.8 (10)	H63A—C63—H63B	108.2
H11A—C11—H11B	108.1	C64—C63—H63A	109.7
C12—C11—H11A	109.5	C64—C63—H63B	109.7
C12—C11—H11B	109.5	O23—C64—C63	111.9 (17)
N4—C12—C11	112.1 (10)	O23—C64—H64A	109.2
N4—C12—H12A	109.2	O23—C64—H64B	109.2
N4—C12—H12B	109.2	C63—C64—H64A	109.2
C11—C12—H12A	109.2	C63—C64—H64B	109.2
C11—C12—H12B	109.2	H64A—C64—H64B	107.9
H12A—C12—H12B	107.9	C65—O25—C68	106.9 (14)
N3—C15—H15A	109.8	C67—O26—C66	108.4 (17)
N3—C15—H15B	109.8	O25—C65—H65A	110.1
N3—C15—C16	109.3 (10)	O25—C65—H65B	110.1
H15A—C15—H15B	108.3	O25—C65—C66	108.1 (16)
C16—C15—H15A	109.8	H65A—C65—H65B	108.4
C16—C15—H15B	109.8	C66—C65—H65A	110.1
N4—C16—C15	110.8 (9)	C66—C65—H65B	110.1
N4—C16—H16A	109.5	O26—C66—C65	104.6 (17)
N4—C16—H16B	109.5	O26—C66—H66A	110.8
C15—C16—H16A	109.5	O26—C66—H66B	110.8
C15—C16—H16B	109.5	C65—C66—H66A	110.8
H16A—C16—H16B	108.1	C65—C66—H66B	110.8
N3—C19—H19A	109.8	H66A—C66—H66B	108.9
N3—C19—H19B	109.8	O26—C67—H67A	109.1
N3—C19—C20	109.4 (9)	O26—C67—H67B	109.1
H19A—C19—H19B	108.2	O26—C67—C68	113 (2)
C20—C19—H19A	109.8	H67A—C67—H67B	107.8
C20—C19—H19B	109.8	C68—C67—H67A	109.1
N4—C20—C19	111.3 (10)	C68—C67—H67B	109.1
N4—C20—H20A	109.4	O25—C68—H68A	110.1
N4—C20—H20B	109.4	O25—C68—H68B	110.1
C19—C20—H20A	109.4	C67—C68—O25	108.1 (14)
C19—C20—H20B	109.4	C67—C68—H68A	110.1

H20A—C20—H20B	108.0	C67—C68—H68B	110.1
N3—C13—H13A	110.4	H68A—C68—H68B	108.4
N3—C13—H13B	110.4	C72—O27—C69	110.5 (13)
N3—C13—C14	106.6 (17)	C70—O28—C71	112.7 (12)
H13A—C13—H13B	108.6	O27—C69—H69A	109.6
C14—C13—H13A	110.4	O27—C69—H69B	109.6
C14—C13—H13B	110.4	O27—C69—C70	110.3 (18)
N4—C14—C13	111.5 (17)	H69A—C69—H69B	108.1
N4—C14—H14A	109.3	C70—C69—H69A	109.6
N4—C14—H14B	109.3	C70—C69—H69B	109.6
C13—C14—H14A	109.3	O28—C70—C69	107.1 (14)
C13—C14—H14B	109.3	O28—C70—H70A	110.3
H14A—C14—H14B	108.0	O28—C70—H70B	110.3
N3—C17—H17A	109.3	C69—C70—H70A	110.3
N3—C17—H17B	109.3	C69—C70—H70B	110.3
N3—C17—C18	111.4 (16)	H70A—C70—H70B	108.5
H17A—C17—H17B	108.0	O28—C71—H71A	110.2
C18—C17—H17A	109.3	O28—C71—H71B	110.2
C18—C17—H17B	109.3	O28—C71—C72	107.5 (14)
N4—C18—C17	111.3 (15)	H71A—C71—H71B	108.5
N4—C18—H18A	109.4	C72—C71—H71A	110.2
N4—C18—H18B	109.4	C72—C71—H71B	110.2
C17—C18—H18A	109.4	O27—C72—C71	111.5 (13)
C17—C18—H18B	109.4	O27—C72—H72A	109.3
H18A—C18—H18B	108.0	O27—C72—H72B	109.3
N3—C21—H21A	109.9	C71—C72—H72A	109.3
N3—C21—H21B	109.9	C71—C72—H72B	109.3
N3—C21—C22	109.1 (16)	H72A—C72—H72B	108.0
H21A—C21—H21B	108.3	C76—O29—C73	107.8 (9)
C22—C21—H21A	109.9	C74—O30—C75	110.4 (10)
C22—C21—H21B	109.9	O29—C73—H73A	108.5
N4—C22—C21	111.9 (16)	O29—C73—H73B	108.5
N4—C22—H22A	109.2	O29—C73—C74	115.1 (11)
N4—C22—H22B	109.2	H73A—C73—H73B	107.5
C21—C22—H22A	109.2	C74—C73—H73A	108.5
C21—C22—H22B	109.2	C74—C73—H73B	108.5
H22A—C22—H22B	107.9	O30—C74—C73	111.0 (10)
C40—O11—C37	107.3 (11)	O30—C74—H74A	109.4
C38—O12—C39	109.7 (12)	O30—C74—H74B	109.4
O11—C37—H37A	109.5	C73—C74—H74A	109.4
O11—C37—H37B	109.5	C73—C74—H74B	109.4
O11—C37—C38	110.9 (14)	H74A—C74—H74B	108.0
H37A—C37—H37B	108.1	O30—C75—H75A	110.0
C38—C37—H37A	109.5	O30—C75—H75B	110.0
C38—C37—H37B	109.5	O30—C75—C76	108.4 (9)
O12—C38—C37	110.1 (13)	H75A—C75—H75B	108.4
O12—C38—H38A	109.6	C76—C75—H75A	110.0
O12—C38—H38B	109.6	C76—C75—H75B	110.0

C37—C38—H38A	109.6	O29—C76—C75	115.1 (10)
C37—C38—H38B	109.6	O29—C76—H76A	108.5
H38A—C38—H38B	108.2	O29—C76—H76B	108.5
O12—C39—H39A	110.4	C75—C76—H76A	108.5
O12—C39—H39B	110.4	C75—C76—H76B	108.5
O12—C39—C40	106.7 (11)	H76A—C76—H76B	107.5
H39A—C39—H39B	108.6		

Cu1—O1—C1—O2	1.7 (13)	C15—N3—C19—C20	69.3 (11)
Cu1—O1—C1—C2	−178.0 (6)	C16—N4—C12—C11	66.3 (14)
Cu1—O3—C3—O4	3.4 (12)	C16—N4—C20—C19	−49.5 (12)
Cu1—O3—C3—C4	−177.2 (6)	C19—N3—C11—C12	60.5 (14)
Cu1—O5—C23—O6	−0.1 (17)	C19—N3—C15—C16	−55.0 (12)
Cu1—O5—C23—C24	−177.5 (7)	C20—N4—C12—C11	−50.5 (15)
Cu1—O8—C30—O9	5.1 (14)	C20—N4—C16—C15	63.0 (13)
Cu1—O8—C30—C31	−176.4 (6)	C13—N3—C17—C18	58 (2)
Cu1—N1—C5—C6	176.1 (8)	C13—N3—C21—C22	−71 (3)
Cu1—N1—C7—C8	179.0 (7)	C14—N4—C18—C17	−56 (3)
Cu1—N1—C9—C10	175.2 (8)	C14—N4—C22—C21	55 (3)
Cu2—O2—C1—O1	1.7 (13)	C17—N3—C13—C14	−69 (3)
Cu2—O2—C1—C2	−178.5 (6)	C17—N3—C21—C22	44 (3)
Cu2—O4—C3—O3	0.9 (12)	C18—N4—C14—C13	44 (3)
Cu2—O4—C3—C4	−178.5 (6)	C18—N4—C22—C21	−63 (3)
Cu2—O6—C23—O5	4.2 (17)	C21—N3—C13—C14	53 (3)
Cu2—O6—C23—C24	−178.5 (7)	C21—N3—C17—C18	−55 (3)
Cu2—O9—C30—O8	−1.4 (15)	C22—N4—C14—C13	−73 (3)
Cu2—O9—C30—C31	−179.9 (7)	C22—N4—C18—C17	52 (3)
Cu2—N3—C11—C12	−175.7 (9)	O11—C37—C38—O12	58.1 (18)
Cu2—N3—C15—C16	−172.7 (8)	O12—C39—C40—O11	−64.1 (16)
Cu2—N3—C19—C20	−171.4 (8)	C37—O11—C40—C39	64.1 (17)
Cu2—N3—C13—C14	171 (2)	C38—O12—C39—C40	60.2 (16)
Cu2—N3—C17—C18	178.6 (17)	C39—O12—C38—C37	−57.7 (18)
Cu2—N3—C21—C22	170.3 (19)	C40—O11—C37—C38	−59.5 (17)
O5—C23—C24—C25	172.2 (10)	O13—C41—C42—O14	−64 (3)
O5—C23—C24—C29	−5.5 (15)	O14—C43—C44—O13	35 (5)
O6—C23—C24—C25	−5.4 (15)	C41—O13—C44—C43	−31 (4)
O6—C23—C24—C29	176.9 (10)	C42—O14—C43—C44	−49 (4)
O7—C27—C28—C29	−177.7 (10)	C43—O14—C42—C41	61 (3)
O8—C30—C31—C32	−176.3 (9)	C44—O13—C41—C42	46 (3)
O8—C30—C31—C36	4.8 (14)	O16—C47—C48—O15	54.8 (19)
O9—C30—C31—C32	2.3 (14)	O15—C45—C46—O16	−54 (2)
O9—C30—C31—C36	−176.6 (10)	C45—O15—C48—C47	−60.3 (19)
O10—C34—C35—C36	179.8 (9)	C46—O16—C47—C48	−49.6 (18)
N1—C5—C6—N2	5.1 (15)	C47—O16—C46—C45	49.6 (18)
N1—C7—C8—N2	5.0 (11)	C48—O15—C45—C46	60.1 (19)
N1—C9—C10—N2	6.7 (14)	O17—C49—C50—O18	−56 (2)
N3—C11—C12—N4	−10.6 (17)	O18—C51—C52—O17	64 (2)
N3—C15—C16—N4	−8.9 (14)	C49—O17—C52—C51	−61.3 (19)

N3—C19—C20—N4	−16.8 (14)	C50—O18—C51—C52	−60 (2)
N3—C13—C14—N4	18 (4)	C51—O18—C50—C49	57 (2)
N3—C17—C18—N4	4 (3)	C52—O17—C49—C50	56.0 (16)
N3—C21—C22—N4	13 (3)	O19—C53—C54—O20	51 (3)
C23—C24—C25—C26	179.5 (10)	O20—C55—C56—O19	−57.9 (19)
C23—C24—C29—C28	−178.6 (10)	C53—O19—C56—C55	54 (2)
C24—C25—C26—C27	1.9 (17)	C54—O20—C55—C56	58 (2)
C25—C24—C29—C28	3.7 (16)	C55—O20—C54—C53	−53 (3)
C25—C26—C27—O7	178.5 (10)	C56—O19—C53—C54	−52 (3)
C25—C26—C27—C28	−1.9 (17)	O21—C57—C58—O22	−56.1 (15)
C26—C27—C28—C29	2.7 (16)	O22—C59—C60—O21	59 (2)
C27—C28—C29—C24	−3.6 (16)	C57—O21—C60—C59	−56 (2)
C29—C24—C25—C26	−2.8 (16)	C58—O22—C59—C60	−57.3 (16)
C30—C31—C32—C33	−175.8 (9)	C59—O22—C58—C57	55.2 (14)
C30—C31—C36—C35	177.2 (9)	C60—O21—C57—C58	53.9 (19)
C31—C32—C33—C34	−0.8 (15)	O23—C61—C62—O24	63 (3)
C32—C31—C36—C35	−1.7 (15)	O24—C63—C64—O23	−56 (2)
C32—C33—C34—O10	−178.1 (10)	C61—O23—C64—C63	58 (2)
C32—C33—C34—C35	−3.1 (15)	C62—O24—C63—C64	56 (2)
C33—C34—C35—C36	4.5 (15)	C63—O24—C62—C61	−58 (2)
C34—C35—C36—C31	−2.1 (15)	C64—O23—C61—C62	−62 (3)
C36—C31—C32—C33	3.2 (15)	O25—C65—C66—O26	−66 (2)
C5—N1—C7—C8	57.1 (10)	O26—C67—C68—O25	61 (3)
C5—N1—C9—C10	−62.2 (12)	C65—O25—C68—C67	−62 (2)
C6—N2—C8—C7	−62.9 (11)	C66—O26—C67—C68	−61 (3)
C6—N2—C10—C9	54.2 (12)	C67—O26—C66—C65	61 (3)
C7—N1—C5—C6	−62.4 (12)	C68—O25—C65—C66	67 (2)
C7—N1—C9—C10	55.3 (11)	O27—C69—C70—O28	61 (2)
C8—N2—C6—C5	57.2 (13)	O28—C71—C72—O27	−54.6 (15)
C8—N2—C10—C9	−64.3 (11)	C69—O27—C72—C71	59 (2)
C9—N1—C5—C6	54.6 (13)	C70—O28—C71—C72	56.9 (19)
C9—N1—C7—C8	−61.7 (10)	C71—O28—C70—C69	−60 (2)
C10—N2—C6—C5	−60.3 (13)	C72—O27—C69—C70	−61.5 (19)
C10—N2—C8—C7	56.9 (10)	O29—C73—C74—O30	−55.5 (15)
C11—N3—C15—C16	63.2 (12)	O30—C75—C76—O29	57.5 (14)
C11—N3—C19—C20	−45.2 (13)	C73—O29—C76—C75	−55.2 (13)
C12—N4—C16—C15	−53.6 (13)	C74—O30—C75—C76	−55.2 (13)
C12—N4—C20—C19	65.7 (13)	C75—O30—C74—C73	54.9 (14)
C15—N3—C11—C12	−52.8 (13)	C76—O29—C73—C74	53.4 (12)

1-Aza-4-azoniabicyclo[2.2.2]octane 4-hydroxybenzoate (compound_12)

Crystal data

$C_6H_{13}N_2^+·C_7H_5O_3^-$
M_r = 250.29
Monoclinic, $P2_1/n$
a = 15.3994 (5) Å
b = 10.6592 (3) Å
c = 16.9261 (6) Å
β = 110.424 (4)°
V = 2603.67 (15) Å^3
Z = 8
$F(000)$ = 1072
D_x = 1.277 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å

Cell parameters from 3137 reflections
θ = 4.8–76.7°
μ = 0.75 mm^{-1}

T = 130 K
Irregular, clear colourless
0.22 × 0.16 × 0.13 mm

Data collection

Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.2273 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2023)

T_{min} = 0.952, T_{max} = 1.000
11079 measured reflections
5386 independent reflections
3805 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 76.9°, θ_{min} = 4.8°
h = −19→15
k = −13→11
l = −21→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.120
S = 1.01
5386 reflections
337 parameters
2 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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. Peaks of electron density approximately midway between O1 and N1 and between O4 and N3 were assigned as H atoms and allowed to refine independently.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O3	1.07534 (9)	0.92832 (11)	0.26476 (8)	0.0325 (3)
O6	0.39246 (9)	0.06837 (12)	0.72789 (8)	0.0344 (3)
H6A	0.377242	0.123074	0.756348	0.052*
O1	0.85260 (9)	0.81944 (13)	0.50408 (8)	0.0396 (3)
O4	0.65348 (9)	0.17927 (12)	0.52275 (9)	0.0387 (3)
O5	0.64976 (10)	0.37381 (13)	0.56921 (9)	0.0433 (3)
O2	0.83541 (10)	0.63012 (13)	0.44495 (9)	0.0449 (4)
N2	0.65699 (9)	0.69665 (13)	0.67705 (8)	0.0235 (3)
N4	0.82538 (9)	0.31541 (13)	0.33188 (8)	0.0243 (3)
N3	0.74510 (9)	0.25762 (13)	0.43525 (8)	0.0255 (3)
N1	0.75472 (9)	0.75015 (14)	0.58792 (8)	0.0271 (3)
C18	0.44712 (10)	0.12068 (16)	0.68975 (10)	0.0244 (3)
C16	0.53758 (10)	0.08932 (15)	0.60035 (10)	0.0246 (3)
H16	0.559584	0.034981	0.566996	0.030*
C5	1.02615 (10)	0.87741 (16)	0.30869 (10)	0.0239 (3)
C6	1.00103 (11)	0.95660 (16)	0.36279 (10)	0.0257 (3)

H6	1.019823	1.042010	0.368411	0.031*
C15	0.56429 (10)	0.21443 (15)	0.60853 (9)	0.0228 (3)
C7	0.94857 (10)	0.91040 (16)	0.40832 (10)	0.0242 (3)
H7	0.931289	0.965032	0.444654	0.029*
C3	0.94708 (10)	0.70619 (15)	0.34851 (9)	0.0245 (3)
H3A	0.929034	0.620488	0.343731	0.029*
C4	0.99942 (10)	0.75138 (15)	0.30237 (9)	0.0242 (3)
H4	1.017093	0.696478	0.266421	0.029*
C17	0.47946 (11)	0.04216 (16)	0.63987 (10)	0.0263 (3)
H17	0.461564	−0.043593	0.633079	0.032*
C2	0.92081 (10)	0.78541 (16)	0.40172 (9)	0.0233 (3)
C20	0.53078 (11)	0.29304 (16)	0.65709 (10)	0.0252 (3)
H20	0.547877	0.379110	0.662769	0.030*
C26	0.89072 (10)	0.32690 (18)	0.41950 (10)	0.0295 (4)
H26A	0.913877	0.414185	0.430027	0.035*
H26B	0.944450	0.270862	0.427788	0.035*
C22	0.74505 (11)	0.39917 (16)	0.32048 (10)	0.0263 (3)
H22A	0.700086	0.390449	0.262372	0.032*
H22B	0.766231	0.487474	0.328729	0.032*
C14	0.62704 (11)	0.26345 (16)	0.56480 (10)	0.0262 (3)
C11	0.73355 (11)	0.60533 (16)	0.69291 (10)	0.0269 (3)
H11A	0.707725	0.520356	0.676195	0.032*
H11B	0.770748	0.603757	0.753853	0.032*
C13	0.69696 (11)	0.82297 (15)	0.69902 (10)	0.0265 (3)
H13A	0.739074	0.824194	0.758553	0.032*
H13B	0.646796	0.884498	0.692605	0.032*
C19	0.47265 (11)	0.24713 (16)	0.69734 (10)	0.0263 (3)
H19	0.450186	0.301899	0.730123	0.032*
C9	0.59948 (10)	0.69311 (17)	0.58667 (10)	0.0276 (4)
H9A	0.550405	0.757766	0.574570	0.033*
H9B	0.569188	0.610087	0.572522	0.033*
C1	0.86523 (11)	0.73765 (17)	0.45246 (10)	0.0278 (4)
C8	0.65960 (11)	0.71719 (19)	0.53215 (10)	0.0314 (4)
H8A	0.661187	0.641106	0.499140	0.038*
H8B	0.632892	0.786679	0.492248	0.038*
C24	0.79187 (12)	0.18504 (16)	0.31742 (11)	0.0296 (4)
H24A	0.844859	0.127738	0.325455	0.035*
H24B	0.748538	0.175801	0.258691	0.035*
C10	0.79599 (11)	0.64116 (17)	0.64256 (11)	0.0321 (4)
H10A	0.858764	0.662874	0.681704	0.039*
H10B	0.801488	0.569320	0.607531	0.039*
C21	0.69766 (11)	0.36560 (18)	0.38415 (11)	0.0317 (4)
H21A	0.700207	0.438359	0.421250	0.038*
H21B	0.631754	0.344697	0.353719	0.038*
C25	0.84260 (11)	0.29168 (19)	0.48231 (10)	0.0334 (4)
H25A	0.875059	0.219960	0.517418	0.040*
H25B	0.844925	0.363548	0.520040	0.040*
C12	0.75124 (12)	0.85972 (16)	0.64089 (11)	0.0311 (4)

H12A	0.720265	0.931079	0.604516	0.037*
H12B	0.814915	0.885815	0.675367	0.037*
C23	0.74232 (14)	0.14950 (18)	0.37884 (13)	0.0389 (4)
H23A	0.677167	0.126647	0.346843	0.047*
H23B	0.773184	0.076051	0.412906	0.047*
H4A	0.7006 (19)	0.227 (3)	0.4812 (18)	0.090 (9)*
H1	0.8039 (18)	0.777 (2)	0.5461 (17)	0.082 (9)*
H3	1.101 (2)	0.875 (2)	0.240 (2)	0.123*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0442 (7)	0.0266 (7)	0.0378 (7)	−0.0010 (6)	0.0286 (6)	−0.0001 (5)
O6	0.0462 (7)	0.0299 (7)	0.0401 (7)	−0.0063 (6)	0.0313 (6)	−0.0061 (6)
O1	0.0475 (7)	0.0408 (8)	0.0442 (7)	−0.0145 (6)	0.0335 (7)	−0.0141 (6)
O4	0.0478 (7)	0.0321 (7)	0.0522 (8)	−0.0038 (6)	0.0378 (7)	−0.0018 (6)
O5	0.0606 (8)	0.0353 (8)	0.0462 (8)	−0.0216 (7)	0.0342 (7)	−0.0131 (6)
O2	0.0585 (8)	0.0354 (8)	0.0555 (9)	−0.0181 (7)	0.0383 (7)	−0.0120 (7)
N2	0.0254 (6)	0.0237 (7)	0.0229 (6)	0.0003 (5)	0.0102 (5)	−0.0004 (5)
N4	0.0255 (6)	0.0264 (7)	0.0233 (6)	0.0017 (5)	0.0114 (6)	0.0011 (5)
N3	0.0250 (6)	0.0271 (7)	0.0281 (7)	−0.0004 (6)	0.0139 (6)	0.0011 (6)
N1	0.0256 (6)	0.0324 (8)	0.0271 (7)	−0.0027 (6)	0.0139 (6)	−0.0014 (6)
C18	0.0251 (7)	0.0279 (9)	0.0231 (7)	−0.0011 (6)	0.0118 (6)	0.0015 (6)
C16	0.0259 (7)	0.0261 (8)	0.0252 (7)	0.0018 (6)	0.0132 (6)	−0.0006 (6)
C5	0.0265 (7)	0.0256 (8)	0.0222 (7)	0.0031 (6)	0.0116 (6)	0.0029 (6)
C6	0.0299 (8)	0.0214 (8)	0.0276 (8)	0.0001 (7)	0.0122 (7)	−0.0013 (6)
C15	0.0208 (7)	0.0290 (9)	0.0183 (7)	−0.0011 (6)	0.0065 (6)	0.0008 (6)
C7	0.0241 (7)	0.0273 (9)	0.0217 (7)	0.0023 (6)	0.0085 (6)	−0.0022 (6)
C3	0.0261 (7)	0.0229 (8)	0.0242 (7)	−0.0029 (6)	0.0086 (6)	−0.0036 (6)
C4	0.0279 (7)	0.0233 (8)	0.0232 (7)	0.0017 (7)	0.0114 (6)	−0.0037 (6)
C17	0.0314 (8)	0.0205 (8)	0.0311 (8)	−0.0004 (7)	0.0162 (7)	−0.0001 (7)
C2	0.0206 (6)	0.0286 (9)	0.0209 (7)	−0.0014 (6)	0.0076 (6)	−0.0020 (6)
C20	0.0297 (8)	0.0232 (8)	0.0232 (7)	−0.0035 (7)	0.0097 (7)	−0.0022 (6)
C26	0.0211 (7)	0.0408 (10)	0.0260 (8)	−0.0030 (7)	0.0073 (7)	0.0013 (7)
C22	0.0287 (7)	0.0255 (9)	0.0252 (7)	0.0050 (7)	0.0100 (7)	0.0022 (7)
C14	0.0258 (7)	0.0298 (9)	0.0231 (7)	−0.0051 (7)	0.0088 (6)	−0.0017 (7)
C11	0.0302 (8)	0.0223 (8)	0.0264 (8)	0.0027 (7)	0.0075 (7)	0.0003 (6)
C13	0.0307 (8)	0.0217 (8)	0.0291 (8)	0.0018 (7)	0.0129 (7)	−0.0006 (6)
C19	0.0298 (8)	0.0285 (9)	0.0232 (7)	−0.0010 (7)	0.0126 (7)	−0.0048 (7)
C9	0.0222 (7)	0.0358 (10)	0.0239 (8)	−0.0023 (7)	0.0069 (6)	−0.0002 (7)
C1	0.0264 (7)	0.0316 (9)	0.0274 (8)	−0.0042 (7)	0.0118 (7)	−0.0026 (7)
C8	0.0269 (8)	0.0446 (11)	0.0233 (8)	−0.0009 (8)	0.0094 (7)	0.0004 (7)
C24	0.0323 (8)	0.0277 (9)	0.0320 (8)	0.0028 (7)	0.0155 (7)	−0.0034 (7)
C10	0.0244 (7)	0.0343 (10)	0.0376 (9)	0.0054 (7)	0.0109 (7)	−0.0003 (8)
C21	0.0271 (7)	0.0394 (10)	0.0298 (8)	0.0100 (7)	0.0113 (7)	0.0036 (8)
C25	0.0255 (8)	0.0498 (12)	0.0236 (8)	−0.0012 (8)	0.0069 (7)	0.0055 (8)
C12	0.0358 (8)	0.0258 (9)	0.0363 (9)	−0.0038 (7)	0.0184 (8)	−0.0013 (7)
C23	0.0476 (10)	0.0276 (10)	0.0532 (12)	−0.0090 (8)	0.0325 (10)	−0.0091 (9)

Geometric parameters (Å, º)

O3—C5	1.3470 (18)	C3—C4	1.390 (2)
O3—H3	0.875 (17)	C3—C2	1.394 (2)
O6—H6A	0.8400	C4—H4	0.9500
O6—C18	1.3474 (18)	C17—H17	0.9500
O1—C1	1.296 (2)	C2—C1	1.497 (2)
O1—H1	1.28 (3)	C20—H20	0.9500
O4—C14	1.296 (2)	C20—C19	1.389 (2)
O4—H4A	1.28 (3)	C26—H26A	0.9900
O5—C14	1.222 (2)	C26—H26B	0.9900
O2—C1	1.225 (2)	C26—C25	1.540 (2)
N2—C11	1.479 (2)	C22—H22A	0.9900
N2—C13	1.473 (2)	C22—H22B	0.9900
N2—C9	1.4766 (19)	C22—C21	1.540 (2)
N4—C26	1.478 (2)	C11—H11A	0.9900
N4—C22	1.4824 (19)	C11—H11B	0.9900
N4—C24	1.473 (2)	C11—C10	1.539 (2)
N3—C21	1.472 (2)	C13—H13A	0.9900
N3—C25	1.478 (2)	C13—H13B	0.9900
N3—C23	1.488 (2)	C13—C12	1.547 (2)
N3—H4A	1.25 (3)	C19—H19	0.9500
N1—C8	1.482 (2)	C9—H9A	0.9900
N1—C10	1.483 (2)	C9—H9B	0.9900
N1—C12	1.485 (2)	C9—C8	1.540 (2)
N1—H1	1.24 (3)	C8—H8A	0.9900
C18—C17	1.398 (2)	C8—H8B	0.9900
C18—C19	1.397 (2)	C24—H24A	0.9900
C16—H16	0.9500	C24—H24B	0.9900
C16—C15	1.388 (2)	C24—C23	1.536 (2)
C16—C17	1.385 (2)	C10—H10A	0.9900
C5—C6	1.396 (2)	C10—H10B	0.9900
C5—C4	1.398 (2)	C21—H21A	0.9900
C6—H6	0.9500	C21—H21B	0.9900
C6—C7	1.387 (2)	C25—H25A	0.9900
C15—C20	1.393 (2)	C25—H25B	0.9900
C15—C14	1.501 (2)	C12—H12A	0.9900
C7—H7	0.9500	C12—H12B	0.9900
C7—C2	1.392 (2)	C23—H23A	0.9900
C3—H3A	0.9500	C23—H23B	0.9900
C5—O3—H3	116 (2)	O5—C14—O4	124.17 (15)
C18—O6—H6A	109.5	O5—C14—C15	121.66 (15)
C1—O1—H1	112.8 (12)	N2—C11—H11A	109.6
C14—O4—H4A	111.8 (12)	N2—C11—H11B	109.6
C13—N2—C11	108.62 (12)	N2—C11—C10	110.40 (13)
C13—N2—C9	109.36 (13)	H11A—C11—H11B	108.1
C9—N2—C11	108.85 (12)	C10—C11—H11A	109.6

C26—N4—C22	109.01 (13)	C10—C11—H11B	109.6
C24—N4—C26	108.75 (13)	N2—C13—H13A	109.7
C24—N4—C22	108.58 (13)	N2—C13—H13B	109.7
C21—N3—C25	109.09 (14)	N2—C13—C12	109.98 (13)
C21—N3—C23	109.36 (14)	H13A—C13—H13B	108.2
C21—N3—H4A	107.5 (13)	C12—C13—H13A	109.7
C25—N3—C23	109.41 (14)	C12—C13—H13B	109.7
C25—N3—H4A	113.8 (13)	C18—C19—H19	119.9
C23—N3—H4A	107.5 (12)	C20—C19—C18	120.24 (15)
C8—N1—C10	108.96 (14)	C20—C19—H19	119.9
C8—N1—C12	109.59 (13)	N2—C9—H9A	109.6
C8—N1—H1	110.9 (12)	N2—C9—H9B	109.6
C10—N1—C12	109.74 (13)	N2—C9—C8	110.36 (12)
C10—N1—H1	108.7 (12)	H9A—C9—H9B	108.1
C12—N1—H1	109.0 (12)	C8—C9—H9A	109.6
O6—C18—C17	117.09 (15)	C8—C9—H9B	109.6
O6—C18—C19	123.92 (15)	O1—C1—C2	114.09 (15)
C19—C18—C17	119.00 (14)	O2—C1—O1	124.09 (15)
C15—C16—H16	119.4	O2—C1—C2	121.82 (15)
C17—C16—H16	119.4	N1—C8—C9	108.98 (13)
C17—C16—C15	121.29 (15)	N1—C8—H8A	109.9
O3—C5—C6	117.17 (15)	N1—C8—H8B	109.9
O3—C5—C4	123.51 (14)	C9—C8—H8A	109.9
C6—C5—C4	119.31 (14)	C9—C8—H8B	109.9
C5—C6—H6	120.1	H8A—C8—H8B	108.3
C7—C6—C5	119.90 (15)	N4—C24—H24A	109.6
C7—C6—H6	120.1	N4—C24—H24B	109.6
C16—C15—C20	118.61 (14)	N4—C24—C23	110.37 (13)
C16—C15—C14	120.30 (14)	H24A—C24—H24B	108.1
C20—C15—C14	121.09 (15)	C23—C24—H24A	109.6
C6—C7—H7	119.4	C23—C24—H24B	109.6
C6—C7—C2	121.17 (15)	N1—C10—C11	108.97 (12)
C2—C7—H7	119.4	N1—C10—H10A	109.9
C4—C3—H3A	119.7	N1—C10—H10B	109.9
C4—C3—C2	120.61 (15)	C11—C10—H10A	109.9
C2—C3—H3A	119.7	C11—C10—H10B	109.9
C5—C4—H4	119.9	H10A—C10—H10B	108.3
C3—C4—C5	120.21 (14)	N3—C21—C22	109.50 (13)
C3—C4—H4	119.9	N3—C21—H21A	109.8
C18—C17—H17	120.0	N3—C21—H21B	109.8
C16—C17—C18	120.05 (15)	C22—C21—H21A	109.8
C16—C17—H17	120.0	C22—C21—H21B	109.8
C7—C2—C3	118.79 (14)	H21A—C21—H21B	108.2
C7—C2—C1	120.03 (14)	N3—C25—C26	109.32 (13)
C3—C2—C1	121.17 (15)	N3—C25—H25A	109.8
C15—C20—H20	119.6	N3—C25—H25B	109.8
C19—C20—C15	120.81 (15)	C26—C25—H25A	109.8
C19—C20—H20	119.6	C26—C25—H25B	109.8

N4—C26—H26A	109.6	H25A—C25—H25B	108.3
N4—C26—H26B	109.6	N1—C12—C13	109.03 (13)
N4—C26—C25	110.44 (13)	N1—C12—H12A	109.9
H26A—C26—H26B	108.1	N1—C12—H12B	109.9
C25—C26—H26A	109.6	C13—C12—H12A	109.9
C25—C26—H26B	109.6	C13—C12—H12B	109.9
N4—C22—H22A	109.6	H12A—C12—H12B	108.3
N4—C22—H22B	109.6	N3—C23—C24	109.48 (14)
N4—C22—C21	110.28 (13)	N3—C23—H23A	109.8
H22A—C22—H22B	108.1	N3—C23—H23B	109.8
C21—C22—H22A	109.6	C24—C23—H23A	109.8
C21—C22—H22B	109.6	C24—C23—H23B	109.8
O4—C14—C15	114.17 (15)	H23A—C23—H23B	108.2

O3—C5—C6—C7	178.56 (14)	C2—C3—C4—C5	−0.2 (2)
O3—C5—C4—C3	−178.71 (14)	C20—C15—C14—O4	179.26 (15)
O6—C18—C17—C16	−178.61 (14)	C20—C15—C14—O5	−1.1 (2)
O6—C18—C19—C20	178.79 (15)	C26—N4—C22—C21	−57.79 (17)
N2—C11—C10—N1	−4.97 (19)	C26—N4—C24—C23	59.89 (17)
N2—C13—C12—N1	−6.37 (18)	C22—N4—C26—C25	58.89 (18)
N2—C9—C8—N1	−6.1 (2)	C22—N4—C24—C23	−58.59 (18)
N4—C26—C25—N3	−0.3 (2)	C14—C15—C20—C19	179.93 (14)
N4—C22—C21—N3	−1.77 (19)	C11—N2—C13—C12	63.29 (16)
N4—C24—C23—N3	−1.0 (2)	C11—N2—C9—C8	−55.57 (18)
C16—C15—C20—C19	0.7 (2)	C13—N2—C11—C10	−56.90 (16)
C16—C15—C14—O4	−1.5 (2)	C13—N2—C9—C8	62.96 (17)
C16—C15—C14—O5	178.14 (16)	C19—C18—C17—C16	1.4 (2)
C5—C6—C7—C2	0.5 (2)	C9—N2—C11—C10	62.09 (17)
C6—C5—C4—C3	1.0 (2)	C9—N2—C13—C12	−55.37 (16)
C6—C7—C2—C3	0.3 (2)	C8—N1—C10—C11	−57.43 (17)
C6—C7—C2—C1	179.22 (14)	C8—N1—C12—C13	63.48 (17)
C15—C16—C17—C18	−0.5 (2)	C24—N4—C26—C25	−59.32 (18)
C15—C20—C19—C18	0.2 (2)	C24—N4—C22—C21	60.53 (17)
C7—C2—C1—O1	−4.3 (2)	C10—N1—C8—C9	63.89 (18)
C7—C2—C1—O2	176.04 (16)	C10—N1—C12—C13	−56.12 (17)
C3—C2—C1—O1	174.60 (15)	C21—N3—C25—C26	−60.07 (19)
C3—C2—C1—O2	−5.1 (3)	C21—N3—C23—C24	60.41 (18)
C4—C5—C6—C7	−1.2 (2)	C25—N3—C21—C22	61.18 (17)
C4—C3—C2—C7	−0.5 (2)	C25—N3—C23—C24	−59.03 (19)
C4—C3—C2—C1	−179.37 (14)	C12—N1—C8—C9	−56.19 (18)
C17—C18—C19—C20	−1.2 (2)	C12—N1—C10—C11	62.56 (17)
C17—C16—C15—C20	−0.5 (2)	C23—N3—C21—C22	−58.45 (17)
C17—C16—C15—C14	−179.76 (14)	C23—N3—C25—C26	59.53 (19)