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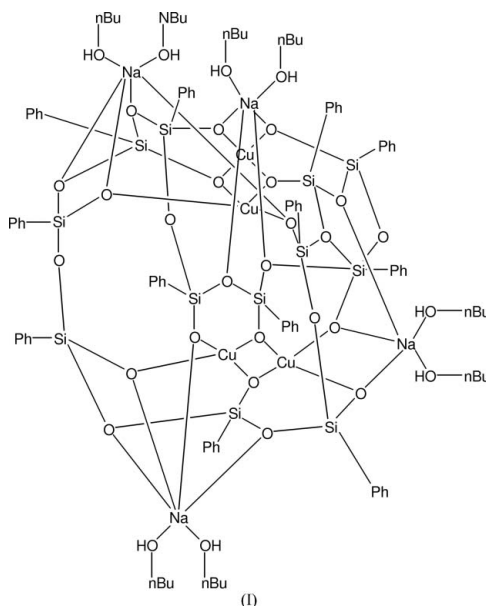
Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$
 R factor = 0.046
 wR factor = 0.133
Data-to-parameter ratio = 15.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Octabutanol(dodecaoxidododecaphenyl[18.4.0]-
dodecasiloxane)tetracopper(II)tetrasodium(I)Molecules of the title compound, $[\text{Cu}_4\text{Na}_4(\text{C}_6\text{H}_5\text{O}_2\text{Si})_{12}(\text{C}_4\text{H}_{10}\text{O})_8]$, are located on special positions of site symmetry $\bar{4}$. The Cu atoms are coordinated in a square planar fashion. The Na atoms, on the other hand, show an irregular sixfold coordination mode. The molecular conformation is stabilized by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

Copper(II)-polyorganosiloxanolate clusters are starting compounds for the synthesis of molecular-based magnets, due to magnetic coupling of the oxygen-bridged Cu atoms in the rigid siloxanolate framework. The title compound, (I), shows antiferromagnetic coupling (Rentschler *et al.*, 1996) of the four Cu centres and it is intended to link these metallapolysiloxanolate units with suitable ligands in order to obtain magnetic materials.

The structure of compound (I) has already been reported by Igonin *et al.* (1991), but those workers did not publish any coordinates. Thus, no comparison between the two structures can be made. The molecule of (I) is located on a special position of site symmetry $\bar{4}$. As a result, there is just one quarter of a molecule in the asymmetric unit. The Cu atoms are coordinated by four O atoms in a square planar fashion, forming Cu_2O_2 four-membered rings. The Na atoms are bonded to four siloxanolate O atoms and two butanol O atoms. The molecules form a cage composed of Cu, O, Si and Na atoms. The structure is stabilized by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

Experimental

To a mixture of butanol (100 ml), NaOH (1.24 g, 30.86 mmol) and water (0.56 g, 30.86 mmol), phenyltributoxysilane (10 g, 30.86 mmol) was added and the resulting solution was heated under reflux for 1 h. A solution of anhydrous CuCl₂ (1.39 g, 10.29 mmol) in butanol (12 ml) was added dropwise to the refluxing mixture over a period of 25 min. The resulting deep-blue suspension was refluxed for an additional 15 min, filtered to remove NaCl, and evaporated to half its volume using a rotary evaporator. The resulting blue crystals of (I) were filtered off and washed with butanol (yield 2.59 g, 39%).

Crystal data

[Cu₄Na₄(C₆H₅O₂Si)₁₂(C₄H₁₀O)₈] Mo K α radiation
M_r = 2585.36 Cell parameters from 30540 reflections
 Tetragonal, I4₁/a θ = 2.0–25.3°
a = 31.3142 (14) Å μ = 0.88 mm⁻¹
c = 12.6792 (7) Å *T* = 173 (2) K
V = 12433.0 (10) Å³ Block, blue
Z = 4 0.51 × 0.22 × 0.18 mm
D_x = 1.381 Mg m⁻³

Data collection

Stoe IPDS-II two-circle diffractometer 5642 independent reflections
 ω scans 4291 reflections with *I* > 2σ(*I*)
 Absorption correction: multi-scan *R*_{int} = 0.061
 (MULABS; Spek, 2003; Blessing, 1995) θ_{max} = 25.3°
*T*_{min} = 0.664, *T*_{max} = 0.858 *h* = -37 → 35
 41700 measured reflections *k* = -30 → 37
l = -15 → 15

Refinement

Refinement on *F*² H-atom parameters constrained
R [*F*² > 2σ(*F*²)] = 0.046 *w* = 1/[σ²(*F*_o²) + (0.0928*P*)²]
wR(*F*²) = 0.133 where *P* = (*F*_o² + 2*F*_c²)/3
S = 1.01 (Δ/*σ*)_{max} = 0.001
 5642 reflections Δ*ρ*_{max} = 1.35 e Å⁻³
 354 parameters Δ*ρ*_{min} = -0.49 e Å⁻³

Table 1

Selected bond lengths (Å).

Cu1—O3	1.903 (2)	O3—Na1	2.307 (3)
Cu1—O2	1.910 (2)	Na1—O51	2.239 (5)
Cu1—O1	1.949 (2)	Na1—O41	2.374 (4)
Cu1—O1 ⁱ	1.949 (2)	Na1—O11 ⁱⁱ	2.713 (3)
O2—Na1	2.444 (3)	Na1—O12 ⁱⁱ	2.856 (3)

Symmetry codes: (i) -*x* + 1, -*y* + ½, *z*; (ii) -*y* + ¾, *x* - ¼, -*z* + ¾.

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O41—H41...O2	0.84	2.10	2.848 (4)	148
O51—H51...O12 ⁱⁱ	0.84	2.08	2.857 (5)	154

Symmetry code: (ii) -*y* + ¾, *x* - ¼, -*z* + ¾.

H atoms were located in a difference Fourier synthesis, but they were positioned geometrically and refined as riding, with *U*_{iso}(H) = 1.2*U*_{eq}(C,O) or 1.5*U*_{eq}(C_{methyl}) and with O—H = 0.84 Å and C—H in the range 0.95–0.99 Å. In addition, the torsion angles about the C—O bonds of the hydroxyl groups were refined. The C—C bonds in the butanol molecules were refined with a distance restraint of 1.50 (1) Å. The highest peak is located 1.04 Å from atom Na1.

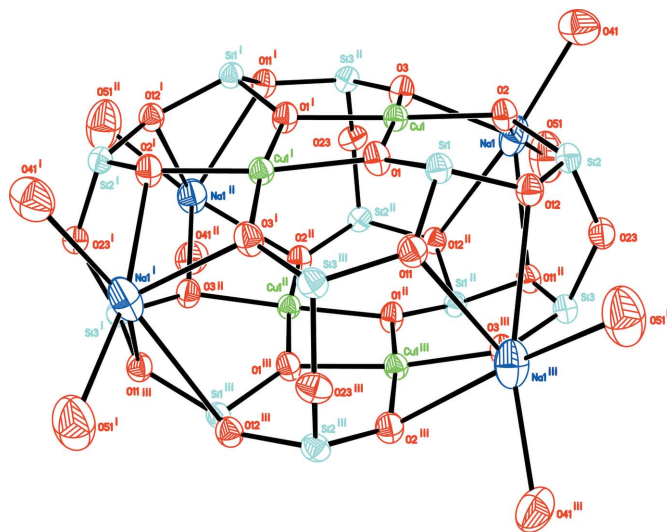


Figure 1

A perspective view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. C and H atoms have been omitted for clarity. [Symmetry codes: (i) -*x* + 1, -*y* + ½, *z*; (ii) -*y* + ¾, *x* - ¼, -*z* + ¾; (iii) *y* + ¼, -*x* + ¾, -*z* + ¾.]

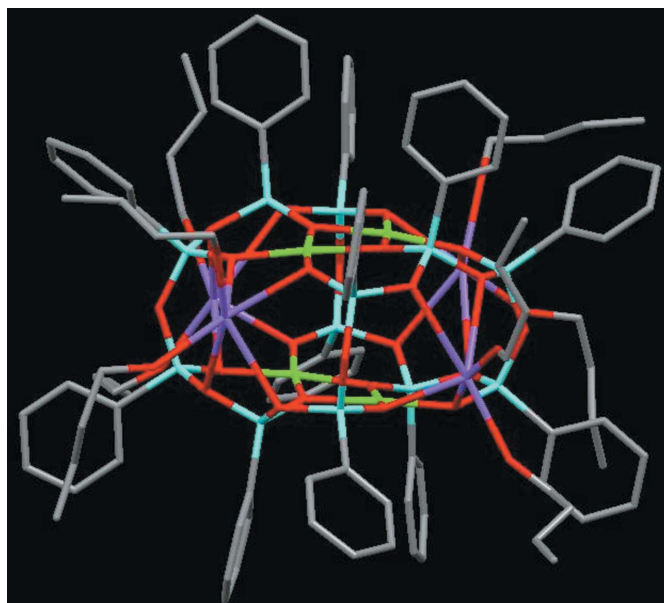


Figure 2

A perspective view of the structure of (I). H atoms have been omitted for clarity.

Data collection: X-AREA (Stoe & Cie, 2001); cell refinement: X-AREA; data reduction: X-RED (Stoe & Cie, 2001); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP in SHELXTL-Plus (Sheldrick, 1991) and MERCURY (Bruno *et al.*, 2002); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2003).

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