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Dimeric copper(II) trimethylsilylacetate adducts with pyridine, 2-methylpyridine, 3-methylpyridine and quinoline, and a polymeric copper(II) trimethylsilylacetate

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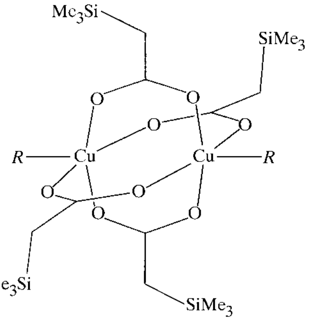
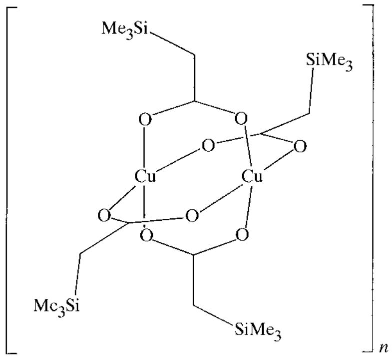
In the crystals of bis(pyridine-*N*)tetrakis(μ-trimethylsilylacetato-*O*:*O*′)dicopper(II), $[Cu_2(C_5H_{11}O_2Si)_4(C_5H_5N)_2]$, (I), the dinuclear Cu^{II} complexes have cage structures with Cu···Cu distances of 2.632 (1) and 2.635 (1) Å. In the crystals of bis(2-methylpyridine-*N*)tetrakis(μ-trimethylsilylacetato-*O*:*O*′)dicopper(II), $[Cu_2(C_5H_{11}O_2Si)_4(C_6H_7N)_2]$, (II), bis(3-methylpyridine-*N*)tetrakis(μ-trimethylsilylacetato-*O*:*O*′)dicopper(II), $[Cu_2(C_5H_{11}O_2Si)_4(C_6H_7N)_2]$, (III), and bis(quinoline-*N*)(μ-trimethylsilylacetato-*O*:*O*′)dicopper(II), $[Cu_2(C_5H_{11}O_2Si)_4(C_9H_7N)_2]$, (IV), the centrosymmetric dinuclear Cu^{II} complexes have a cage structure with Cu···Cu distances of 2.664 (1), 2.638 (3) and 2.665 (1) Å, respectively. In the crystals of *catena*-poly[tetrakis(μ-trimethylsilylacetato-*O*:*O*′)dicopper(II)], $[Cu_2(C_5H_{11}O_2Si)_4]_n$, (V), the dinuclear Cu^{II} units of a cage structure are linked by the cyclic Cu—O bonds at the apical positions to form a linear chain by use of a glide translation.

Comment

The $-2J$ values of copper(II) carboxylates, *i.e.* the singlet–triplet energy separation, greatly depend on the kind of atom directly bonded to the COO bridge (Kato & Muto, 1988). There is a correlation between the electron density at the central C atom of the COO bridge and the antiferromagnetic spin-exchange interaction (Yamanaka *et al.*, 1991; Harada *et al.*, 1997). Silanecarboxylatocopper complexes show very large $-2J$ values due to the σ-electron donating character of the Si atom; for $[Cu_2(PhMe_2SiCOO)_4(H_2O)_2]$, $-2J = 1000 \pm 150$ cm^{-1} (Steward *et al.*, 1986). In (I)–(IV), an Si atom is bonded to the α-carbon of the carboxylate ions. Their $-2J$ values were determined to be (I) 323, (II) 334, (III) 322 and (IV) 333 cm^{-1} ($H = -2JS_1S_2$). These $-2J$ values are slightly smaller than those of trimethylacetatocopper complexes ($-2J \sim 356$ cm^{-1}; Goto *et al.*, 2000). In conclusion, the electronic influence of the Si atom on the spin-exchange interaction is very small if the Si atom is not bonded directly to the carboxylate ion.



(I) $R = C_5H_5N$
(II) $R = o$-Me-C_5H_4N
(III) $R = m$-Me-C_5H_4N
(IV) $R = C_9H_7N$
Ligands are N-coordinated



(V)

The complexes (I)–(V) (Figs. 1–5) lie about inversion centres. The trimethylsilyl groups are arranged to have approximately fourfold rotational symmetry along the Cu···Cu axis. Displacement ellipsoids of the terminal methyl-C atoms are large, indicating librational and rotational disorder. In (I)–(IV), the geometry around the Cu atoms is a typical square-pyramidal coordination, and the O—Cu—O bond angle between the bridges opposite each other is in the narrow range 166.9 (1) to 168.7 (2)°.

On the other hand, complex (V) (Fig. 5) has a polymeric structure generated by a glide translation and the Cu atoms have a distorted trigonal-bipyramidal coordination geometry with O—Cu—O bond angles of 178.0 (3)–178.5 (3)° and 158.0 (3)–159.0 (3)°. The Cu—O—Cu($x-\frac{1}{2}$, $\frac{1}{2}-y$, z) bond angles in the polymer chain of (V) are 100.7 (2) and 101.3 (2)°. In the April 1999 release of the CSD (Cambridge Structural Database, 1999), the number of entries for polymeric copper carboxylates was 14, and the Cu—O—Cu angles range from 98.2 to 109.3°.

Polymeric copper carboxylates are essentially antiferromagnetic, although the spin–spin interaction through the Cu—O—Cu bonds is ferromagnetic. The $-2J$ values are generally much less than those of dimeric complexes (Muto *et al.*, 1985). This may be due to a trigonal bipyramidal deformation of the cage structure, in accord with the magnetostructure correlations established in the study on the dimeric copper(II) trichloroacetates (Uekusa *et al.*, 1992).

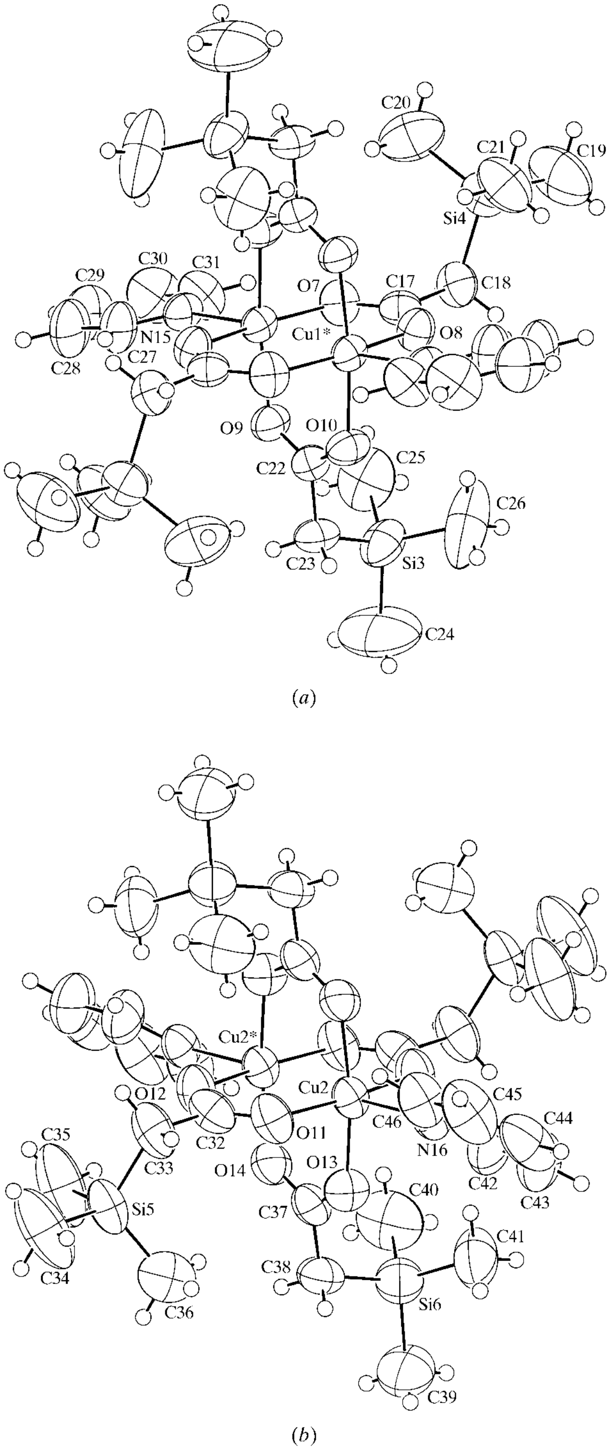


Figure 1
The molecular structures of (I); (*a*) and (*b*) show the two independent molecules. Displacement ellipsoids are plotted at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

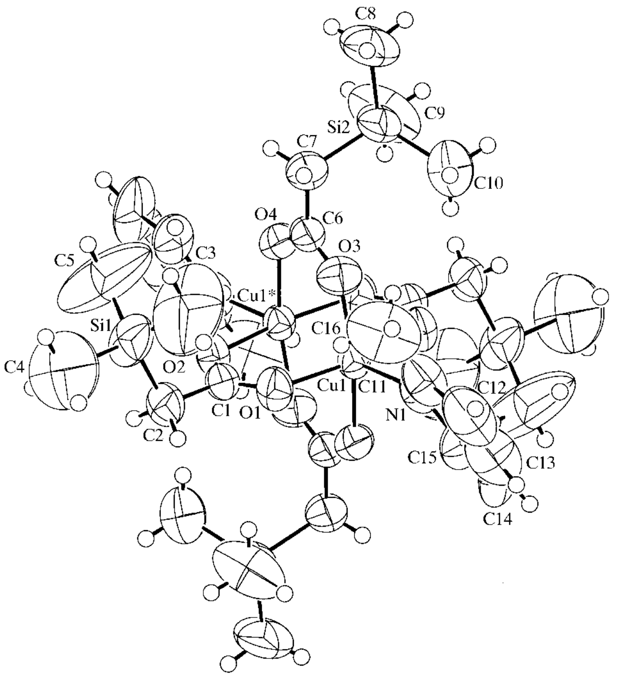


Figure 2
The molecular structure of (II) with displacement ellipsoids at the 50% probability level. The 2-methylpyridine ligand has an orientational disorder, and one of two possible orientations is shown. H atoms are shown as spheres of arbitrary radii.

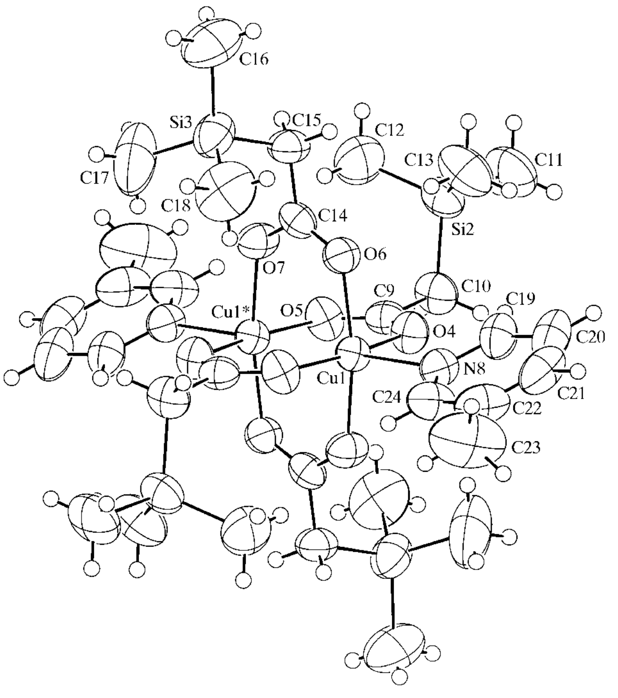


Figure 3
The molecular structure of (III) with displacement ellipsoids at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

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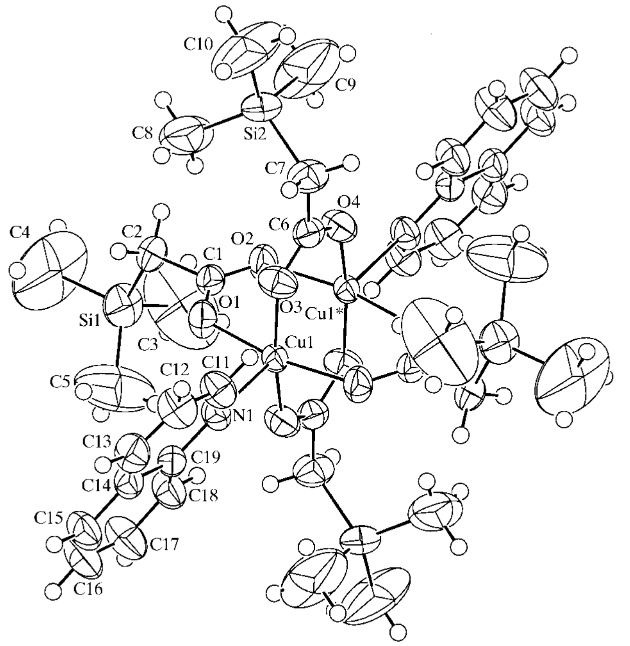

Figure 4
The molecular structure of (IV) with displacement ellipsoids at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

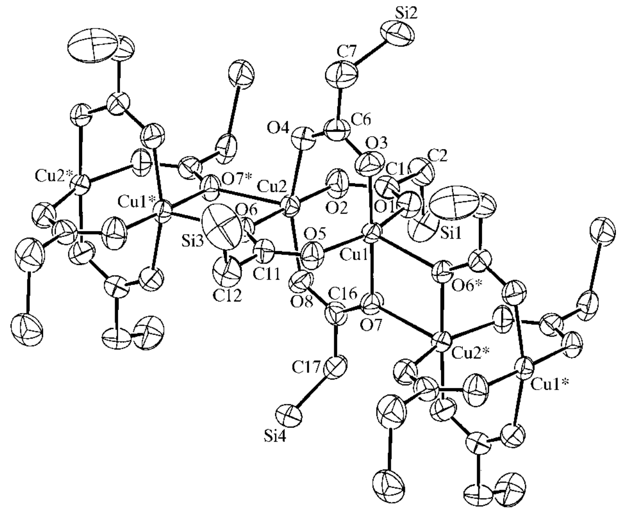

Figure 5
The linear chain structure of (V) with displacement ellipsoids at the 50% probability level. Methyl groups of the trimethylsilylacetato ligands and H atoms are omitted for clarity.

Experimental

Trimethylsilylacetic acid (132 mg, 1.0 mmol) and $CuCO_3{\cdot}Cu(OH)_2{\cdot}H_2O$ (60 mg, 0.25 mmol) were suspended in a mixture of water and methanol (3:1, 20 ml). After the solution was stirred for 1 h at room temperature, a green precipitate was collected and dissolved in ethanol. When pyridine (40 mg, 0.50 mmol) was added to the solution, the colour changed from green to blue, and from the solution, green crystals of (I) were grown by slow evaporation. Crystals of (II), (III) and (IV) were prepared in a similar way using 2-methylpyridine, 3-methylpyridine and quinoline as axial ligands. When these monodentate ligands were not introduced and the ethanol solution was kept in a desiccator, small blue–green crystals of polymer (V) were obtained together with pale-green plate-like crystals of a tetranuclear complex, $[Cu_2(Me_3SiCH_2COO)_4(H_2O)]_2$: monoclinic, $P2_1/n$, a = 12.697 (5), b = 12.085 (5), c = 22.609 (3) Å, β = 94.30 (2)°, V = 3459 (2) Å^3, Z = 2.

Compound (I)

Crystal data

$[Cu_2(C_5H_{11}O_2Si)_4(C_5H_5N)_2]$
M_r = 810.20
Triclinic, $P\overline{1}$
a = 11.392 (1) Å
b = 18.508 (2) Å
c = 10.851 (1) Å
α = 91.87 (1)°
β = 102.79 (1)°
γ = 76.82 (1)°
V = 2171.9 (5) Å^3

Z = 2
D_x = 1.239 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–15°
μ = 1.131 mm^{-1}
T = 299 K
Prism, green
0.4 × 0.3 × 0.3 mm

Data collection

Rigaku AFC-5 diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
T_{min} = 0.971, T_{max} = 0.978
10 462 measured reflections
9960 independent reflections
5122 reflections with $|F_o| > 3\sigma(|F_o|)$

R_{int} = 0.017
θ_{max} = 27.5°
$h = 0 \rightarrow 15$
$k = -24 \rightarrow 24$
$l = -14 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F
R = 0.068
wR = 0.053
S = 1.34
5122 reflections
415 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.000225F^2]$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

Cu1—Cu1^i	2.632 (1)	Cu2—O14^{ii}	1.972 (4)
Cu1—O7	1.962 (4)	Cu2—N16	2.198 (6)
Cu1—O8^i	1.961 (4)	O7—C17	1.263 (7)
Cu1—O9	1.982 (4)	O8—C17	1.276 (7)
Cu1—O10^i	1.972 (4)	O9—C22	1.255 (7)
Cu1—N15	2.194 (5)	O10—C22	1.268 (7)
Cu2—Cu2^{ii}	2.635 (1)	O11—C32	1.252 (10)
Cu2—O11	1.961 (4)	O12—C32	1.271 (8)
Cu2—O12^{ii}	1.965 (4)	O13—C37	1.276 (8)
Cu2—O13	1.959 (4)	O14—C37	1.258 (8)
O7—Cu1—O8^i	168.3 (2)	O7—C17—O8	123.6 (6)
O9—Cu1—O10^i	168.7 (2)	O9—C22—O10	125.5 (5)
O11—Cu2—O12^{ii}	168.3 (2)	O11—C32—O12	125.0 (6)
O13—Cu2—O14^{ii}	168.5 (2)	O13—C37—O14	124.6 (6)

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

Compound (II)

Crystal data

$[Cu_2(C_5H_{11}O_2Si)_4(C_6H_7N)_2]$
M_r = 838.26
Triclinic, $P\overline{1}$
a = 10.874 (2) Å
b = 11.508 (2) Å
c = 10.330 (2) Å
α = 104.26 (2)°
β = 109.10 (2)°
γ = 74.91 (2)°
V = 1160.9 (4) Å^3

Z = 1
D_x = 1.199 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–15°
μ = 1.060 mm^{-1}
T = 298 K
Plate-like, green
0.5 × 0.5 × 0.2 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\rm min} = 0.567$, $T_{\rm max} = 0.809$
8822 measured reflections
8412 independent reflections
5169 reflections with $I > 1.5\sigma(I)$
$R_{\rm int} = 0.009$
$\theta_{\rm max} = 32.5°$
$h = -16 \rightarrow 16$
$k = -17 \rightarrow 17$
$l = -15 \rightarrow 0$
3 standard reflections every 150 reflections intensity decay: 10.1%

Refinement

Refinement on *F*
$R = 0.051$
$wR = 0.079$
$S = 1.39$
5352 reflections
253 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00181|F_o|^2]$
$(\Delta/\sigma)_{\rm max} = 0.03$
$\Delta\rho_{\rm max} = 0.45$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.32$ e Å^{-3}

Table 2
Selected geometric parameters (Å, °) for (II).

Cu1—Cu1$^{\rm i}$	2.664 (1)	Cu1—N1	2.244 (3)
Cu1—O1	1.977 (2)	O1—C1	1.243 (4)
Cu1—O2$^{\rm i}$	1.960 (2)	O2—C1	1.261 (4)
Cu1—O3	1.953 (2)	O3—C6	1.264 (4)
Cu1—O4$^{\rm i}$	1.965 (2)	O4—C6	1.254 (4)
O1—Cu1—O2$^{\rm i}$	167.1 (1)	O1—C1—O2	125.4 (3)
O3—Cu1—O4$^{\rm i}$	167.2 (1)	O3—C6—O4	124.8 (3)

Symmetry code: (i) $-x, -y, -2-z$.

Compound (III)

Crystal data

$[\mathrm{Cu_2(C_5H_{11}O_2Si)_4(C_6H_7N)_2}]$
$M_r = 838.26$
Triclinic, $P\overline{1}$
$a = 11.148$ (3) Å
$b = 11.755$ (1) Å
$c = 10.563$ (3) Å
$\alpha = 110.91$ (1)°
$\beta = 117.70$ (2)°
$\gamma = 84.23$ (2)°
$V = 1141.3$ (5) Å^3
$Z = 1$
$D_x = 1.220$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 1.078$ mm^{-1}
$T = 297$ K
Plate-like, light green
0.4 × 0.3 × 0.1 mm

Data collection

Rigaku AFC-5 diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\rm min} = 0.676$, $T_{\rm max} = 0.893$
5532 measured reflections
5261 independent reflections
3377 reflections with $|F_o| > 3\sigma(|F_o|)$
$R_{\rm int} = 0.017$
$\theta_{\rm max} = 27.5°$
$h = 0 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -14 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: none

Table 3
Selected geometric parameters (Å, °) for (III).

Cu1—Cu1$^{\rm i}$	2.638 (3)	Cu1—N8	2.176 (3)
Cu1—O4	1.971 (3)	O4—C9	1.273 (5)
Cu1—O5$^{\rm i}$	1.958 (3)	O5—C9	1.240 (5)
Cu1—O6	1.976 (3)	O6—C14	1.249 (4)
Cu1—O7$^{\rm i}$	1.973 (3)	O7—C14	1.267 (5)
O4—Cu1—O5$^{\rm i}$	168.2 (2)	O4—C9—O5	125.5 (4)
O6—Cu1—O7$^{\rm i}$	168.1 (1)	O6—C14—O7	125.3 (4)

Symmetry code: (i) $2-x, -y, 1-z$.

Refinement

Refinement on *F*
$R = 0.064$
$wR = 0.054$
$S = 1.23$
3377 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.000225F^2]$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.66$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.51$ e Å^{-3}

Compound (IV)

Crystal data

$[\mathrm{Cu_2(C_5H_{11}O_2Si)_4(C_9H_7N)_2}]$
$M_r = 910.32$
Triclinic, $P\overline{1}$
$a = 11.298$ (2) Å
$b = 11.495$ (3) Å
$c = 10.261$ (3) Å
$\alpha = 104.03$ (2)°
$\beta = 110.10$ (2)°
$\gamma = 76.99$ (2)°
$V = 1199.4$ (5) Å^3
$Z = 1$
$D_x = 1.260$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 1.032$ mm^{-1}
$T = 298$ K
Plate-like, dark green
0.8 × 0.4 × 0.1 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\rm min} = 0.659$, $T_{\rm max} = 0.902$
9728 measured reflections
8692 independent reflections
6024 reflections with $I > 1.5\sigma(I)$
$R_{\rm int} = 0.040$
$\theta_{\rm max} = 32.5°$
$h = -17 \rightarrow 17$
$k = -17 \rightarrow 17$
$l = -16 \rightarrow 0$
3 standard reflections every 150 reflections intensity decay: 2.2%

Refinement

Refinement on *F*
$R = 0.048$
$wR = 0.069$
$S = 1.18$
6191 reflections
244 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00181|F_o|^2]$
$(\Delta/\sigma)_{\rm max} = 0.004$
$\Delta\rho_{\rm max} = 0.53$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Table 4
Selected geometric parameters (Å, °) for (IV).

Cu1—Cu1$^{\rm i}$	2.665 (1)	Cu1—N1	2.274 (2)
Cu1—O1	1.969 (2)	O1—C1	1.259 (3)
Cu1—O2$^{\rm i}$	1.965 (2)	O2—C1	1.254 (3)
Cu1—O3	1.973 (2)	O3—C6	1.258 (3)
Cu1—O4$^{\rm i}$	1.959 (2)	O4—C6	1.251 (3)
O1—Cu1—O2$^{\rm i}$	167.3 (1)	O1—C1—O2	125.3 (2)
O3—Cu1—O4$^{\rm i}$	166.9 (1)	O3—C6—O4	124.3 (2)

Symmetry code: (i) $-x, 1-y, 1-z$.

Compound (V)

Crystal data

$[\mathrm{Cu_2(C_5H_{11}O_2Si)_4}]$
$M_r = 652.00$
Monoclinic, $P2_1/a$
$a = 10.288$ (6) Å
$b = 24.692$ (5) Å
$c = 13.116$ (5) Å
$\beta = 97.71$ (4)°
$V = 3302$ (2) Å^3
$Z = 4$
$D_x = 1.312$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 1.468$ mm^{-1}
$T = 298$ K
Plate-like, blue–green
0.3 × 0.1 × 0.05 mm

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Data collection

Rigaku AFC-7*R* diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.852$, $T_{\max} = 0.929$
7986 measured reflections
7574 independent reflections
3260 reflections with $I > 1.5\sigma(I)$

$R_{\rm int} = 0.042$
$\theta_{\max} = 27.5°$
$h = -13 \rightarrow 0$
$k = 0 \rightarrow 32$
$l = -17 \rightarrow 17$
3 standard reflections every 150 reflections intensity decay: 1.4%

Refinement

Refinement on F
$R = 0.068$
$wR = 0.100$
$S = 1.484$
3309 reflections
247 parameters

H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00212|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -0.60$ e Å^{-3}

During the data collection of (II), an intensity decay of 10.1% was observed and was corrected for based on the standard reflections.

In (II), there is an orientational disorder of the 2-methylpyridine ligand. The site-occupation factors of N1, C12 and C13 were assumed to be 100%, and the remaining four C atoms are either of two sets of positions, (C11, C14, C15, C16) or (C17, C18, C19, C20), with 50% probabilities. The max/min ratio of the atom displacement parameters are abnormally large for the methyl-C atoms of the trimethylsilyl groups, which may be due to the librational and rotational disorder. To avoid insufficient convergence in the refinement of (V), all 12 terminal methyl-C atoms were refined isotropically.

Table 5
Selected geometric parameters (Å, °) for (V).

Cu1—Cu2	2.618 (2)	Cu2—O8	2.003 (6)
Cu1—O1	1.988 (6)	O1—C1	1.25 (1)
Cu1—O3	1.916 (7)	O2—C1	1.28 (1)
Cu1—O5	1.997 (6)	O3—C6	1.26 (1)
Cu1—O6$^{\rm i}$	2.291 (6)	O4—C6	1.25 (1)
Cu1—O7	1.950 (6)	O5—C11	1.24 (1)
Cu2—O2	1.892 (6)	O6—C11	1.28 (1)
Cu2—O4	1.970 (7)	O7—C16	1.27 (1)
Cu2—O6	1.935 (6)	O8—C16	1.25 (1)
Cu2—O7$^{\rm ii}$	2.261 (6)		
O1—Cu1—O5	159.0 (3)	Cu1—O7—Cu2$^{\rm i}$	101.3 (2)
O3—Cu1—O7	178.5 (3)	O1—C1—O2	124.2 (9)
O2—Cu2—O6	178.0 (3)	O3—C6—O4	124.1 (9)
O4—Cu2—O8	158.0 (3)	O5—C11—O6	123.5 (9)
Cu1—O6$^{\rm i}$—Cu2$^{\rm i}$	100.7 (2)	O7—C16—O8	123.4 (8)

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

The magnetic susceptibilities over the temperature range 80–300 K were determined by the Faraday method at Saga University. The procedure for determining the $-2J$ value from cryomagnetic data based on the Bleaney–Bowers equation is described elsewhere (Harada *et al.*, 1997).

For all compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: local programs for (I) and (III); *TEXSAN* (Molecular Structure Corporation, 1998) for (II), (IV) and (V). Program(s) used to solve structure: *CRYSTAN-GM* (Edwards *et al.*, 1996) for (I) and (III). Program(s) used to refine structure: *CRYSTAN-GM* for (I) and (III); *TEXSAN* for (II), (IV) and (V). Molecular graphics: *CRYSTAN-GM* for (I) and (III). Software used to prepare material for publication: *CRYSTAN-GM* for (I) and (III); *TEXSAN* for (II), (IV) and (V).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1095). Services for accessing these data are described at the back of the journal.

References

Cambridge Structural Database (1999). Version 5.17. Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, England.

Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.

Edwards, C., Gilmore, C. J., Mackay, S. & Stewart, N. (1996). *CRYSTAN-GM. Program for the Solution and Refinement of Crystal Structures.* Version 6.3. MacScience Co. Ltd, Yokohama, Japan.

Goto, M., Kani, Y., Tsuchimoto, M., Ohba, S., Matsushima, H. & Tokii, T. (2000). *Acta Cryst.* C**56**, 7–11.

Harada, A., Tsuchimoto, M., Ohba, S., Iwasawa, K. & Tokii, T. (1997). *Acta Cryst.* B**53**, 654–661.

Kato, M. & Muto, Y. (1988). *Coord. Chem. Rev.* **92**, 45–83.

Molecular Structure Corporation (1993). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1998). *TEXSAN. Single Crystal Structure Analysis Software.* Version 1.9. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Muto, Y., Sasaki, A., Tokii, T. & Nakashima, M. (1985). *Bull. Chem. Soc. Jpn*, **58**, 2572–2576.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Steward, O. W., McAfee, R. C., Chang, S. C., Piskor, S. R., Schreiber, W. J., Jury, C. F., Taylor, C. E., Pletcher, J. F. & Chen, C. S. (1986). *Inorg. Chem.* **25**, 771–777.

Uekusa, H., Ohba, S., Tokii, T., Muto, Y., Kato, M., Husebye, S., Stewart, O. W., Chang, S.-C., Rose, J. P., Pletcher, J. F. & Suzuki, I. (1992). *Acta Cryst.* B**48**, 650–667.

Yamanaka, M., Uekusa, H., Ohba, S., Saito, Y., Iwata, S., Kato, M., Tokii, T., Muto, Y. & Steward, O. W. (1991). *Acta Cryst.* B**47**, 344–355.