Table 2. *Bond lengths* (Å) *and selected bond angles* (°)

| | | | |
|---|---|---|---|
| W(1)—C(1) | 1.994 (11) | O(3)—C(3) | 1.146 (14) |
| W(1)—C(2) | 1.968 (12) | O(4)—C(9) | 1.201 (14) |
| W(1)—C(3) | 1.982 (14) | O(5)—C(9) | 1.353 (14) |
| W(1)—C(4) | 2.322 (11) | O(5)—C(10) | 1.477 (16) |
| W(1)—C(5) | 2.357 (11) | C(4)—C(5) | 1.425 (16) |
| W(1)—C(6) | 2.412 (11) | C(4)—C(8) | 1.441 (15) |
| W(1)—C(7) | 2.365 (11) | C(5)—C(6) | 1.415 (16) |
| W(1)—C(8) | 2.293 (10) | C(6)—C(7) | 1.417 (15) |
| O(1)—C(1) | 1.149 (12) | C(7)—C(8) | 1.434 (14) |
| O(2)—C(2) | 1.155 (14) | C(8)—C(9) | 1.473 (16) |
| W(1)—W(1$^{i}$) | 3.216 (1) | | |

| | | | |
|---|---|---|---|
| C(1)—W(1)—C(2) | 106.6 (5) | C(6)—C(7)—C(8) | 107 (1) |
| C(1)—W(1)—C(3) | 77.2 (5) | C(4)—C(8)—C(7) | 108 (1) |
| C(2)—W(1)—C(3) | 77.8 (6) | C(4)—C(8)—C(9) | 124 (1) |
| W(1)—C(1)—O(1) | 172 (1) | C(7)—C(8)—C(9) | 128 (1) |
| W(1)—C(2)—O(2) | 176 (1) | O(4)—C(9)—O(5) | 123 (1) |
| W(1)—C(3)—O(3) | 177 (1) | O(4)—C(9)—C(8) | 125 (1) |
| C(5)—C(4)—C(8) | 107 (1) | O(5)—C(9)—C(8) | 111 (1) |
| C(4)—C(5)—C(6) | 109 (1) | C(9)—O(5)—C(10) | 118 (1) |
| C(5)—C(6)—C(7) | 109 (1) | | |

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

soluble carboxylic acid [W—W 3.215 (3) Å] (Avey, Tenhaeff, Weakley & Tyler, 1991) and of polyesters with W—W bonds in the backbone (Tenhaeff & Tyler, 1991). The W—W bond lengths in related compounds include: $W_2(C_5H_5)_2(CO)_6$, 3.222 (1) Å (Adams, Collins & Cotton, 1974); $W_2(C_5Me_5)_2(CO)_6$, 3.288 (1) Å (Rheingold & Harper, 1991).

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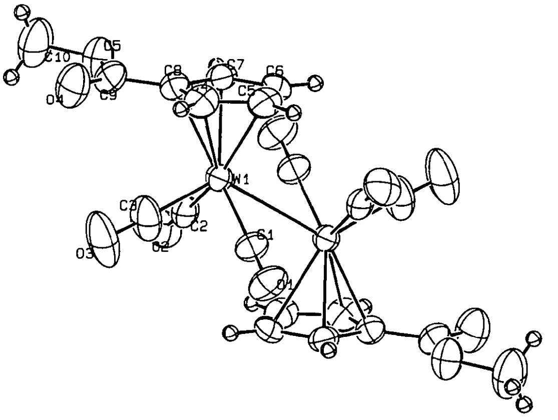

Fig. 1. Molecular structure of $W_2(\eta^5\text{-}C_5H_4COOCH_3)_2(CO)_6$ showing thermal ellipsoids (H atoms, arbitrary radius) and numbering scheme.

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*Acta Cryst.* (1992). C**48**, 155–157

# Structure of (μ-Oxalato)-*trans*-bis[*N*,*N*,*N*′,*N*′-tetramethylethylenediamineperfluoro-*tert*-butoxycopper(II)] Benzene Solvate

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**Abstract.** (μ-Oxalato)-*trans*-bis[*N*,*N*,*N*′,*N*′-tetramethylethylenediamineperfluoro-*tert*-butoxycopper(II)] benzene solvate, $C_{22}H_{32}Cu_2F_{18}N_4O_6.C_6H_6$, $M_r = 995.70$, monoclinic, $C2/m$, $a = 14.581\,(5)$, $b = 12.484\,(4)$, $c = 13.239\,(5)$ Å, $\beta = 123.05\,(2)°$, $V = 2019.9\,(12)$ Å$^3$, $Z = 2$, $D_x = 1.637$ Mg m$^{-3}$, $\lambda(\text{Mo}\,K\alpha) = 0.71073$ Å, $\mu = 1.18$ mm$^{-1}$, $F(000) = 1004$, $T = 295$ K, final $R = 0.055$, $wR = 0.067$ for 1629 independent observed reflections. The distorted square-pyramidal five-coordinate Cu$^{\text{II}}$ complex and the benzene solvate both have $C_{2h}$ molecular symmetry. The asymmetric unit consists of $\frac{1}{4}$ of the Cu complex and $\frac{1}{4}$ of the solvate molecule. All of the $CF_3$ groups are poorly defined and the fluorines of one of the crystallographically independent $CF_3$ groups were treated as a disorder with occupancies of 52 and 48% respectively.

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Table 1. *Atomic coordinates* (× $10^4$) *and equivalent isotropic displacement coefficients* (Å$^2$ × $10^3$)

| | $x$ | $y$ | $z$ | $U_{eq}$* |
|---|---|---|---|---|
| Cu(1) | 2050 (1) | 0 | 1777 (1) | 52 (1) |
| O(1) | 884 (2) | 1061 (2) | 719 (3) | 69 (1) |
| C(1) | 0 | 619 (5) | 0 | 56 (3) |
| O(2) | 2013 (3) | 0 | 3312 (4) | 62 (2) |
| C(2) | 1436 (5) | 0 | 3804 (6) | 59 (3) |
| C(3) | 748 (9) | 1016 (9) | 3496 (11) | 163 (10) |
| F(1′) | − 196 (10) | 682 (19) | 2273 (13) | 185 (11) |
| F(2′) | 1201 (12) | 1838 (9) | 3427 (23) | 142 (13) |
| F(3′) | 322 (22) | 1221 (17) | 4142 (24) | 189 (17) |
| F(1) | 217 (20) | 1277 (18) | 2380 (17) | 208 (18) |
| F(2) | 1553 (17) | 1762 (16) | 4390 (25) | 202 (19) |
| F(3) | − 51 (18) | 834 (21) | 3728 (25) | 222 (17) |
| C(4) | 2204 (9) | 0 | 5188 (10) | 157 (9) |
| F(4) | 2888 (6) | − 828 (7) | 5550 (5) | 213 (4) |
| F(5) | 1746 (5) | 0 | 5810 (6) | 173 (5) |
| N(1) | 3250 (3) | 1115 (4) | 2337 (4) | 83 (2) |
| C(5) | 4274 (5) | 581 (3) | 2978 (10) | 210 (7) |
| C(6) | 3158 (6) | 1700 (6) | 1322 (6) | 132 (5) |
| C(7) | 3178 (6) | 1949 (6) | 3108 (6) | 126 (5) |
| C(1′) | 5000 | 1077 (9) | 0 | 153 (16) |
| C(2′) | 5763 (8) | 521 (7) | 930 (9) | 160 (8) |

* Equivalent isotropic $U$ defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

**Experimental.** The title compound was produced from a mixture of $Cu(OH)_2$† (0.71 g, 7.3 mmol), $(CF_3)_3OH$ (0.38 g, 1.6 mmol), benzene (10 ml), THF (5 ml), *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (2 ml) and 3 Å molecular sieve (0.13 g); which was stirred at 373 K for 3 d, filtered and evaporated, forming a viscous green residue. Recrystallized from heptane/benzene as a minor product. A clear blue prism 0.48 × 0.42 × 0.53 mm data crystal was sealed in a glass capillary under an inert atmosphere. Automated Siemens *R*3*m*/*V* diffractometer with incident beam monochromator. 25 centered reflections within $30 \leq 2\theta \leq 35°$ used for determining lattice parameters. $[\sin(\theta)/\lambda]_{max} = 0.54$ Å$^{-1}$, range of *hkl*: $-17 \leq h \leq 14$, $0 \leq k \leq 14$, $0 \leq l \leq 15$. Standards 005, 060, 400, monitored every 97 reflections with random variation of 2.6% over data collection, $\theta/2\theta$ scan mode, scan width $[2\theta(K\alpha_1) - 1.0]$ to $[2\theta(K\alpha_2) + 1.0]°$, $\omega$ scan rate a function of count rate (5.0° min$^{-1}$ minimum, 15.0° min$^{-1}$ maximum), 1957 reflections measured, 1871 unique, $R_{int} = 1.7\%$, 1629 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz, polarization and absorption effects, max. and min. transmission: 0.92 and 0.82. The structure solution by direct methods, and the full-matrix least-squares refinement used programs in *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.00023$. Secondary-extinction parameter $p = 0.0003$ (1) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 169 parameters were refined: atomic coordinates and anisotropic thermal parameters for all non-H atoms. H atoms included using riding model [coordinate shifts of C applied to attached H atoms, C—H distance set to 0.96 Å, H angles idealized, $U_{iso}$(H) set to *ca* 1.1 $U_{eq}$(C)]. The disordered $CF_3$ group F atom site occupation factors were refined and required to sum to 1.0. The C—F and F···F separations were restrained to be 1.34 (3) Å and 2.21 (5) Å, respec-

Table 2. *Bond lengths* (Å) *and bond angles* (°)

| | | | |
|---|---|---|---|
| Cu(1)—O(1) | 2.002 (3) | Cu(1)—O(2) | 2.062 (6) |
| Cu(1)—N(1) | 2.033 (4) | Cu(1)—O(1*B*) | 2.002 (3) |
| Cu(1)—N(1*A*) | 2.033 (4) | O(1)—C(1) | 1.238 (4) |
| C(1)—O(1*A*) | 1.238 (4) | C(1)—C(1*A*) | 1.545 (12) |
| O(2)—C(2) | 1.314 (11) | C(2)—C(3) | 1.529 (12) |
| C(2)—C(4) | 1.540 (12) | C(2)—C(3*A*) | 1.529 (12) |
| C(3)—F(1′) | 1.504 (16) | C(3)—F(2′) | 1.250 (22) |
| C(3)—F(3′) | 1.327 (43) | C(3)—F(1) | 1.281 (23) |
| C(3)—F(2) | 1.459 (22) | C(3)—F(3) | 1.375 (38) |
| C(4)—F(4) | 1.332 (11) | C(4)—F(5) | 1.313 (19) |
| C(4)—F(4*A*) | 1.332 (11) | N(1)—C(5) | 1.418 (7) |
| N(1)—C(6) | 1.470 (10) | N(1)—C(7) | 1.501 (10) |
| C(5)—C(5*A*) | 1.450 (9) | C(1′)—C(2′) | 1.319 (10) |
| C(1′)—C(2′*A*) | 1.319 (10) | C(2′)—C(2′*B*) | 1.301 (17) |
| | | | |
| O(1)—Cu(1)—O(2) | 102.7 (1) | O(1)—Cu(1)—N(1) | 92.1 (1) |
| O(2)—Cu(1)—N(1) | 96.9 (2) | O(1)—Cu(1)—O(1*B*) | 82.8 (1) |
| O(2)—Cu(1)—O(1*B*) | 102.7 (1) | N(1)—Cu(1)—O(1*B*) | 160.3 (2) |
| O(1)—Cu(1)—N(1*A*) | 160.3 (2) | O(2)—Cu(1)—N(1*A*) | 96.9 (2) |
| N(1)—Cu(1)—N(1*A*) | 86.4 (3) | O(1*B*)—Cu(1)—N(1*A*) | 92.1 (1) |
| Cu(1)—O(1)—C(1) | 112.0 (3) | O(1)—C(1)—O(1*A*) | 127.1 (5) |
| O(1)—C(1)—C(1*A*) | 116.5 (3) | O(1*A*)—C(1)—C(1*A*) | 116.5 (3) |
| Cu(1)—O(2)—C(2) | 148.9 (3) | O(2)—C(2)—C(3) | 111.9 (7) |
| O(2)—C(2)—C(4) | 110.1 (7) | C(3)—C(2)—C(4) | 105.2 (7) |
| O(2)—C(2)—C(3*A*) | 111.9 (7) | C(3)—C(2)—C(3*A*) | 112.2 (10) |
| C(4)—C(2)—C(3*A*) | 105.2 (7) | C(2)—C(3)—F(1′) | 98.0 (11) |
| C(2)—C(3)—F(2′) | 113.9 (13) | F(1′)—C(3)—F(2′) | 111.0 (16) |
| C(2)—C(3)—F(3′) | 117.3 (13) | F(1′)—C(3)—F(3′) | 105.8 (14) |
| F(2′)—C(3)—F(3′) | 109.9 (18) | C(2)—C(3)—F(1) | 114.5 (16) |
| C(2)—C(3)—F(2) | 101.1 (10) | F(1)—C(3)—F(2) | 119.1 (18) |
| C(2)—C(3)—F(3) | 108.4 (14) | F(1)—C(3)—F(3) | 104.3 (18) |
| F(2)—C(3)—F(3) | 109.2 (21) | C(2)—C(4)—F(4) | 110.1 (8) |
| C(2)—C(4)—F(5) | 117.3 (9) | F(4)—C(4)—F(5) | 108.2 (8) |
| C(2)—C(4)—F(4*A*) | 110.1 (8) | F(4)—C(4)—F(4*A*) | 101.9 (10) |
| F(5)—C(4)—F(4*A*) | 108.2 (8) | Cu(1)—N(1)—C(5) | 108.2 (3) |
| Cu(1)—N(1)—C(6) | 112.1 (3) | C(5)—N(1)—C(6) | 108.8 (7) |
| Cu(1)—N(1)—C(7) | 111.0 (4) | C(5)—N(1)—C(7) | 111.1 (5) |
| C(6)—N(1)—C(7) | 105.7 (5) | N(1)—C(5)—C(5*A*) | 118.0 (3) |
| C(2′)—C(1′)—C(2′*A*) | 116.5 (11) | C(1′)—C(2′)—C(2′*B*) | 121.7 (6) |

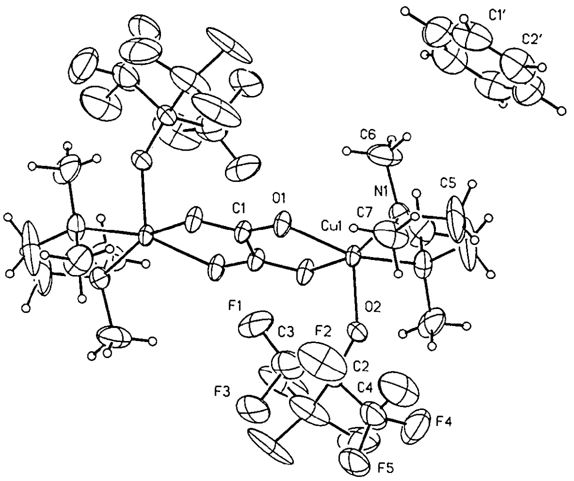

Fig. 1. Thermal ellipsoid plot of title compound with ellipsoids drawn at the 20% probability level; lower-occupancy disordered fluorines are omitted for clarity.

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† $Cu(OH)_2$ may have absorbed $CO_2$ from the air during its preparation from $CuCl_2$ and $NH_4OH$.

tively. $(\Delta/\sigma)_{\max} = 0.86$, ratio of observations to parameters 9.6:1, $R = 0.055$, $wR = 0.067$, $S = 2.76$. $R = 0.062$ for all data. Final difference Fourier excursions 0.61 and $-0.60$ e Å$^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atom numbering for Table 1, atom coordinates, and Table 2, bond distances and angles, follows that shown in Fig. 1.*

**Related literature.** Compounds with similar Cu bonding environment include (oxalato)-bis(aqua-*N*,*N*,*N*′,*N*′-tetramethylethylenediaminecopper(II)bis(hexafluorophosphate) dihydrate (Sletten, 1983), and (oxalato)-bis(aqua-*N*,*N*,*N*′,*N*′-tetramethylenediaminecopper(II) diperchlorate hydrate (Julve, Verdaguer, Gleizes, Philoche-Levisalles & Kahn, 1984).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54141 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Bis(triphenylphosphine oxide)sodium Nitrido[*N*,*N*′-*o*-phenylenebis(salicylamidato)-*N*,*N*′,*O*,*O*′]osmate(VI)

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**Abstract.** $[Na(C_{18}H_{15}OP)_2][OsN(C_{20}H_{12}N_2O_4)]$, $M_r = 1128.12$, triclinic, $P\bar{1}$, $a = 14.027$ (2), $b = 14.738$ (3), $c = 14.939$ (3) Å, $\alpha = 99.95$ (2), $\beta = 106.90$ (2), $\gamma = 117.11$ (1)°, $V = 2455.3$ (9) Å$^3$, $Z = 2$, $D_x = 1.526$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71073 Å, $\mu = 26.75$ cm$^{-1}$, $F(000) = 1128$, $T = 296$ K, $R_F = 3.45\%$ for 7384 observed reflections and 551 parameters. The geometry about the anionic Os$^{VI}$ coordination complex is square pyramidal, as found for other five-coordinate highly oxidized metal nitrides. The Os atom is displaced 0.57 Å above the basal plane of the pyramid. The Os—N(1) bond distance of 1.639 (6) Å is indicative of an Os—N triple bond. The Na$^+$ counterion is associated with the two Os-bound O atoms and two molecules of Ph$_3$PO.

**Experimental.** Yellow cube-shaped crystals (0.25 × 0.25 × 0.25 mm) were the gift of Terrence Collins at Carnegie Mellon University. Nicolet *R*3*m* diffractometer with graphite monochromator; $\omega$ scans; lattice parameters from least-squares fit of 25 reflections ($20 \leq 2\theta \leq 25°$); empirical absorption correction was applied ($T_{\max}/T_{\min} = 1.17$); $2\theta_{\max} = 50°$ ($h = \pm 17$, $k = \pm 18$, $l = 18$); standard reflections $\bar{4}29$, $\bar{5}90$, $\bar{8}26$. 9382 reflections collected, 8876 unique ($R_{\rm int} = 2.28\%$), 7384 observed with $F_o > 4.0\sigma(F_o)$, 1492 unobserved reflections. Patterson structure solution; least-squares refinement on 551 parameters; all non-H atoms anisotropic, H atoms included as idealized contributors ($d_{\rm CH} = 0.960$ Å; $U = 1.2$ times the $U$ value for the attached C atom), $R_F = 3.45\%$, $wR_F = 3.79\%$, $S = 1.081$, $w^{-1} = \sigma(F_o) + gF_o^2$, $g = 0.001$; $(\Delta/\sigma)_{\max} = 0.139$; $\Delta\rho_{\max} = 0.79$, $\Delta\rho_{\min} = -0.93$ e Å$^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 144); *SHELXTL* computer program (Sheldrick, 1985).

Atomic and equivalent isotropic thermal parameters are given in Table 1, and selected bond lengths and angles are listed in Table 2. Fig. 1 shows the molecular structure of the compound. An isolated view of the anion displaying the coordination sphere of Os is shown in Fig. 2.†

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† Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and full lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54448 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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