O(1)	0.27183 (11)	0.36472 (15)	0.02334 (15)	3.57 (9)
O(2)	0.23308 (11)	0.46996 (17)	0.34209 (16)	3.98 (9)
N(1)	0.36420 (16)	0.42941 (18)	−0.05929 (21)	3.91 (11)
N(2)	0.33394 (14)	0.32598 (16)	0.22833 (16)	2.99 (9)
N(3)	0.16663 (13)	0.32497 (15)	0.20953 (16)	2.94 (9)
N(4)	0.14399 (15)	0.55346 (19)	0.41008 (20)	3.94 (11)
N(5)	0.16956 (14)	0.50539 (17)	0.13551 (19)	3.58 (11)
N(6)	0.33237 (14)	0.51144 (17)	0.17289 (20)	3.75 (11)
C(1)	0.34168 (16)	0.38159 (19)	0.01128 (20)	2.97 (11)
C(2)	0.40668 (16)	0.34532 (19)	0.07769 (21)	3.06 (11)
C(3)	0.37540 (18)	0.28303 (20)	0.14991 (22)	3.43 (12)
C(4)	0.39381 (18)	0.36101 (24)	0.29873 (23)	4.05 (13)
C(5)	0.28763 (20)	0.25550 (21)	0.27374 (24)	3.98 (14)
C(6)	0.21157 (19)	0.24072 (20)	0.21664 (24)	3.72 (14)
C(7)	0.10714 (17)	0.31764 (22)	0.12839 (23)	3.71 (12)
C(8)	0.12357 (18)	0.33585 (22)	0.29751 (23)	3.83 (13)
C(9)	0.09762 (17)	0.42938 (22)	0.31660 (22)	3.77 (13)
C(10)	0.16309 (16)	0.48617 (21)	0.35703 (19)	3.14 (12)
C(11)	0.12152 (17)	0.55491 (20)	0.10840 (21)	3.12 (11)
C(12)	0.3726 (3)	0.5599 (3)	0.1354 (3)	3.93 (19)
C(12′)	0.3881 (5)	0.5509 (6)	0.1673 (8)	2.3 (4)

Table 2. *Selected geometric parameters* (Å, °)

Cu—O(1)	2.560 (3)	N(2)—C(4)	1.485 (4)
Cu—O(2)	2.352 (3)	N(2)—C(5)	1.498 (4)
Cu—N(2)	2.091 (2)	N(3)—C(6)	1.488 (4)
Cu—N(3)	2.078 (2)	N(3)—C(7)	1.496 (4)
Cu—N(5)	1.978 (3)	N(3)—C(8)	1.502 (4)
Cu—N(6)	1.969 (3)	N(4)—C(10)	1.323 (4)
S(1)—C(11)	1.641 (3)	N(5)—C(11)	1.158 (4)
S(1′)—C(11)	1.612 (5)	N(6)—C(12)	1.156 (5)
S(2)—C(12)	1.631 (5)	N(6)—C(12′)	1.13 (1)
S(2′)—C(12′)	1.63 (1)	C(1)—C(2)	1.517 (4)
S(2″)—C(12)	1.620 (6)	C(2)—C(3)	1.520 (4)
O(1)—C(1)	1.236 (3)	C(5)—C(6)	1.502 (5)
O(2)—C(10)	1.245 (3)	C(8)—C(9)	1.515 (5)
N(1)—C(1)	1.318 (4)	C(9)—C(10)	1.494 (4)
N(2)—C(3)	1.508 (4)		
O(1)—Cu—O(2)	177.79 (8)	Cu—N(3)—C(8)	114.6 (2)
O(1)—Cu—N(2)	83.29 (9)	C(6)—N(3)—C(7)	108.0 (2)
O(1)—Cu—N(3)	91.89 (9)	C(6)—N(3)—C(8)	108.1 (2)
O(1)—Cu—N(5)	90.1 (1)	C(7)—N(3)—C(8)	108.5 (2)
O(1)—Cu—N(6)	89.3 (1)	Cu—N(5)—C(11)	177.2 (3)
O(2)—Cu—N(2)	94.52 (9)	Cu—N(6)—C(12)	158.7 (3)
O(2)—Cu—N(3)	87.59 (9)	Cu—N(6)—C(12′)	168.1 (5)
O(2)—Cu—N(5)	92.1 (1)	O(1)—C(1)—N(1)	123.1 (3)
O(2)—Cu—N(6)	91.1 (1)	O(1)—C(1)—C(2)	120.5 (3)
N(2)—Cu—N(3)	85.78 (9)	N(1)—C(1)—C(2)	116.4 (3)
N(2)—Cu—N(5)	173.1 (1)	C(1)—C(2)—C(3)	112.1 (2)
N(2)—Cu—N(6)	92.0 (1)	N(2)—C(3)—C(2)	115.9 (2)
N(3)—Cu—N(5)	92.7 (1)	N(2)—C(5)—C(6)	109.1 (2)
N(3)—Cu—N(6)	177.3 (1)	N(3)—C(6)—C(5)	109.2 (3)
N(5)—Cu—N(6)	89.8 (1)	N(3)—C(8)—C(9)	114.5 (3)
Cu—O(1)—C(1)	104.6 (2)	C(8)—C(9)—C(10)	113.0 (3)
Cu—O(2)—C(10)	113.2 (2)	O(2)—C(10)—N(4)	121.4 (3)
Cu—N(2)—C(3)	115.9 (2)	O(2)—C(10)—C(9)	121.0 (3)
Cu—N(2)—C(4)	111.3 (2)	N(4)—C(10)—C(9)	117.6 (3)
Cu—N(2)—C(5)	104.4 (2)	S(1)—C(11)—N(5)	177.9 (3)
C(3)—N(2)—C(4)	109.1 (2)	S(1′)—C(11)—N(5)	168.2 (3)
C(3)—N(2)—C(5)	107.2 (2)	S(2)—C(12)—N(6)	172.2 (5)
C(4)—N(2)—C(5)	108.5 (2)	S(2″)—C(12)—N(6)	171.8 (5)
Cu—N(3)—C(6)	105.4 (2)	S(2′)—C(12′)—N(6)	179 (1)
Cu—N(3)—C(7)	111.9 (2)		

The structure was solved by direct and Fourier methods, and refined by full-matrix least squares. H atoms were solved by difference Fourier methods. Program used: *NRCVAX* (Gabe, Le Page, White & Lee, 1987).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and possible hydrogen bonds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71842 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1076]

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Bis(μ-4-methylphenoxido-1:2κ^2O)tris(triphenylphosphine)-1κ^2P,2κP-dicopper(I)

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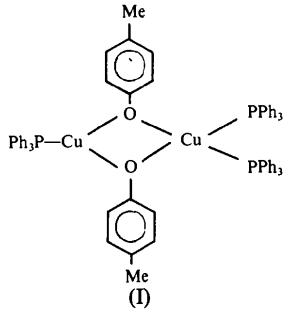
Abstract

$(Ph_3P)_2Cu(\mu\text{-}OC_6H_4Me\text{-}4)_2Cu(PPh_3)$ has one Cu atom, Cu(1), in a distorted tetrahedral coordination environment and the other, Cu(2), in a distorted trigonal planar environment. The P(1)—Cu(1)—P(2) angle [127.0 (1)°] involving the phosphines is significantly larger than the O(1)—Cu(1)—O(2) angle [79.3 (2)°] involving the bridging 4-methylphenoxido ligands. The P—Cu(1)—O angles fall within the range 106.6 (2)–112.9 (2)°. The dihedral angle between the plane defined by the Cu atom and its two coordinated P atoms and that defined by the Cu atom and the two O atoms is 89.2°. The sum of the three *L*—Cu—*L* angles around Cu(2) is 360.0°. However, the triangular environment around Cu(2) is not symmetric: O(2)—Cu(2)—P(3) 142.9 (2), O(1)—Cu(2)—P(3) 132.0 (2) and O(1)—Cu(2)—O(2) 85.0 (2)°.

Comment

The structure determination was undertaken to determine how the 4-methylphenoxido ligand would assemble Cu^I centers in the presence of triphenylphosphine. Phenoxido ligands are useful bridging ligands for the formation of bimetallic copper complexes of general formula $Cu_2(OAr)_2L_4$, where L is a phosphine (Naldini, Panzanelli, Rassu, Cariati, Demartin, Manassero & Masciocchi, 1984) or an isocyanide (Pasquali, Fiaschi, Floriani & Gaetani-Manfredotti, 1983). Structural data for a 2:3 phenoxide:phosphine complex, $(PPh_3)_2Cu(\mu\text{-}OC_6H_4NO_2)_2Cu(PPh_3)$, have also been reported (Naldini *et al.*, 1984). With 4-methylphenoxide, formation of the 2:3 complex $(Ph_3P)_2Cu(\mu\text{-}OC_6H_4Me\text{-}4)_2Cu(PPh_3)$ is facile. The title complex (I) crystallizes with the familiar structure of $Cu_2Cl_2(PPh_3)_3$ (Albano, Bellon, Ciani & Manassero, 1972). Except for the difference in the substitution at the *para* position of the phenoxido group, the structure of this molecule is the same as that of $Cu_2(OC_6H_4NO_2)_2(PPh_3)_3$. The Cu—O···P core distances of the title complex and the nitrophenoxido analog are listed in Table 3 for comparison.



(I)

The Cu(1)—O distances are 0.085–0.145 Å longer than the Cu(2)—O distances. This is similar to the 0.13–0.17 Å difference found for the nitrophenoxido complex. The Cu(1)—P distances are also longer, by 0.11–0.13 Å, than the Cu(2)—P distance. These differences are larger than the 0.07 Å extrapolated difference between three and four-coordinate Cu^I (Shannon, 1976).

All of the metal–ligand distances in the title complex are shorter than those in the nitrophenoxido analogue. Since both structures are similar and both data sets were obtained at ambient temperatures, the decrease in bond lengths can be attributed to methyl *versus* nitro substituent effects. A similar trend is observed for the average Cu—O distances of the four-coordinate Cu atoms in the title complex [2.084 (13) Å], in $Cu_2(OC_6H_5)_2(CNC_6H_4Me)_4$ [2.074 (8) Å] (Pasquali, Fiaschi, Floriani & Gaetani-Manfreddotti, 1983) and in $Cu_2(OC_6H_4NO_2)_2(PPh_3)_3$ [2.154 (21) Å]. The Cu—O distance appears to lengthen when an electron-withdrawing substituent is in the *para* position of the phenoxido group.

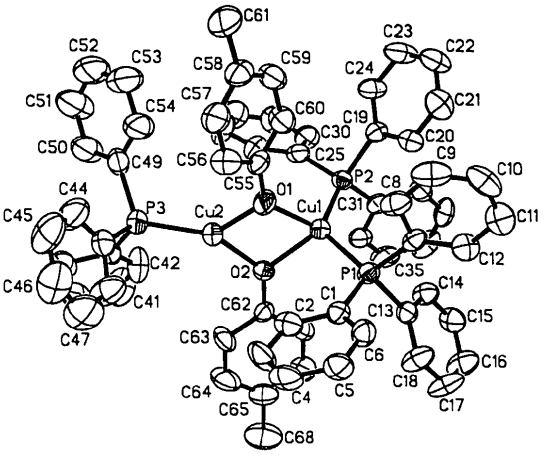


Fig. 1. View of $Cu_2(OC_6H_4Me\text{-}4)_2(PPh_3)_3$ with the probability ellipsoids drawn at the 50% level. H atoms have been omitted for clarity.

Experimental

Crystal data

$[Cu_2(C_7H_7O)_2(C_{18}H_{15}P)_3]$
$M_r = 1128.1$
Monoclinic
$P2_1/c$
$a = 10.5568\ (13)$ Å
$b = 28.186\ (4)$ Å
$c = 19.640\ (3)$ Å
$\beta = 101.809\ (10)°$
$V = 5720.4\ (13)$ Å^3
$Z = 4$
$D_x = 1.310$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10\text{–}13.5°$
$\mu = 0.871$ mm^{-1}
$T = 296$ K
Prism
0.30 × 0.24 × 0.20 mm
Colorless
Crystal source: toluene solution layered with hexane

Data collection

Siemens P3 diffractometer
ω scans
Absorption correction: empirical, ψ-scans (*XEMP* in *SHELXTL/PC*; Sheldrick, 1990)
$T_{min} = 0.677$, $T_{max} = 0.729$
8163 measured reflections
6398 independent reflections
4800 observed reflections [$F > 3.0\sigma(F)$]
$R_{int} = 0.0156$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 30$
$l = -21 \rightarrow 20$
3 standard reflections monitored every 97 reflections
intensity variation: 2%

Refinement

Refinement on F
$R = 0.076$
$wR = 0.076$
$S = 1.25$
4800 reflections
677 parameters
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}
Extinction correction: $F^* = F_c[1 + (0.002\chi \times F_c^2/\sin 2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.00004$

H-atom parameters not refined

$w = 1/[\sigma^2(F) + 0.0015(F)^2]$

$(\Delta/\sigma)_{max} = 0.001$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Cu(1)	0.3673 (1)	0.6426 (1)	0.2674 (1)	0.040 (1)
Cu(2)	0.3545 (1)	0.5355 (1)	0.2648 (1)	0.049 (1)
P(1)	0.3615 (2)	0.6836 (1)	0.3655 (1)	0.039 (1)
P(2)	0.3783 (2)	0.6727 (1)	0.1630 (1)	0.038 (1)
P(3)	0.3265 (2)	0.4623 (1)	0.2391 (1)	0.051 (1)
O(1)	0.2310 (5)	0.5890 (2)	0.2609 (3)	0.054 (2)
O(2)	0.4878 (5)	0.5834 (2)	0.2943 (3)	0.047 (2)
C(1)	0.3445 (8)	0.6481 (3)	0.4408 (4)	0.042 (3)
C(2)	0.3760 (9)	0.6009 (3)	0.4430 (5)	0.057 (4)
C(3)	0.3659 (10)	0.5729 (3)	0.4997 (5)	0.065 (4)
C(4)	0.3232 (9)	0.5926 (4)	0.5546 (5)	0.062 (4)
C(5)	0.2908 (9)	0.6396 (3)	0.5541 (5)	0.054 (4)
C(6)	0.2999 (8)	0.6670 (3)	0.4969 (4)	0.051 (4)
C(7)	0.2290 (8)	0.7259 (3)	0.3609 (4)	0.041 (3)
C(8)	0.1028 (9)	0.7085 (4)	0.3402 (4)	0.056 (4)
C(9)	−0.0018 (10)	0.7396 (5)	0.3351 (5)	0.072 (5)
C(10)	0.0191 (11)	0.7869 (4)	0.3483 (5)	0.068 (5)
C(11)	0.1414 (10)	0.8036 (4)	0.3695 (5)	0.062 (4)
C(12)	0.2464 (9)	0.7737 (3)	0.3753 (5)	0.052 (4)
C(13)	0.5043 (8)	0.7203 (3)	0.3946 (4)	0.041 (3)
C(14)	0.5513 (9)	0.7451 (3)	0.3448 (5)	0.049 (4)
C(15)	0.6575 (9)	0.7739 (3)	0.3633 (5)	0.056 (4)
C(16)	0.7192 (9)	0.7785 (4)	0.4301 (6)	0.073 (5)
C(17)	0.6735 (9)	0.7543 (4)	0.4814 (5)	0.073 (5)
C(18)	0.5664 (9)	0.7255 (3)	0.4629 (5)	0.064 (4)
C(19)	0.2386 (8)	0.7105 (3)	0.1277 (4)	0.038 (3)
C(20)	0.1944 (8)	0.7398 (3)	0.1743 (5)	0.047 (4)
C(21)	0.0877 (9)	0.7687 (3)	0.1519 (5)	0.064 (4)
C(22)	0.0280 (9)	0.7700 (4)	0.0847 (5)	0.064 (4)
C(23)	0.0688 (9)	0.7405 (4)	0.0384 (5)	0.069 (4)
C(24)	0.1750 (8)	0.7109 (3)	0.0592 (5)	0.053 (4)
C(25)	0.3842 (8)	0.6312 (3)	0.0925 (4)	0.046 (3)
C(26)	0.3356 (9)	0.5864 (3)	0.0950 (5)	0.058 (4)
C(27)	0.3416 (11)	0.5543 (4)	0.0426 (7)	0.088 (6)
C(28)	0.3878 (12)	0.5666 (5)	−0.0139 (6)	0.087 (6)
C(29)	0.4352 (11)	0.6122 (4)	−0.0174 (5)	0.075 (5)
C(30)	0.4317 (9)	0.6440 (3)	0.0338 (4)	0.057 (4)
C(31)	0.5167 (8)	0.7117 (3)	0.1609 (4)	0.040 (3)
C(32)	0.5046 (9)	0.7596 (3)	0.1448 (4)	0.049 (4)
C(33)	0.6161 (10)	0.7873 (3)	0.1496 (5)	0.060 (4)
C(34)	0.7349 (11)	0.7674 (4)	0.1694 (5)	0.070 (5)
C(35)	0.7490 (9)	0.7205 (4)	0.1849 (5)	0.065 (4)
C(36)	0.6402 (9)	0.6925 (3)	0.1810 (4)	0.050 (4)
C(37)	0.4750 (9)	0.4295 (3)	0.2360 (5)	0.052 (4)
C(38)	0.4842 (10)	0.3814 (3)	0.2480 (5)	0.061 (4)
C(39)	0.5991 (12)	0.3578 (4)	0.2461 (5)	0.075 (5)
C(40)	0.7016 (12)	0.3821 (4)	0.2338 (5)	0.078 (5)
C(41)	0.6969 (12)	0.4302 (5)	0.2227 (7)	0.098 (6)
C(42)	0.5815 (11)	0.4536 (3)	0.2245 (6)	0.076 (5)
C(43)	0.2582 (10)	0.4314 (3)	0.3048 (5)	0.054 (4)
C(44)	0.1414 (12)	0.4084 (4)	0.2922 (6)	0.087 (5)
C(45)	0.0931 (12)	0.3884 (4)	0.3460 (8)	0.097 (6)
C(46)	0.1595 (16)	0.3918 (4)	0.4116 (7)	0.097 (7)
C(47)	0.2785 (15)	0.4125 (5)	0.4258 (6)	0.103 (7)
C(48)	0.3242 (11)	0.4330 (4)	0.3721 (6)	0.084 (5)
C(49)	0.2168 (11)	0.4482 (3)	0.1575 (5)	0.064 (4)
C(50)	0.2441 (14)	0.4152 (4)	0.1111 (6)	0.109 (6)
C(51)	0.1562 (18)	0.4065 (5)	0.0504 (7)	0.156 (9)
C(52)	0.0423 (17)	0.4292 (5)	0.0352 (7)	0.149 (9)
C(53)	0.0157 (13)	0.4633 (5)	0.0777 (7)	0.113 (6)
C(54)	0.1034 (11)	0.4732 (4)	0.1389 (6)	0.082 (5)
C(55)	0.1051 (8)	0.5861 (3)	0.2354 (5)	0.043 (3)
C(56)	0.0275 (9)	0.5552 (3)	0.2647 (5)	0.058 (4)
C(57)	−0.1011 (9)	0.5497 (3)	0.2367 (5)	0.061 (4)
C(58)	−0.1600 (9)	0.5743 (3)	0.1781 (5)	0.055 (4)
C(59)	−0.0856 (9)	0.6055 (3)	0.1515 (5)	0.058 (4)
C(60)	0.0420 (9)	0.6122 (3)	0.1794 (5)	0.057 (4)
C(61)	−0.3015 (9)	0.5651 (4)	0.1458 (6)	0.088 (5)
C(62)	0.6068 (8)	0.5803 (3)	0.3332 (4)	0.038 (3)
C(63)	0.6551 (8)	0.5375 (3)	0.3640 (5)	0.056 (4)
C(64)	0.7807 (9)	0.5345 (4)	0.4016 (5)	0.073 (5)
C(65)	0.8624 (9)	0.5728 (4)	0.4116 (5)	0.061 (4)
C(66)	0.8149 (9)	0.6160 (4)	0.3833 (5)	0.062 (4)
C(67)	0.6896 (9)	0.6189 (3)	0.3449 (5)	0.055 (4)
C(68)	1.0021 (9)	0.5688 (4)	0.4513 (6)	0.098 (6)

Table 2. *Selected geometric parameters* (Å, °)

Cu(1)—P(1)	2.258 (2)	Cu(1)—P(2)	2.244 (2)
Cu(1)—O(1)	2.071 (6)	Cu(1)—O(2)	2.097 (5)
Cu(2)—P(3)	2.129 (3)	Cu(2)—O(1)	1.986 (6)
Cu(2)—O(2)	1.952 (5)	P(1)—C(1)	1.827 (9)
P(1)—C(7)	1.826 (9)	P(1)—C(13)	1.821 (8)
P(2)—C(19)	1.837 (8)	P(2)—C(25)	1.822 (9)
P(2)—C(31)	1.836 (9)	P(3)—C(37)	1.832 (10)
P(3)—C(43)	1.823 (11)	P(3)—C(49)	1.820 (9)
O(1)—C(55)	1.324 (9)	O(2)—C(62)	1.334 (9)
P(1)—Cu(1)—P(2)	127.0 (1)	P(1)—Cu(1)—O(1)	106.6 (2)
P(1)—Cu(1)—O(2)	108.2 (2)	P(2)—Cu(1)—O(1)	112.6 (2)
P(2)—Cu(1)—O(2)	112.9 (2)	O(1)—Cu(1)—O(2)	79.3 (2)
P(3)—Cu(2)—O(1)	132.0 (2)	P(3)—Cu(2)—O(2)	142.9 (2)
O(1)—Cu(2)—O(2)	85.0 (2)	Cu(1)—O(1)—Cu(2)	96.3 (2)
Cu(1)—O(1)—C(55)	134.5 (5)	Cu(2)—O(1)—C(55)	124.6 (5)
Cu(1)—O(2)—Cu(2)	96.5 (2)	Cu(1)—O(2)—C(62)	130.4 (5)
Cu(2)—O(2)—C(62)	131.4 (5)		

Table 3. *Comparison of core distances* (Å)

	$Cu_2(OC_6H_4Me\text{-}4)_2(PPh_3)_3$	$Cu_2(OC_6H_4NO_2\text{-}4)_2(PPh_3)_3$
Cu(1)—P	2.244 (2)	2.259
	2.258 (2)	2.259
Cu(2)—P	2.129 (3)	2.153
Cu(1)—O	2.071 (6)	2.132
	2.097 (5)	2.175
Cu(2)—O	1.952 (5)	1.998
	1.986 (2)	2.005

Background counts were estimated from a 96-step profile using the program *CARESS* as implemented in the *UCLA Crystallographic Computing Package* (Strouse, 1981). All subsequent crystallographic calculations (empirical absorption correction, structure solution, refinement, *etc.*) were performed using the Siemens *SHELXTL/PC* program set (Sheldrick, 1990). H atoms were included using a riding model with C—H = 0.95 Å and U_{iso} = 0.08 Å^2.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71806 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1062]

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