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Structure of Tetrakis(triphenylphosphine oxide)lithium Dibromocuprate(I)

BY JOHN P. FACKLER JR,* CÉSAR A. LÓPEZ AND RICHARD E. P. WINPENNY

Department of Chemistry and Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, Texas 77843, USA

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Abstract. $[Li{OP(C_6H_5)_3}_4][CuBr_2], M_r = 1343.4,$ triclinic. $P\overline{1}$, a = 14.60(1), b = 16.28(1), c =17.13 (1) Å, $\alpha = 70.20(5), \quad \beta = 69.07(6),$ $\gamma =$ $V = 3328 (5) \text{ Å}^3$, Z = 2, $D_x =$ 63.68 (6)°, 1.34 Mg m^{-3} , $\lambda(\text{Mo }K\alpha) = 0.71073 \text{ Å},$ $\mu =$ 1.66 mm^{-1} , F(000) = 1372, T = 293 K, R = 0.0473for 3163 unique observed reflections. The structure consists of a tetrahedrally coordinated $[Li(OPPh_3)_4]^+$ (Li $-O \approx 1.90$ Å) cation in a general position and two linear $[CuBr_2]^-$ anions with the Cu atoms on inversion centers.

Experimental. Reaction of methyllithium (1.4 M solution in diethyl ether) with damp triphenylphosphine oxide in THF gave a yellow solution, which gave a dark green precipitate upon addition of copper(II) bromide. The mixture was filtered and colorless crystals were obtained by recrystallization from a tetrahydrofuran/diethyl ether solution.

A crystal with dimensions $0.25 \times 0.25 \times 0.30$ mm was mounted on a glass fiber. Intensity data were collected on a Nicolet R3m/E diffractometer (graphite-monochromated Mo $K\alpha$ radiation) using the ω -scanning technique in bisecting geometry. Refined cell parameters were obtained from the setting angles of 23 reflections with $5 < 2\theta < 20^{\circ}$. Intensities were measured for 4617 unique reflections (h 0to 15, k - 14 to 16, l - 15 to 17) with $2 < 2\theta < 40^{\circ}$ (scan rate variable, $3-30^{\circ}$ min⁻¹). The intensities of three check reflections $(23\overline{5}, \overline{2}1\overline{1} \text{ and } \overline{1}\overline{2}\overline{5})$ varied less than 2%. Data were corrected for Lorentz and polarization effects. No absorption correction was applied. The structure was solved by direct methods and subsequent electron-density difference maps. Non-H atoms were refined anisotropically. Phenyl rings were refined as hexagons (C—C = 1.395 Å, $C-C-C = 120^{\circ}$). H atoms were included in calculated positions (C—H 0.96 Å), assigned isotropic thermal parameters of $U(H) = 1.2U_{eq}(C)$, and allowed to ride on their parent C atoms. Blockcascade-matrix least-squares refinement gave R =0.0473, wR = 0.0495 and S = 1.422 for 3163 reflections with $F > 2.5\sigma(F)$, and 616 refined parameters. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00016F^2$. Maximum and minimum residual electron densities were 0.26 and $-0.28 \text{ e} \text{Å}^{-3}$, respectively. All calculations were performed using *SHELXTL* (Sheldrick, 1985).

Fractional atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,[†] and selected bond distances and angles in Table 2. Table

[†] 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 55427 (45 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0305]



Fig. 1. $[Li(OPPh_3)_4][CuBr_2]$ showing the atom-labeling scheme. Phenyl-group C atoms are of arbitrary radii. Thermal ellipsoids have been drawn at the 50% probability level. H atoms have been omitted for clarity. The $[CuBr_2]$ anion is shown with the Cu atom at the inversion center.

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^{*} Author to whom correspondence should be addressed.

Table 1. Atomic coordinates $(\times 10^4)$ and equivalent isotropic thermal parameters $(Å^2 \times 10^3)$ for $[Li(OPPh_3)_4][CuBr_2]$

 $U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	у	Z	U_{eq}
Li	2501 (10)	7361 (9)	2535 (8)	51 (8)
P(1)	947 (2)	7307 (2)	4534 (1)	47 (1)
O(1)	1710 (4)	7242 (4)	3696 (3)	64 (4)
C(12)	- 1246 (6)	8215 (4)	5029 (3)	69 (6)
C(13)	- 2261 (6)	8507 (4)	4934 (3)	77 (7)
C(14)	- 2412 (6)	8293 (4)	4277 (3)	89 (8)
C(15)	- 1546 (6)	7787 (4)	3715 (3)	121 (9)
C(16)	- 531 (6)	7494 (4)	3810 (3)	98 (8)
C(11)	- 381 (6)	7708 (4)	4467 (3)	49 (5)
C(22)	2248 (6)	5560 (7)	5117 (4)	86 (8)
C(23)	2518 (6)	4673 (7)	5657 (4)	110 (9)
C(24)	1/44 (6)	4414 (7)	6330 (4)	135 (11)
C(25)	/00 (6)	5027 (7)	0401 (4) 5021 (4)	180 (13)
C(20)	431 (0)	5927 (7)	5248 (4)	130 (9)
C(21)	662 (4)	8081 (4)	5885 (5)	60 (6)
C(33)	754 (4)	8716 (4)	6205 (5)	76 (7)
C(34)	1210 (4)	9363 (4)	5651 (5)	82 (7)
C(35)	1573 (4)	9376 (4)	4777 (5)	86 (8)
C(36)	1481 (4)	8741 (4)	4456 (5)	65 (6)
C(31)	1025 (4)	8094 (4)	5011 (5)	49 (5)
P(2)	2150 (2)	9585 (2)	1620 (2)	60 (2)
O(2)	2528 (4)	8600 (3)	2093 (3)	71 (4)
C(42)	151 (8)	9587 (4)	2326 (4)	70 (7)
C(43)	- 937 (8)	9950 (4)	2423 (4)	93 (9)
C(44)	- 1401 (8)	10768 (4)	1865 (4)	138 (11)
C(45)	- 776 (8)	11222 (4)	1208 (4)	199 (13)
C(46)	312 (8)	10859 (4)	1111 (4)	162 (10)
C(41)	776 (8)	10042 (4)	1670 (4)	73 (7)
C(52)	2809 (5)	9122 (4)	/5 (/)	84 (8)
C(53)	3341 (3)	9159 (4)	- /80 (/)	112 (10)
C(54)	3007 (3)	9709 (4)	-1200(7)	01 (9)
C(55)	3368 (5)	10345 (4)	96 (7)	75 (7)
C(50)	2822 (5)	9696 (4)	516 (7)	60 (6)
C(62)	1745 (5)	11286 (6)	2035 (4)	86 (7)
C(63)	1971 (5)	11783 (6)	2418 (4)	109 (10)
C(64)	2811 (5)	11333 (6)	2809 (4)	118 (11)
C(65)	3424 (5)	10387 (6)	2817 (4)	108 (10)
C(66)	3198 (5)	9890 (6)	2433 (4)	83 (8)
C(61)	2358 (5)	10339 (6)	2042 (4)	58 (6)
P(3)	1775 (2)	6505 (2)	1521 (1)	50 (1)
O(3)	1790 (4)	7184 (3)	1910 (3)	58 (3)
C(72)	- 136 (7)	7794 (5)	1318 (4)	74 (7)
C(73)	- 1151 (7)	8125 (5)	1207 (3)	95 (8)
C(74)	- 1548 (7)	/501 (5)	11/8 (3)	88 (8)
C(75)	- 930 (7)	6347 (3)	1200(3)	89 (8) 78 (7)
C(71)	482 (7)	6840 (5)	1309 (3)	54 (6)
C(82)	2672 (5)	4546 (6)	1788 (3)	71 (6)
C(83)	3000 (5)	3652 (6)	2306 (3)	89 (8)
C(84)	2817 (5)	3547 (6)	3185 (3)	85 (8)
C(85)	2305 (5)	4336 (6)	3545 (3)	87 (8)
C(86)	1976 (5)	5230 (6)	3027 (3)	70 (7)
C(81)	2160 (5)	5335 (6)	2148 (3)	50 (6)
C(92)	2438 (4)	6427 (4)	- 213 (5)	70 (6)
C(93)	3191 (4)	6387 (4)	- 993 (5)	90 (8)
C(94)	4184 (4)	6360 (4)	- 1062 (5)	98 (9)
C(95)	4426 (4)	6372 (4)	- 349 (5)	98 (8)
C(96)	3673 (4)	6411 (4)	432 (5)	79 (7)
C(91)	2680 (4)	6439 (4)	500 (5) 2424 (1)	50 (6)
P(4)	4990 (2)	6233 (2)	2424 (1)	57 (1)
C(102)	5000 (4) 6730 (6)	5948 (4)	2477 (3)	69 (6)
C(102)	7248 (6)	6768 (4)	149 (5)	85 (8)
C(103)	6766 (6)	7165 (4)	-308(5)	92 (9)
C(105)	5766 (6)	7741 (4)	65 (5)	91 (8)
C(106)	5247 (6)	7420 (4)	896 (5)	66 (7)
C(101)	5729 (6)	6524 (4)	1353 (5)	49 (6)
C(112)	4310 (5)	7096 (5)	3756 (5)	91 (8)
C(113)	4339 (5)	7636 (5)	4222 (5)	116 (9)
C(114)	5138 (5)	7995 (5)	3935 (5)	97 (9)
C(115)	5908 (5)	7815 (5)	3181 (5)	97 (8)
C(116)	5879 (5)	7274 (5)	2714 (5)	67 (6)
C(111)	5080 (5)	6915 (5)	3002 (5)	56 (6)
C(122)	0043 (6)	4030 (0)	2904 (4)	85 (/)
C(123)	/119 (0) 6572 (6)	3082 (6)	3233 (4)	112 (9)
C(124) C(125)	5549 (6)	3454 (6)	3344 (4)	92 (8)
C(125)	JJ47 (U)	5454 (0)	JJ (-)	74 (0)

Table 1 (cont.)

	x	у	z	U_{eq}
C(126)	5073 (6)	4417 (6)	3044 (4)	72 (7)
C(121)	5620 (6)	5008 (6)	2849 (4)	55 (6)
Cu(2)	0	5000	0	128 (2)
Br(2)	- 323 (1)	3938 (1)	1170 (1)	153 (1)
Cu(1)	5000	0	5000	125 (2)
Br(1)	5352 (1)	850 (1)	3697 (1)	160 (1)

Table 2. Bond lengths (Å) and angles (°) for [Li(OPPh₃)₄][CuBr₂]

$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{l} \text{Li-O(1)} \\ \text{Li-O(3)} \\ P(1)-O(1) \\ P(1)-O(2) \\ P(2)-O(2) \\ P(2)-O(2) \\ P(3)-O(3) \\ P(3)-O(3) \\ P(3)-O(4) \\ P(4)-O(4) \\ P(4)-O(4) \\ P(4)-O(11) \\ Cu(2)-Br(2) \\ Cu(1)-Br(1) \end{array}$	1.904 (13) 1.890 (20) 1.473 (5) 1.781 (8) 1.787 (8) 1.780 (10) 1.480 (7) 1.480 (7) 1.487 (8) 1.475 (6) 1.787 (8) 1.2219 (1) 2.219 (1)	$\begin{array}{c} \text{Li-O(2)} \\ \text{Li-O(4)} \\ P(1)-C(11) \\ P(1)-C(31) \\ P(2)-C(41) \\ P(2)-C(61) \\ P(3)-C(71) \\ P(3)-C(91) \\ P(4)-C(101) \\ P(4)-C(121) \\ Cu(2)-Br(2a) \\ Cu(1)-Br(1a) \end{array}$	1.912 (15) 1.894 (12) 1.784 (9) 1.786 (9) 1.784 (11) 1.794 (12) 1.789 (10) 1.779 (8) 1.791 (7) 1.794 (8) 2.220 (1) 2.209 (1)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	$\begin{array}{l} O(1)-Li-O(2)\\ O(2)-Li-O(3)\\ O(2)-Li-O(4)\\ O(1)-P(1)-C(11)\\ C(11)-P(1)-C(21)\\ C(11)-P(1)-C(31)\\ Li-O(1)-P(1)\\ P(1)-C(21)-C(26)\\ P(1)-C(31)-C(36)\\ O(2)-P(2)-C(51)\\ O(2)-P(2)-C(51)\\ O(2)-P(2)-C(51)\\ O(2)-P(2)-C(51)-C(52)\\ P(2)-C(41)-C(42)\\ P(2)-C(51)-C(52)\\ P(2)-C(51)-C(52)\\ P(2)-C(51)-C(52)\\ P(2)-C(61)-C(62)\\ O(3)-P(3)-C(71)\\ C(71)-P(3)-C(81)\\ C(71)-P(3)-C(91)\\ Li-O(3)-P(3)\\ P(3)-C(71)-C(76)\\ P(3)-C(71)-C(76)\\ P(3)-C(81)-C(80)\\ P(3)-C(11)-C(10)\\ O(4)-P(4)-C(121)\\ C(111)-P(4)-C(121)\\ C(111)-P(4)-C(121)\\ C(111)-P(4)-C(121)\\ C(111)-P(4)-C(121)\\ C(111)-P(4)-C(121)\\ C(110)-C(100)\\ C(101)-C(100)\\ C(100)-C(100)\\ C(100)-C(100$	109.7 (8) 108.3 (7) 111.3 (8) 112.8 (3) 106.6 (4) 107.9 (3) 169.4 (5) 116.6 (2) 123.5 (2) 116.5 (2) 111.1 (3) 111.1 (3) 111.1 (4) 107.9 (4) 117.8 (2) 110.0 (3) 108.7 (4) 108.5 (4) 144.7 (5) 124.2 (3) 117.3 (3) 116.3 (3) 111.0 (3) 109.9 (4) 109.5 (4) 125.2 (2)	$\begin{array}{c} O(1)-Li-O(3)\\ O(1)-Li-O(4)\\ O(3)-Li-O(4)\\ O(1)-P(1)-C(21)\\ O(1)-P(1)-C(21)\\ O(1)-P(1)-C(31)\\ P(1)-C(1)-C(12)\\ P(1)-C(21)-C(22)\\ P(1)-C(21)-C(22)\\ P(1)-C(21)-C(22)\\ O(2)-P(2)-C(41)\\ C(41)-P(2)-C(51)\\ C(41)-P(2)-C(51)\\ C(41)-P(2)-C(51)\\ C(41)-P(2)-C(51)\\ C(41)-P(2)-C(51)\\ C(41)-P(2)-C(51)\\ C(3)-P(3)-C(81)\\ O(3)-P(3)-C(91)\\ C(81)-P(3)-C(91)\\ C(81)-P(3)-C(91)\\ C(81)-P(3)-C(91)\\ P(3)-C(91)-C(92)\\ P(3)-C$	107.1 (8) 108.9 (7) 111.4 (8) 110.7 (3) 110.8 (4) 107.9 (4) 123.4 (2) 116.5 (2) 123.5 (2) 113.0 (4) 106.8 (4) 106.8 (4) 106.7 (4) 133.6 (7) 122.0 (2) 122.9 (3) 114.0 (2) 112.9 (4) 105.1 (3) 115.8 (3) 112.9 (4) 109.4 (3) 109.4 (4) 109.4 (4)
	P(4)—C(111)—C(112) P(4)—C(121)—C(122) Br(2)—Cu(2)—Br(2a)	117.9 (2) 121.9 (4) 180.0	P(4)—C(111)—C(116 P(4)—C(121)—C(126 Br(1)—Cu(1)—Br(1a	5) 122.1 (2) 5) 118.1 (4)) 180.0

Table 3. Torsion angles (°) for [Li(OPPh₃)₄][CuBr₂]

O(2) - Li - O(1) - P(1)	52.5 (4.6)	O(1) - P(1) - C(31) - C(36)	20.7 (0.3)
O(1) - Li - O(2) - P(2)	- 96.6 (1.4)	O(2) - P(2) - C(41) - C(42)	19.8 (0.4)
O(1) - Li - O(3) - P(3)	- 100.5 (0.8)	C(41) - P(2) - O(2) - Li	30.6 (1.2)
O(1) - Li - O(4) - P(4)	- 98.7 (1.0)	C(51) - P(2) - O(2) - Li	- 89.4 (1.2)
O(3) - Li - O(1) - P(1)	- 64.9 (4.4)	O(2) - P(2) - C(41) - C(46)	- 164.2 (0.3
O(3) - Li - O(2) - P(2)	20.0 (1.4)	C(61) - P(2) - O(2) - Li	150.5 (1.1)
O(2) - Li - O(3) - P(3)	141.3 (0.6)	C(51) - P(2) - C(41) - C(42)	142.2 (0.3
O(2) - Li - O(4) - P(4)	22.3 (1.5)	C(71) - P(3) - O(3) - Li	157.6 (0.8
O(4) - Li - O(1) - P(1)	174.6 (3.6)	C(81)—P(3)—O(3)—Li	36.4 (0.9)
O(4) - Li - O(2) - P(2)	142.8 (0.9)	C(91) - P(3) - O(3) - Li	- 81.6 (0.9
O(4) - Li - O(3) - P(3)	18.5 (1.2)	C(101) - P(4) - O(4) - Li	- 83.4 (1.0)
O(3)—Li—O(4)—P(4)	143.3 (0.8)	C(111)-P(4)-O(4)-Li	34.1 (1.0)
C(11) - P(1) - O(1) - Li	36.5 (4.3)	C(121)-P(4)-O(4)-Li	155.4 (0.9
C(21) - P(1) - O(1) - Li	155.8 (4.1)	C(21) - P(1) - C(11) - C(16)	- 92.3 (0.3)
C(31) - P(1) - O(1) - Li	- 84.5 (4.2)	O(1) - P(1) - C(11) - C(12)	- 150.5 (0.3)
O(1) - P(1) - C(11) - C(12)	- 150.5 (0.3)	O(1) - P(1) - C(21) - C(22)	28.5 (0.5
O(1) - P(1) - C(11) - C(16)	29.4 (0.4)	C(11) - P(1) - C(21) - C(26)	- 29.5 (0.4
O(1) - P(1) - C(21) - C(22)	28.5 (0.5)	O(1) - P(1) - C(31) - C(32)	- 159.2 (0.2)
O(1) - P(1) - C(21) - C(26)	- 152.5 (0.3)	C(11) - P(1) - C(31) - C(36)	- 103.2 (0.2)
O(1) - P(1) - C(31) - C(32)	- 159.2 (0.2)		

3 gives torsion angles for the complex. Fig. 1 shows a view of the compound.

Related literature. The crystal structure of the tetrahedral cation $[Li(OPPh_3)_4]^+$ has been reported previously (Yasin, Hodder & Powell, 1966), although the Li-atom position was not well defined and Li-O bond lengths were inferred to be approximately 1.97 Å, considerably longer than the 1.90 Å reported here. Other phosphine oxide complexes of Li include of $Ph_2P(O)PP(O)Ph_2$ (Schmidpeter, complexes Burget, von Schnering & Weber, 1984) and of N[CH₂CH₂P(O)Ph₂]₃ (Ghilardi, Innocenti, Midollini & Orlandini, 1986). The P-O distance in Ph₃PO is 1.483 Å (Ruban & Zabel, 1976), slightly longer than the one reported here (1.469 Å). $[CuBr_2]^-$ anions have also been reported previously (Asplund, Jagner & Nilsson, 1983; Cecconi, Ghilardi, Midollini & Orlandini, 1983). Iodide has also been reported as a counterion (Issleib & Krech, 1964).

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Structure of (4-Pentynoato-O,O'){tris[2-(diphenylphosphino)ethyl]phosphine}iron(II) Tetraphenylborate

BY KLAUS LINN, DANTE MASI, CARLO MEALLI, CLAUDIO BIANCHINI AND MAURIZIO PERUZZINI

Istituto per lo Studio della Stereochimica ed Energetica dei Composti di Coordinazione, CNR, Via J. Nardi 39, 50132 Firenze, Italy

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Abstract. [Fe(C₅H₅O₂)(C₄₂H₄₂P₄)][B(C₆H₅)₄], $M_r = 1142.9$, monoclinic, $P2_1/a$, a = 26.296 (3), b = 12.304 (4), c = 19.061 (2) Å, $\beta = 103.63$ (2)°, V = 5993.4 (22) Å³, Z = 4, λ (Cu $K\alpha$) = 1.5418 Å, $\mu = 33.9$ cm⁻¹, F(000) = 2404, room temperature, final R = 0.086 for 4321 significant reflections. The molecular structure consists of tetraphenylborate anions and discrete complex cations in which an Fe^{II} metal is pseudo-octahedrally coordinated by the ligand pp₃ [pp₃ = P(CH₂CH₂PPh₂)₃] and by a 4-pentynoate group. The latter ligand is bonded in a chelating mode through the two O atoms.

Introduction. As part of our ongoing studies into the factors that control the oxidative cleavage of C—H bonds as opposed to O—H bonds at metal centers (Bianchini, Masi, Meli, Peruzzini, Ramirez, Vacca & Zanobini, 1989), we have reacted the hydride dinitrogen complex [(pp₃)Fe(H)(η^1 -N₂)]BPh₄ (Bianchini, Peruzzini & Zanobini, 1988) with a stoichiometric amount of 4-pentynoic acid, HCCCH₂CH₂COOH.

As a result the novel carboxylate complex $[(pp_3)Fe-(O_2CCH_2CH_2CCH)]BPh_4$ has been synthesized as cherry red crystals. The product has also been characterized by spectroscopic techniques.

Experimental. Selected spectroscopic data for the $^{31}P[^{1}H]$ NMR (CD₂Cl₂, title compound are: 121.42 MHz, 173 K); ABCD spin system, δ_A 164.92, δ_B 90.2, δ_C 68.06, δ_D 57.22; J_{AB} 10.0, J_{AC} 21.2, J_{AD} 21.0, J_{BC} 32.5, J_{BD} 33.1, J_{CD} 148.0 Hz. ¹H NMR $(CD_2Cl_2, 299.94 \text{ MHz}, 294 \text{ K}) \delta 1.79 (t, 1H, C = CH,)$ ${}^{4}J_{\rm HH}$ 2.30 Hz), δ 1.33 (td, 2H, CH₂C=C, ${}^{3}J_{\rm HH}$ 7.70 Hz), δ 0.92 (t, 2H, CH₂CH₂). ¹³C[¹H] NMR (CD₂Cl₂, 50.32 MHz, 294 K), δ 186.05 (s, OOC), δ 83.23 (s, C=CH), δ 36.70 (s, OOCCH₂), δ 13.07 (s, CH_2CH_2 ; the assignment of the ¹³C resonances was confirmed by a DEPT 135° experiment; the quaternary C atom of the acetylene was not detected as it is obscured by the resonances of the aromatic C atoms in the 138–122 p.p.m. region of the spectrum. IR: ν (C=CH) 3276 cm⁻¹ (m), ν (C=C) 2118 cm⁻¹ (vw),

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