

Fig. 1. PLUTO diagram (Motherwell & Clegg, 1978) of the unit cell of Li<sub>2</sub>NaK(SO<sub>4</sub>)<sub>2</sub> viewed along the *x* crystallographic axis.

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## Tricarbonyl( $\eta^2$ -cyclenphosphanido)cobalt(I)

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**Abstract.** Tricarbonyl(octahydro-2a,4a,6a,8a-tetraaza-8b-phosphapentaleno[6,1-*cd*]pentalene-*N,P*)cobalt(I), [Co(CO)<sub>3</sub>(C<sub>8</sub>H<sub>16</sub>N<sub>4</sub>P)], *M<sub>r</sub>* = 342.18, orthorhombic, *Pca*2<sub>1</sub>, *a* = 15.599 (4), *b* = 7.445 (2), *c* = 12.216 (3) Å, *V* = 1418.7 (7) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.602 g cm<sup>-3</sup>,  $\lambda$ (Mo *K* $\alpha$ ) = 0.71069 Å,  $\mu$ (Mo *K* $\alpha$ ) = 13.28 cm<sup>-1</sup>, *F*(000) = 704, *T* = 295 K, final *R* = 0.035 for 927 observed reflections with *I* > 3 $\sigma$ (*I*). The cyclenphosphanido ligand coordinates to the Co atom through P and one of its N atoms with Co–P and Co–N bonds of 2.195 (2) and 2.043 (6) Å, respectively. Three carbonyl groups complete the coordination at the Co atom with Co–C bonds ranging from 1.763 (9) to 1.801 (8) Å.

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crystallization and Raman scattering studies of this compound have already been published (Kitahama & Frech, 1985).

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### References

- ALCOCK, N. W., EVANS, D. A. & JENKINS, H. D. B. (1973). *Acta Cryst.* **B29**, 360–361.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- KITAHAMA, K. & FRECH, R. (1985). *J. Chem. Phys.* **82**, 720–725.
- MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J.-P. (1976). *MULTAN76. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- OKADA, K. & OSSAKA, J. (1980). *Acta Cryst.* **B36**, 919–921.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- ZILBER, R., DURIF, A. & AVERBUCH-POUCHOT, M. (1980). *Acta Cryst.* **B36**, 2743–2745.
- ZILBER, R., TORDJMAN, I. & GUITEL, J. C. (1980). *Acta Cryst.* **B36**, 2741–2743.

**Experimental.** Crystals of the title compound are yellow plates. Unit-cell parameters by least-squares fit of 15 reflections in the range  $14 < 2\theta < 24^\circ$ . Crystal 0.30 × 0.24 × 0.06 mm, Syntex *P2*<sub>1</sub> automatic diffractometer, graphite-monochromatized Mo *K* $\alpha$  radiation,  $\theta/2\theta$  scanning mode, 1324 independent reflections in the range  $3 < 2\theta < 50^\circ$ , *hkl* range: *h* 0–18, *k* 0–8, *l* 0–14, 927 observed reflections with *I* > 3 $\sigma$ (*I*),  $\sigma$ (*I*) from counting statistics; three standard reflections (200, 020 and 002) remeasured after every 100 reflections did not show any significant change in intensity during data collection. Lorentz–polarization correction, no absorption or extinction correction. Co position located by the heavy-atom method; its *z* coordinate was arbitrarily fixed at  $\frac{1}{2}$  to specify the origin. Remaining non-H atoms located by difference Fourier methods.

Full-matrix least-squares refinement using *SHELX76* (Sheldrick, 1976), function minimized  $\sum w(|F_o| - |F_c|)^2$ , non-H atoms anisotropic, all H atoms located and included in refinement as fixed-atom contribution

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors for the non-H atoms with *e.s.d.*'s in parentheses

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Co	0.10850 (5)	-0.0924 (1)	$\frac{1}{2}$	0.0445 (1)
P	0.0669 (1)	0.1878 (2)	0.5171 (2)	0.0387 (3)
N(1)	0.1415 (4)	0.1277 (7)	0.4095 (5)	0.0499 (14)
C(1)	0.2275 (4)	0.2080 (10)	0.4268 (7)	0.0634 (22)
C(2)	0.2266 (5)	0.2815 (11)	0.5392 (8)	0.0748 (25)
N(2)	0.1414 (4)	0.3214 (8)	0.5705 (6)	0.0542 (14)
C(3)	0.1243 (8)	0.3615 (12)	0.6874 (8)	0.0847 (30)
C(4)	0.0290 (7)	0.3604 (14)	0.6985 (9)	0.0894 (31)
N(3)	-0.0049 (7)	0.2393 (11)	0.6193 (6)	0.0629 (21)
C(5)	-0.0902 (8)	0.2835 (19)	0.5764 (10)	0.0848 (34)
C(6)	-0.0923 (7)	0.2438 (19)	0.4606 (10)	0.0804 (32)
N(4)	-0.0062 (5)	0.2418 (10)	0.4197 (6)	0.0516 (18)
C(7)	0.0097 (6)	0.1555 (11)	0.3151 (7)	0.0656 (24)
C(8)	0.1042 (6)	0.1476 (11)	0.3012 (7)	0.0702 (25)
C(9)	0.1668 (5)	-0.1071 (10)	0.6233 (9)	0.0594 (24)
O(9)	0.2003 (4)	-0.1259 (10)	0.7064 (5)	0.0893 (20)
C(10)	0.1573 (5)	-0.2628 (11)	0.4160 (7)	0.0575 (21)
O(10)	0.1865 (5)	-0.3674 (8)	0.3598 (6)	0.0934 (20)
C(11)	0.0028 (5)	-0.1756 (9)	0.5073 (9)	0.0581 (16)
O(11)	-0.0648 (3)	-0.2298 (8)	0.5175 (8)	0.0910 (18)

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

Co-P	2.195 (2)	C(2)-N(2)	1.414 (11)
Co-N(1)	2.043 (6)	N(2)-C(3)	1.483 (12)
Co-C(9)	1.763 (9)	C(3)-C(4)	1.493 (15)
Co-C(10)	1.801 (8)	C(4)-N(3)	1.424 (14)
Co-C(11)	1.764 (9)	N(3)-C(5)	1.467 (16)
P-N(1)	1.812 (6)	C(5)-C(6)	1.446 (18)
P-N(2)	1.663 (7)	C(6)-N(4)	1.433 (15)
P-N(3)	1.720 (9)	N(4)-C(7)	1.452 (12)
P-N(4)	1.696 (8)	C(7)-C(8)	1.485 (12)
N(1)-C(1)	1.484 (10)	C(9)-O(9)	1.150 (11)
N(1)-C(8)	1.453 (11)	C(10)-O(10)	1.134 (11)
C(1)-C(2)	1.478 (11)	C(11)-O(11)	1.136 (11)
P-Co-N(1)	50.5 (2)	P-N(1)-C(1)	112.2 (5)
P-Co-C(9)	97.5 (3)	P-N(1)-C(8)	112.2 (5)
P-Co-C(10)	148.1 (3)	C(1)-N(1)-C(8)	116.8 (6)
P-Co-C(11)	93.0 (3)	N(1)-C(1)-C(2)	105.8 (6)
N(1)-Co-C(9)	112.5 (3)	C(1)-C(2)-N(2)	109.7 (7)
N(1)-Co-C(10)	98.6 (3)	P-N(2)-C(2)	115.1 (5)
N(1)-Co-C(11)	123.0 (3)	P-N(2)-C(3)	111.9 (6)
C(9)-Co-C(10)	103.0 (4)	C(2)-N(2)-C(3)	118.1 (7)
C(9)-Co-C(11)	114.7 (4)	N(2)-C(3)-C(4)	105.4 (8)
C(10)-Co-C(11)	100.2 (4)	C(3)-C(4)-N(3)	108.2 (9)
Co-P-N(1)	60.4 (2)	P-N(3)-C(4)	113.1 (7)
Co-P-N(2)	113.5 (2)	P-N(3)-C(5)	112.4 (7)
Co-P-N(3)	118.3 (3)	C(4)-N(3)-C(5)	115.9 (9)
Co-P-N(4)	110.9 (3)	N(3)-C(5)-C(6)	108.9 (10)
N(1)-P-N(2)	89.1 (3)	C(5)-C(6)-N(4)	108.8 (10)
N(1)-P-N(3)	178.5 (4)	P-N(4)-C(6)	112.8 (7)
N(1)-P-N(4)	88.9 (3)	P-N(4)-C(7)	113.4 (6)
N(2)-P-N(3)	92.1 (4)	C(6)-N(4)-C(7)	118.1 (8)
N(2)-P-N(4)	127.1 (4)	N(4)-C(7)-C(8)	106.7 (7)
N(3)-P-N(4)	91.1 (4)	N(1)-C(8)-C(7)	107.3 (7)
Co-N(1)-P	69.1 (2)	Co-C(9)-O(9)	174.8 (8)
Co-N(1)-C(1)	118.3 (4)	Co-C(10)-O(10)	177.4 (8)
Co-N(1)-C(8)	118.3 (5)	Co-C(11)-O(11)	176.6 (8)

with  $U$ 's set at  $0.08 \text{\AA}^2$ ;  $w = 1/(\sigma^2 + 0.0007F^2)$ ,  $R = 0.035$ ,  $wR = 0.036$  for the 927 observed reflections,  $S = 1.06$ .  $(\Delta/\sigma)_{\max} = 0.09$ ,  $\Delta\rho_{\max,\min} = 0.34, -0.27 \text{ e \AA}^{-3}$ . Atomic scattering factors for C, H, O, N and P, and the effects of the anomalous-dispersion correction for P, were those stored in *SHELX76*. For Co these values were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1. Bond lengths and bond angles are listed in Table 2\*. Fig. 1 shows the molecule and the numbering scheme adopted, and Fig. 2 the packing of the molecules in the cell.

\* Lists of structure factors, anisotropic thermal parameters and fractional coordinates of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43273 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

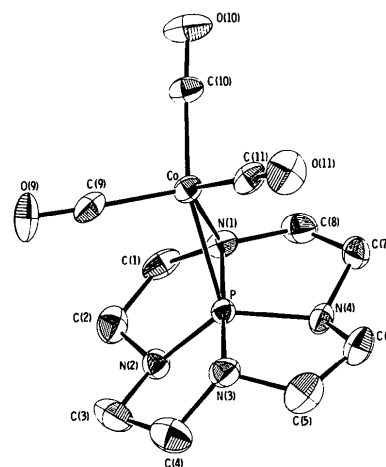


Fig. 1. ORTEP (Johnson, 1976) view of the molecule. Thermal ellipsoids scaled at the 25% probability level. H atoms omitted for clarity.

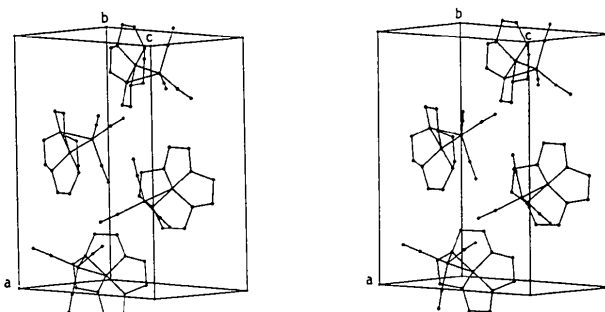


Fig. 2. Stereoscopic view of packing of the molecules in the cell.

**Related literature.** Previous syntheses and structural studies of transition-metal complexes with tetraaza  $\eta^2$ -phosphoranido ligands include ( $\eta^2$ -cyclenphosphoranido)Mo( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)(CO)<sub>2</sub> (Lattman, Chopra, Cowley & Arif, 1986) and ( $\eta^2$ -cyclamphosphoranido)Mo( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)(CO)<sub>2</sub> (Dupart, Grand, Pace & Riess, 1982). Other examples of  $\eta^2$ -coordination of phosphoranido ligands have also been reported (Wachter, Mentzen & Riess, 1981; Jeanneaux, Grand & Riess, 1981).

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## Structure of Triphenyl(4,4,5,5-tetramethyl-2-thioxo-1,3,2-dioxaphospholane-2-thiolato)tin

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**Abstract.** [Sn(C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>PS<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>)]<sub>n</sub>,  $M_r = 561.3$ , monoclinic,  $P2_1/n$ ,  $a = 14.511(6)$ ,  $b = 11.536(7)$ ,  $c = 15.435(14)$  Å,  $\beta = 94.32(6)^\circ$ ,  $U = 2576.5$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.447$  Mg m<sup>-3</sup>,  $\lambda(\text{Ag } K\alpha) = 0.56083$  Å,  $\mu = 0.64$  mm<sup>-1</sup>,  $F(000) = 1136$ ,  $T = 292(1)$  K. Final  $R = 0.041$  for 1796 unique observed X-ray diffractometer data and 273 variables. Besides four atoms directly bound to Sn – three Sn–C(phenyl) bonds [mean bond distance 2.118(9) Å] and one Sn–S bond [2.436(3) Å] – there is a weak chelating interaction between the second S atom and Sn [Sn...S(2) 3.411(3) Å] and the coordination around Sn is intermediate between a tetrahedral and a trigonal-bipyramidal arrangement, with C(21) and S(2) [C(21)–Sn...S(2) 164.9(3)°] in the apical positions. Intermolecular distances exceed van der Waals distances.

**Experimental.** The compound was prepared from (C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>SnOH and HSP(S)(C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>) in ethanol. The precipitate was dried over P<sub>4</sub>O<sub>10</sub>. Colorless crystals from ethanol. M.p. 390–393 K. Crystal size: 0.16 × 0.22 × 0.13 mm.  $\omega/2\theta$  scan. Scan speed: 5° min<sup>-1</sup> in  $\theta$ . Nonius CAD-4 diffractometer, graphite-monochromated Ag  $K\alpha$  radiation. Lattice parameters from least-squares fit with 25 reflexions up to  $2\theta = 28.0^\circ$ ; four standard reflexions recorded every 2.5 h, only random deviations; 4441 reflexions,  $1 \leq \theta \leq 18^\circ$ ,  $-16 \leq h \leq 16$ ,  $-1 \leq k \leq 12$ ,  $0 \leq l \leq 17$ ,  $[(\sin \theta)/\lambda]_{\max}$

### References

- DUPART, J.-M., GRAND, A., PACE, S. & RIESS, J. G. (1982). *J. Am. Chem. Soc.* **104**, 2316–2318.  
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)  
 JEANNEAUX, F., GRAND, A. & RIESS, J. G. (1981). *J. Am. Chem. Soc.* **103**, 4272–4273.  
 JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.  
 LATTMAN, M., CHOPRA, S. K., COWLEY, A. H. & ARIF, A. M. (1986). *Organometallics*, **5**, 677–683.  
 SHELDRIK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.  
 WACHTER, J., MENTZEN, B. F. & RIESS, J. G. (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 284–285.

$= 0.55$  Å<sup>-1</sup>; after averaging 3795 ( $R_{\text{int}} = 0.024$ ) unique reflexions, 1796 with  $I < 3\sigma(I)$ , Lorentz–polarization correction and absorption correction *via*  $\psi$  scans; max./min. transmission 1.00/0.92; systematic absences conform to space group  $P2_1/n$  (No. 14), structure solution *via* direct methods,  $\Delta F$  syntheses and full-matrix least squares on  $F$  with 1796 reflexions; 273 refined parameters, anisotropic temperature factors for all non-H atoms, two common isotropic temperature

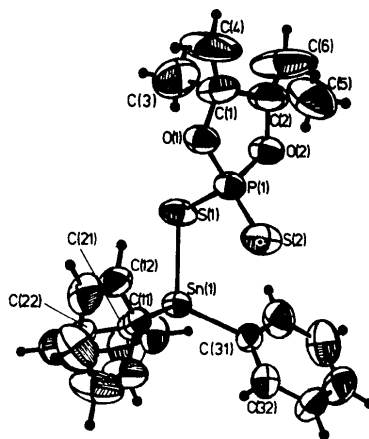


Fig. 1. General view of the molecule, showing atom numbering.