N1—C2	1.469 (2)	C14—C15	1.478 (4)
N1—C18	1.468 (2)	C15—O16	1.427 (2)
C2—C3	1.512 (3)	O16—C17	1.419 (3)
C3—O4	1.400 (3)	C17—C18	1.503 (3)
C18—N1—C2	111.52 (14)	C9—N10—C11	113.64 (14)
N1—C2—C3	113.2 (2)	N10—C11—C12	114.37 (15)
C2—C3—O4	109.8 (2)	C11—C12—O13	108.84 (14)
C3—O4—C5	113.26 (15)	C12—O13—C14	112.35 (15)
O4—C5—C6	108.9 (2)	O13—C14—C15	108.7 (2)
C5—C6—O7	109.0 (2)	C14—C15—O16	110.2 (2)
C6—O7—C8	116.3 (2)	C15—O16—C17	110.6 (2)
O7—C8—C9	113.1 (2)	O16—C17—C18	110.0 (2)
C8—C9—N10	115.4 (2)	C17—C18—N1	113.22 (15)
C18—N1—C2—C3	−72.1 (2)	C9—N10—C11—C12	−68.2 (2)
N1—C2—C3—O4	−55.4 (2)	N10—C11—C12—O13	−54.3 (2)
C2—C3—O4—C5	−179.1 (2)	C11—C12—O13—C14	−178.94 (15)
C3—O4—C5—C6	−170.7 (2)	C12—O13—C14—C15	163.8 (2)
O4—C5—C6—O7	63.8 (2)	O13—C14—C15—O16	63.8 (2)
C5—C6—O7—C8	160.2 (2)	C14—C15—O16—C17	173.9 (2)
C6—O7—C8—C9	70.3 (2)	C15—O16—C17—C18	176.0 (2)
O7—C8—C9—N10	57.1 (2)	C2—N1—C18—C17	165.3 (2)
C8—C9—N10—C11	88.6 (2)	O16—C17—C18—N1	−63.9 (2)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (B. A. Frenz & Associates Inc., 1985). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP*; *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976); *ATOMS* (Dowty, 1994). Software used to prepare material for publication: *SHELXL*93.

The author wish to thank Dr M. Pietraszkiewicz and Mr R. Gąsiorowski for kindly providing the crystals.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KA1105). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.

Alpha, B., Balzani, V., Lehn, J.-M., Perathoner, S. & Sabbatini, N. (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 1266–1267.

Alpha, B., Lehn, J.-M. & Mathis, G. (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 266–267.

B. A. Frenz & Associates Inc. (1985). *SDP. Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

Dowty, E. (1994). *ATOMS. A Computer Program for Displaying Atomic Structures*. IBM-PC version 2.3 32-bit. Shape Software, Kingsport, USA.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Klyne, W. & Prelog, V. (1960). *Experientia*, **16**, 521–568.

Lehn, J.-M. (1987). *Supramolecular Photochemistry*, edited by V. Balzani, pp. 29–42. Dordrecht: Reidel.

Lehn, J.-M., Pietraszkiewicz, M. & Karpiuk, J. (1990). *Helv. Chim. Acta*, **73**, 106–111.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Sabbatini, N., Perathoner, S., Balzani V., Alpha, B. & Lehn, J.-M. (1987). *Supramolecular Photochemistry*, edited by V. Balzani, pp. 29–42. Dordrecht: Reidel.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). C**51**, 2235–2238

trans-Bis(1,10-phenanthroline)-*trans*-bis(2,4,6-trinitrophenolato-*O*,*O*′)calcium(II)

V. Vijayvergiya

Department of Biophysics, University of Delhi, South Campus, New Delhi 110 021, India

B. Padmanabhan and T. P. Singh

Department of Biophysics, All India Institute of Medical Sciences, New Delhi 110 029, India

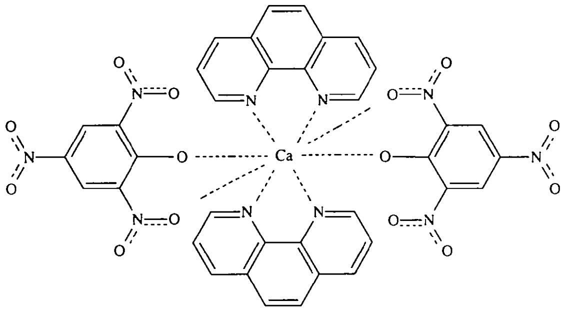
(*Received* 7 *March* 1994; *accepted* 18 *April* 1995)

Abstract

In the title compound, $[Ca(C_6H_2N_3O_7)_2(C_{12}H_8N_2)_2]$, the Ca^{2+} ion exhibits eightfold coordination with four O atoms, two from each 2,4,6-trinitrophenolate (picrate) molecule, and four N atoms from two phenanthroline molecules, forming a twisted square antiprism. The N atoms of the phenanthroline molecule are polarized by the cation so that the Ca—N distances are comparable to the Ca—O(NO) distances.

Comment

The structure determination of the title compound, (I), was undertaken as part of studies of the coordination chemistry of alkali and alkaline earth cations. (I) was synthesized from acetone medium using 2,4,6-trinitrophenolate (picrate) as the anion and 1,10-phenanthroline (phen) as a coordinating ligand (Vijayvergiya, 1984; Kanters, Stouten, Vijayvergiya & Poonia, 1987). This compound is stable in the solid phase (stability constant in acetone 7.143×10^{-3}, m.p. 583 K; Vijayvergiya, 1984), as has been observed for other Ca compounds (Singh, Reinhardt & Poonia, 1980, 1984; Poonia, Chandra, Padmanabhan & Yadav, 1990). The cation is eightfold coordinated by four O atoms of two picrate molecules and four N atoms of two phenanthroline molecules to form a twisted square-antiprismatic environment (Fig. 1).



(I)

The molecular dimensions of the two phenanthroline molecules are very similar and compare well with those observed in the free phenanthroline molecule (Nishigaki, Yoshioka & Nakatsu, 1978), in phananthroline coordinated with various metal atoms (Tiwari, Joshi, Singh & Poonia, 1982; Kanters, Postma, Duisenberg, Venkatsubramanian & Poonia, 1983; Poonia 1975; Frenz & Ibers, 1972; Hughes & Truter, 1972) and in several derivatives of phenanthroline (Battagalia, Comadi, Marcotrigiano & Pellacani, 1977). The phenanthroline ligands are significantly non-planar and appear to be markedly twisted and folded. There is no consistency in the pattern of distortions from planarity, but it appears to be an effect of the packing of the crystals. The ring systems of the two phenanthroline molecules in the present complex are inclined at an angle of 99.6 (1)° with respect to each other.

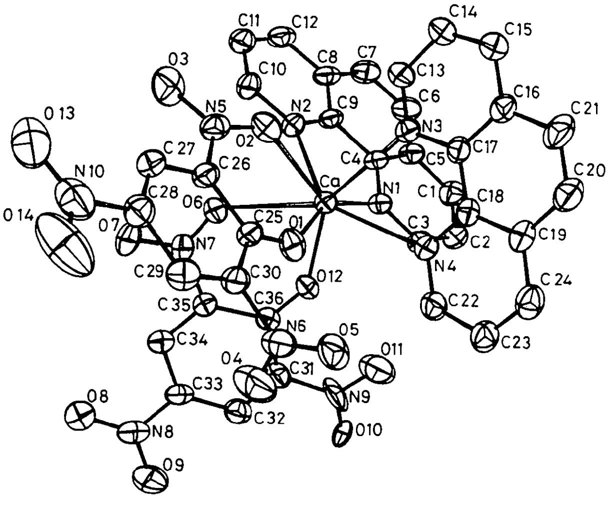


Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of the title compound with displacement ellipsoids at the 40% probability level.

The molecular dimensions of the two picrate molecules are similar. The bond lengths and bond angles in the benzene moieties agree with those reported for a number of nitrophenolate complexes (Bush & Truter, 1971; Kanters, Dekoster, Schouten, Venkatsubramanian & Poonia 1985; Poonia, Chandra, Padmanabhan & Yadav, 1990). Common features are increased C—C bond lengths, decreased C—C—C angles at the phenoxide C atom and enlarged angles (approximately 126°) at *ortho* C atoms. The ring substituents show deviations from the least-squares ring planes. The six-membered rings of the two molecules are inclined at 128.3 (2)°.

Calcium is highly anionphilic towards the picrate anion (Bhagwat, Manohar & Poonia, 1980) and the Ca—O distances are indeed very short [2.296 (3)–2.495 (4) Å], as observed in di(2,2′-bipyridyl)dipicratecalcium [2.292 (2) and 2.357 Å] (Poonia, Chandra, Padmanabhan & Yadav, 1990). However, the cation has successfully polarized the N atoms of the phenanthroline so that the Ca—N distances [2.513 (4)–2.598 (3) Å] are comparable to the Ca—O(NO) distances [2.491 (3) and 2.495 (4) Å]. 'Hard' O-atom donors are usually preferred by the alkaline earth cations.

The coordination polyhedron is a square antiprism formed by two O atoms from each coordinating picrate and two N atoms from each phenanthroline molecule. The molecular packing can be seen in Fig. 2. The picrate NO_2 groups are in close contact with the phenanthroline molecules. The molecules are stacked along the *a* axis, and are held by strong van der Waals forces in the *bc* plane.

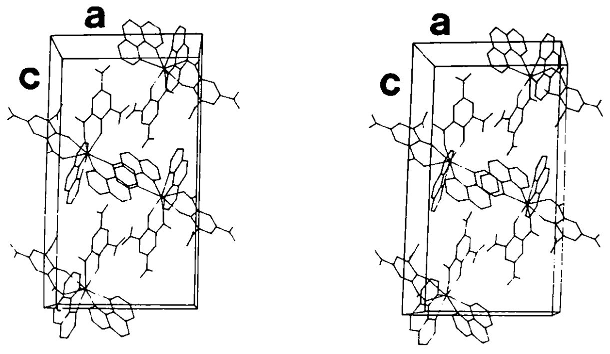


Fig. 2. Molecular packing diagram viewed down the *b* axis.

Experimental

The title compound was prepared by the method of Vijayvergiya (1984). Calcium picrate and 1,10-phenanthroline monohydrate solution in acetone were mixed in a 1:2 ratio. After filtration the clean solution was subjected to slow evaporation in a closed beaker. Yellow rectangular needle-shaped crystals were obtained after 3–4 h. The density D_m was measured by flotation in bromoform.

Crystal data

$[Ca(C_6H_2N_3O_7)_2(C_{12}H_8N_2)_2]$ Cu $K\alpha$ radiation
$M_r = 856.7$ $\lambda = 1.54184$ Å

Monoclinic
$P2_1/c$
$a = 13.538\ (3)$ Å
$b = 10.798\ (3)$ Å
$c = 24.646\ (2)$ Å
$\beta = 91.40\ (3)^\circ$
$V = 3601.7\ (2)$ Å^3
$Z = 4$
$D_x = 1.580$ Mg m^{-3}
$D_m = 1.59$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 20–25^\circ$
$\mu = 2.245$ mm^{-1}
$T = 293$ K
Rectangular
$1.0 \times 0.3 \times 0.2$ mm
Yellow

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.63$, $T_{\max} = 0.92$
7213 measured reflections
6356 independent reflections
5938 observed reflections $[F > 2\sigma(F)]$

$R_{\rm int} = 0.0434$
$\theta_{\max} = 70^\circ$
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 30$
3 standard reflections monitored every 200 reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F
$R = 0.061$
$wR = 0.061$
$S = 1.59$
5921 reflections
586 parameters
H-atom parameters not refined
Unit weights applied

$(\Delta/\sigma)_{\max} = 0.562$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Extinction correction: none
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)

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

	x	y	z	$U_{\rm eq}$
Ca	0.74674 (7)	0.29638 (9)	0.07079 (4)	0.0209 (4)
N(1)	0.5781 (2)	0.2590 (3)	0.0292 (1)	0.022 (2)
N(2)	0.6868 (2)	0.4613 (3)	0.0026 (1)	0.026 (2)
N(3)	0.7997 (2)	0.1909 (3)	−0.0187 (1)	0.022 (2)
N(4)	0.7717 (2)	0.0654 (3)	0.0760 (1)	0.024 (2)
N(5)	0.9659 (3)	0.4600 (3)	0.0741 (1)	0.023 (2)
N(6)	0.9681 (3)	0.1631 (4)	0.2271 (1)	0.033 (2)
N(7)	0.7672 (3)	0.5140 (3)	0.1731 (1)	0.029 (2)
N(8)	0.6526 (3)	0.4840 (5)	0.3566 (1)	0.035 (3)
N(9)	0.5380 (3)	0.1728 (5)	0.2280 (1)	0.028 (3)
N(10)	1.2380 (3)	0.4593 (5)	0.2052 (2)	0.030 (3)
C(1)	0.4147 (3)	0.1824 (5)	−0.0343 (2)	0.028 (3)
C(2)	0.4390 (3)	0.1236 (4)	0.0129 (2)	0.026 (2)
C(3)	0.5213 (3)	0.1630 (4)	0.0436 (1)	0.025 (2)
C(4)	0.5514 (3)	0.3206 (4)	−0.0168 (1)	0.020 (2)
C(5)	0.4716 (3)	0.2864 (4)	−0.0506 (1)	0.025 (2)
C(6)	0.4491 (4)	0.3528 (5)	−0.0990 (2)	0.036 (3)
C(7)	0.5018 (4)	0.4559 (5)	−0.1119 (2)	0.036 (3)
C(8)	0.5812 (3)	0.4963 (4)	−0.0775 (1)	0.024 (2)
C(9)	0.6089 (3)	0.4298 (4)	−0.0310 (1)	0.020 (2)
C(10)	0.7360 (3)	0.5643 (4)	−0.0099 (2)	0.032 (3)
C(11)	0.7115 (4)	0.6378 (4)	−0.0546 (2)	0.030 (3)
C(12)	0.6360 (4)	0.6050 (4)	−0.0887 (2)	0.028 (3)
C(13)	0.8094 (4)	0.2476 (4)	−0.0662 (2)	0.024 (3)
C(14)	0.8545 (4)	0.1947 (5)	−0.1111 (2)	0.023 (3)
C(15)	0.8929 (4)	0.0785 (5)	−0.1061 (2)	0.021 (3)
C(16)	0.8844 (3)	0.0148 (4)	−0.0572 (2)	0.021 (2)
C(17)	0.8346 (3)	0.0733 (4)	−0.0145 (1)	0.021 (2)
C(18)	0.8224 (3)	0.0081 (4)	0.0357 (1)	0.023 (2)
C(19)	0.8624 (3)	−0.1120 (4)	0.0425 (2)	0.031 (2)
C(20)	0.9148 (4)	−0.1665 (4)	−0.0011 (2)	0.035 (3)
C(21)	0.9241 (4)	−0.1072 (5)	−0.0483 (2)	0.029 (3)
C(22)	0.7618 (3)	0.0059 (4)	0.1225 (1)	0.030 (2)
C(23)	0.7985 (4)	−0.1135 (4)	0.1323 (2)	0.039 (3)
C(24)	0.8496 (4)	−0.1716 (4)	0.0925 (2)	0.042 (3)
C(25)	0.9596 (3)	0.3064 (4)	0.1494 (1)	0.023 (2)
C(26)	1.0093 (3)	0.4068 (4)	0.1224 (1)	0.021 (2)
C(27)	1.0982 (3)	0.4564 (5)	0.1415 (1)	0.020 (2)
C(28)	1.1439 (3)	0.4086 (4)	0.1861 (2)	0.023 (2)
C(29)	1.1028 (3)	0.3102 (5)	0.2149 (1)	0.028 (3)
C(30)	1.0143 (3)	0.2647 (4)	0.1962 (1)	0.025 (2)
C(31)	0.6006 (3)	0.2819 (4)	0.2389 (1)	0.023 (2)
C(32)	0.5998 (3)	0.3273 (5)	0.2900 (1)	0.024 (3)
C(33)	0.6556 (3)	0.4320 (4)	0.3025 (1)	0.026 (2)
C(34)	0.7105 (3)	0.4905 (4)	0.2638 (1)	0.029 (2)
C(35)	0.7106 (3)	0.4431 (4)	0.2120 (1)	0.022 (2)
C(36)	0.6574 (3)	0.3333 (4)	0.1947 (1)	0.022 (2)
O(1)	0.8776 (2)	0.2615 (3)	0.1351 (1)	0.033 (2)
O(2)	0.9037 (2)	0.4019 (3)	0.0466 (1)	0.028 (2)
O(3)	0.9927 (3)	0.5635 (3)	0.0601 (1)	0.048 (2)
O(4)	0.9259 (5)	0.1867 (4)	0.2652 (2)	0.034 (5)
O(5)†	0.9553 (8)	0.0668 (1)	0.2059 (4)	0.058 (6)
O(5′)†	1.0080 (12)	0.0588 (14)	0.2228 (7)	0.054 (10)
O(6)	0.7612 (2)	0.4911 (3)	0.1246 (1)	0.027 (2)
O(7)‡	0.8104 (9)	0.6079 (11)	0.1894 (6)	0.043 (7)
O(7′)‡	0.838 (2)	0.572 (3)	0.1925 (17)	0.036 (13)
O(8)	0.6955 (3)	0.5825 (4)	0.3656 (1)	0.043 (3)
O(9)	0.6086 (4)	0.4263 (4)	0.3913 (1)	0.036 (3)
O(10)§	0.4962 (11)	0.1205 (13)	0.2632 (7)	0.031 (8)
O(10′)§	0.4651 (11)	0.1693 (15)	0.2577 (8)	0.018 (9)
O(11)¶	0.5744 (10)	0.0968 (12)	0.1990 (6)	0.019 (7)
O(11′)¶	0.5173 (6)	0.1390 (10)	0.1819 (3)	0.055 (7)
O(12)	0.6563 (2)	0.2880 (3)	0.1483 (1)	0.022 (2)
O(13)	1.2682 (3)	0.5539 (4)	0.1823 (1)	0.037 (3)
O(14)	1.2813 (3)	0.4095 (6)	0.2429 (2)	0.064 (4)

† Occupancies: 0.6 O(5) + 0.4 O(5′).
‡ Occupancies: 0.7 O(7) + 0.3 O(7′).
§ Occupancies: 0.51 O(10) + 0.49 O(10′).
¶ Occupancies: 0.5 O(11) + 0.5 O(11′).

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

Ca—N(1)	2.513 (4)	Ca—O(1)	2.377 (3)
Ca—N(2)	2.567 (3)	Ca—O(12)	2.296 (3)
Ca—N(3)	2.598 (3)	Ca—O(6)	2.491 (3)
Ca—N(4)	2.520 (3)	Ca—O(2)	2.495 (4)
O(6)—Ca—O(2)	71.7 (1)	N(2)—Ca—O(2)	77.5 (1)
O(12)—Ca—O(2)	133.7 (1)	N(2)—Ca—O(6)	77.4 (1)
O(12)—Ca—O(6)	68.1 (1)	N(2)—Ca—O(12)	113.9 (1)
O(1)—Ca—O(2)	66.9 (1)	N(2)—Ca—O(1)	139.9 (1)
O(1)—Ca—O(6)	74.4 (1)	N(2)—Ca—N(4)	139.4 (1)
O(1)—Ca—O(12)	80.9 (1)	N(2)—Ca—N(3)	80.6 (1)
N(4)—Ca—O(2)	110.5 (1)	N(1)—Ca—O(2)	138.2 (1)
N(4)—Ca—O(6)	143.1 (1)	N(1)—Ca—O(6)	114.3 (1)
N(4)—Ca—O(12)	89.6 (1)	N(1)—Ca—O(12)	80.5 (1)
N(4)—Ca—O(1)	73.3 (1)	N(1)—Ca—O(1)	154.3 (1)
N(3)—Ca—O(2)	75.2 (1)	N(1)—Ca—N(4)	88.9 (1)
N(3)—Ca—O(6)	143.4 (1)	N(1)—Ca—N(3)	81.3 (1)
N(3)—Ca—O(12)	148.5 (1)	N(1)—Ca—N(2)	64.8 (1)
N(3)—Ca—O(1)	106.3 (1)	Ca—N(1)—C(4)	118.9 (3)
N(3)—Ca—N(4)	64.5 (1)	Ca—N(1)—C(3)	122.4 (3)

O(8)—N(8)—C(33)—C(32)	−173.7 (5)
O(9)—N(8)—C(33)—C(34)	−175.4 (5)
O(11)—N(9)—C(31)—C(36)	39.8 (1)
O(11)—N(9)—C(31)—C(32)	−140.7 (1)
N(4)—C(22)—C(23)—C(24)	1.6 (8)
N(4)—C(18)—C(19)—C(20)	−179.7 (4)

All non H-atoms were refined anisotropically. Atoms O(5), O(7), O(10) and O(11) were disordered and subjected to occupancy refinement. The H-atom positions were obtained by geometrical calculations and included in the final cycles of refinement as fixed parameters. The refinement was carried using a MicroVAX II computer.

Data collection: *CAD*-4 *Software* (Enraf–Nonus, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (B. A. Frenz & Associates Inc., 1984). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976); *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Financial support from CSIR (India) is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1176). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

B. A. Frenz & Associates Inc. (1984). *SDP-Plus Structure Determination Package*. College Station, Texas, USA.

Battagalia, L. P., Comadi, A. B., Marcotrigiano, G. & Pellacani, G. C. (1977). *Acta Cryst.* B**33**, 3886–3888.

Bhagwat, V. W., Manohar, H. & Poonia, N. S. (1980). *Inorg. Nucl. Chem. Lett.* **16**, 289–292.

Bush, M. A. & Truter, M. R. (1971). *J. Chem. Soc. A*, pp. 795–797.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Frenz, B. A. & Ibers, J. A. (1972). *Inorg. Chem.* **11**, 1109–1116.

Hughes, D. L. & Truter, M. R. (1972). *J. Chem. Soc. Dalton Trans.* pp. 2214–2219.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kanters, J. A., Dekoster, A., Schouten, K., Venkatsubramanian, K. & Poonia, N. S. (1985). *Acta Cryst.* C**41**, 1585–1588.

Kanters, J. A., Postma, R., Duisenberg, A. J. M., Venkatsubramanian, K. & Poonia, N. S. (1983). *Acta Cryst.* C**39**, 1519–1522.

Kanters, J. A., Stouten, P. F., Vijayvergiya, V. & Poonia, N. S. (1987). *Polyhedron*, **6**, 1833–1837.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *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.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Nishigaki, S., Yoshioka, H. & Nakatsu, K. (1978). *Acta Cryst.* B**34**, 875–878.

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

Poonia, N. S. (1975). *J. Inorg. Nucl. Chem.* **37**, 1855–1858.

Poonia, N. S., Chandra, R., Padmanabhan, V. M. & Yadav, V. S. (1990). *J. Coord. Chem.* **21**, 167–174.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Singh, T. P., Reinhardt, R. & Poonia, N. S. (1980). *Inorg. Nucl. Chem. Lett.* **16**, 293–296.

Singh, T. P., Reinhardt, R. & Poonia, N. S. (1984). *Ind. J. Chem.* **23A**, 976–982.

Tiwari, R. K., Joshi, V. V., Singh, T. P. & Poonia, N. S. (1982). *Ind. J. Phys.* **56A**, 381–395.

Vijayvergiya, V. (1984). PhD thesis, Devi Ahilya Vishwavidyalaya, Indore, India.

Acta Cryst. (1995). C**51**, 2238–2242

fac-Tricarbonyltris(triphenyl phosphite)-molybdenum(0)

Elmer C. Alyea, George Ferguson and
Shu-Quan Song

Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

(*Received* 13 *January* 1995; *accepted* 17 *May* 1995)

Abstract

There are two independent molecules of the title compound, $[Mo(CO)_3(C_{18}H_{15}O_3P)_3]$, in the asymmetric unit; the geometry about the Mo atom is distorted octahedral, with principal mean dimensions Mo—P 2.435 (8), Mo—C 1.99 (3), P—O 1.604 (18) Å, P—Mo—P 90 (3), C—Mo—C 86 (3), *cis*-C—Mo—P 92 (5), *trans*-C—Mo—P 174 (3), C—O—P 128 (4) and Mo—C—O 174 (2)°. The short mean Mo—P bond distance reflects the strong π-acceptor ability of $P(OPh)_3$, as also demonstrated by the highly shielded $\delta(^{95}Mo)$ value (−1673 p.p.m.), whereas the various angle distortions attest to the significant but not large steric effect of the $P(OPh_3)_3$ ligand in the *fac* environment. The P-atom geometry is distorted tetrahedral, with larger mean Mo—P—O angles [118 (4)°] and smaller mean O—P—O angles [100 (3)°].

Comment

The compound *fac*-$Mo(CO)_3\{P(OC_6H_5)_3\}_3$, (1), was synthesized during our extensive investigation of the bonding nature of phosphorus(III) ligands using ^{31}P and ^{95}Mo NMR spectroscopy (Alyea & Song, 1992; Song, 1994). The only X-ray structure determination of a transition metal species containing the *fac*-$M(CO)_3\{P(OR)_3\}_3$ moiety (M = a transition metal, R = any substituent) found in a search of the October 1994 release of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) was that for *fac*-$Mo(CO)_3\{P(OMe)_2O\}_3SiMe$ (Greene, Taylor, Kee & Thornton-Pett, 1993); relevant structural determinations for Group 6 metal *fac*-$M(CO)_3P_3$ fragments with monodentate phosphorus ligands are also rare: $Cr(CO)_3(PH_3)_3$ (Huttner & Schelle, 1969), $Cr(CO)_3(PEt_3)_3$ (Holladay, Churchill, Wong & Atwood, 1980), $Mo(CO)_3(PPh_2H)_3$ (Willey, Butcher, Lakin & Downs, 1993) and $Mo(CO)_3\{P(NH^iPr)_3\}_3$ (Tarassoli, Chen, Allured, Hill, Haltiwanger, Thompson & Norman, 1986). Following our structural analysis of *cis*-$Mo(CO)_4\{P(OC_6H_5)_3\}_2$, (2) (Alyea, Ferguson & Zwikker, 1994), which revealed only small geomet-