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).

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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.

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

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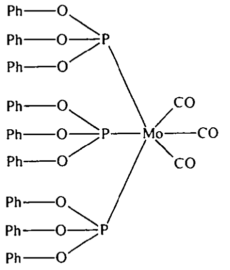
Abstract

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 geometric distortions due to steric effects, we decided to examine the impact on the Mo geometry of replacing a CO group by a third $P(OC_6H_5)_3$ ligand.



(1)

There are two independent molecules, both with *fac* stereochemistry, in the asymmetric unit of (1). One of the molecules is shown in Fig. 1; the other molecule has very similar stereochemistry at the Mo atom. Both have some distortion (Table 2) from idealized octahedral geometry at the Mo atoms and quite different conformations of the $P(OPh)_3$ moieties.

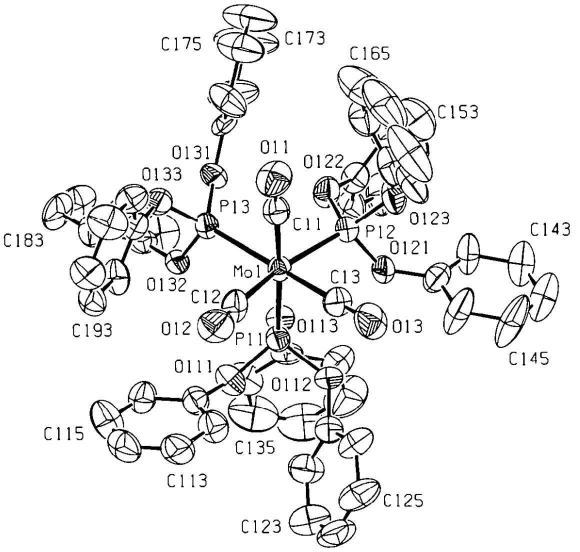


Fig. 1. A view of one of the two independent molecules of (1) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

The mean* Mo—P bond distance in (1) [2.435 (8) Å] is similar to those found in (2) [2.442 (1) Å] and in *fac*-$Mo(CO)_3\{P(OMe)_2O\}_3SiMe$ [2.441 (6) Å]. For comparison, longer Mo—P bond lengths in $Mo(CO)_3(PPh_2H)_3$ [2.500 (7) Å] and in $Mo(CO)_3\{P(NH^iPr)_3\}_3$ [2.545 (7) Å] suggest the presence of less π-bonding ability in these compounds. The relatively strong π-acceptor nature of $P(OPh_3)_3$ in (1) is substantiated by the high field $\delta(^{95}Mo)$ value (−1673 p.p.m.), as compared with Mo chemical shifts for other *fac* species [*e.g.* $Mo(CO)_3(PPh_3)_3$ has $\delta(^{95}Mo)$ = −1265 *versus* −1857 p.p.m. for $Mo(CO)_6$ (Song, 1994)].

The mean Mo—C bond length in (1) [1.99 (3) Å] is similar to the mean Mo—C distances in *fac*-$Mo(CO)_3\{P(OMe)_2O\}_3SiMe$ [2.007 (3) Å] and in (2) [*trans* to P 2.010 (7) Å, *trans* to C 2.037 (5) Å]. That the Mo—C bond distance reflects the amount of π bonding is supported by the even shorter Mo—C distances in $Mo(CO)_3(PPh_2H)_3$ [1.935 (4) Å] and in $Mo(CO)_3\{P(NH^iPr)_3\}_3$ [1.90 (2) Å], in which the phosphorus ligands are considered to be weaker π acceptors compared with those in (1) and (2), as substantiated by the longer Mo—P distances noted above.

The P—Mo—P angles in (1) are in the range 85.58 (11)–95.05 (11)°, reflecting the distortions due to the more congested steric environment at Mo compared with that in (2), where the P—Mo—P angle is 89.55 (5)°. In contrast to the near ideal P—*M*—P angle observed for $Cr(CO)_3(PH_3)_3$ [89.9 (9)°], other Group 6 *fac*-$M(CO)_3(PR_3)_3$ complexes also show significant deviations: 86.7 (11)° in $Mo(CO)_3(PPh_2H)_3$ and 93.9 (2)° in $Mo(CO)_3\{P(NH^iPr)_3\}_3$.

The C—Mo—C angles in (1) range from 82.4 (5) to 91.2 (4)° [mean 86 (3)°]; the corresponding values in (2) are from 87.5 (2) to 92.0 (2)° [mean 89 (2)°]. Mean C—Mo—C angles calculated for other Group 6 *fac*-$M(CO)_3(PR_3)_3$ species are also distorted [87 (3)° in $Cr(CO)_3(PEt_3)_3$ (Holladay, Churchill, Wong & Atwood, 1980); 86.9 (6)° in $Mo(CO)_3(PPh_2H)_3$ (Willey, Butcher, Lakin & Downs, 1993); 86.6 (7)° in $Mo(CO)_3\{P(NH^iPr)_3\}_3$ (Tarassoli, Chen, Allured, Hill, Haltiwanger, Thompson & Norman, 1986)]. As expected, nearly idealized C—*M*—C angles are observed when the phosphorus ligand is less bulky: 89.6 (2)° in $Cr(CO)_3(PH_3)_3$ (Huttner & Schelle, 1969) and 90.7 (11)° in *fac*-$Mo(CO)_3\{P(OMe)_2O\}_3SiMe$ (Greene, Taylor, Kee & Thornton-Pett, 1993).

The remaining angles around the Mo atoms in (1) also show greater departures from the idealized pseudo-octahedral geometry than for (2). The *cis*-P—Mo—C angles [84.72 (11)–98.36 (11)°; *cf.* 88.2 (2)–95.7 (2)° for (2)] and the *trans*-P—Mo—C angles [range 169.2 (3)–179.3 (1)°, mean 174 (3)°; *cf.* 173.9 (2) and 178.0 (2)° for (2)] are also more distorted than the analogous angles in (2). Intramolecular repulsions in (1) also lead to Mo—C—O angles [range 169.7 (11)–177.7 (12)°, mean 174 (2)°] that are more distorted from linearity than in (2) [175.1 (5)–178.2 (5)°, mean 176.7 (15)°].

The geometry about the P atom is distorted tetrahedral, with larger Mo—P—O angles [range 110.2 (3)–124.9 (4)°, mean 118 (4)°] and smaller O—P—O angles [range 96.1 (4)–104.9 (5)°, mean 100 (3)°]. Other bond distances and angles in (1) are normal and similar to those in (2) [*e.g.* P—O in the range 1.568 (8)–1.639 (8) Å,

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* Throughout this paper the e.s.d.'s of the mean of the *n* values were calculated from the expression: e.s.d. = $\{\Sigma[d-d(\text{mean})]^2/n\}^{0.5}$.

mean 1.60 (2) Å; P—O—C 121.1 (5)–132.3 (5)°, mean 128 (4)°]; a full list is available as supplementary material. Intermolecular contacts are of the van der Waals type.

Experimental

Compound (1) was synthesized by adding a twofold excess of $P(OPh)_3$ to a dichloromethane solution of $Mo(CO)_3$(cycloheptatriene) with stirring at room temperature for 2 h under a dry nitrogen atmosphere. Removal of some of the solvent under a nitrogen stream and cooling at *ca* 273 K for 1 h gave a colourless precipitate, which was collected, washed with *n*-hexane and dried *in vacuo*. The infrared spectrum in dichloromethane showed ν(CO) bands at 1989 and 1911 cm^{-1}, in good agreement with literature values and with C_{3v} symmetry (Bond, Carr & Colton, 1984). A single crystal for X-ray analysis was obtained by slowly evaporating solvent from a dichloromethane solution of the complex.

Crystal data

$[Mo(CO)_3(C_{18}H_{15}O_3P)_3]$
$M_r = 1110.78$
Triclinic
$P\overline{1}$
$a = 11.991 (5)$ Å
$b = 20.666 (2)$ Å
$c = 24.082 (4)$ Å
$\alpha = 67.616 (10)°$
$\beta = 77.54 (2)°$
$\gamma = 87.58 (2)°$
$V = 5383 (3)$ Å^3
$Z = 4$
$D_x = 1.371$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 6.00–12.00°$
$\mu = 0.394$ mm^{-1}
$T = 294 (1)$ K
Plate
0.25 × 0.20 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ-scan data (North, Phillips & Mathews, 1968)
$T_{min} = 0.882$, $T_{max} = 0.918$
16 846 measured reflections
16 846 independent reflections

5447 observed reflections $[I > 2\sigma(I)]$
$\theta_{max} = 23.92°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 23$
$l = -24 \rightarrow 27$
3 standard reflections
frequency: 60 min
intensity decay: 5.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0908$
$wR(F^2) = 0.2200$
$S = 0.808$
16 846 reflections
991 parameters
H atoms refined using a riding model [C—H 0.93 Å (*SHELXL*93; Sheldrick, 1993)]

$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.222$
$\Delta\rho_{max} = 0.443$ e Å^{-3}
$\Delta\rho_{min} = -0.900$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
Mo1	0.39327 (9)	0.49151 (5)	0.26578 (5)	0.0400 (3)
P11	0.4471 (3)	0.6163 (2)	0.21837 (14)	0.0452 (8)
P12	0.2524 (3)	0.5216 (2)	0.34028 (14)	0.0445 (9)
P13	0.2683 (3)	0.5092 (2)	0.19382 (14)	0.0415 (8)
O11	0.3264 (8)	0.3301 (5)	0.3189 (4)	0.080 (3)
O12	0.6060 (8)	0.4374 (5)	0.1967 (4)	0.080 (3)
O13	0.5535 (8)	0.4619 (5)	0.3581 (4)	0.085 (3)
O111	0.5190 (6)	0.6549 (4)	0.1490 (3)	0.052 (2)
O112	0.5149 (7)	0.6455 (4)	0.2570 (3)	0.057 (2)
O113	0.3408 (6)	0.6666 (4)	0.2143 (3)	0.048 (2)
O121	0.2877 (7)	0.5858 (4)	0.3547 (3)	0.056 (2)
O122	0.1315 (7)	0.5452 (4)	0.3216 (3)	0.059 (2)
O123	0.2161 (8)	0.4677 (4)	0.4112 (3)	0.067 (3)
O131	0.1326 (6)	0.5130 (4)	0.2123 (3)	0.049 (2)
O132	0.2888 (6)	0.5837 (4)	0.1364 (3)	0.044 (2)
O133	0.2671 (6)	0.4534 (4)	0.1614 (3)	0.051 (2)
C11	0.3483 (11)	0.3887 (7)	0.3003 (5)	0.054 (3)
C12	0.5277 (11)	0.4610 (7)	0.2162 (6)	0.058 (4)
C13	0.4915 (2)	0.47634 (11)	0.32373 (9)	0.055 (4)
C111	0.6005 (2)	0.63118 (11)	0.11275 (9)	0.054 (3)
C112	0.6982 (2)	0.60147 (11)	0.13247 (9)	0.065 (4)
C113	0.7827 (2)	0.58104 (11)	0.09373 (9)	0.079 (4)
C114	0.7695 (2)	0.59032 (11)	0.03526 (9)	0.079 (5)
C115	0.6718 (2)	0.62002 (11)	0.01553 (9)	0.099 (5)
C116	0.5873 (2)	0.64045 (11)	0.05428 (9)	0.066 (4)
C121	0.6249 (2)	0.67026 (11)	0.23954 (9)	0.059 (4)
C122	0.6639 (2)	0.73311 (11)	0.19098 (9)	0.079 (4)
C123	0.7777 (2)	0.75572 (11)	0.17732 (9)	0.112 (6)
C124	0.8525 (2)	0.71548 (11)	0.21223 (9)	0.094 (5)
C125	0.8135 (2)	0.65263 (11)	0.26080 (9)	0.088 (5)
C126	0.6997 (2)	0.63002 (11)	0.27445 (9)	0.071 (4)
C131	0.3570 (2)	0.73807 (11)	0.19661 (9)	0.051 (3)
C132	0.3479 (2)	0.76497 (11)	0.24217 (9)	0.082 (5)
C133	0.3623 (2)	0.83668 (11)	0.22613 (9)	0.120 (6)
C134	0.3859 (2)	0.88148 (11)	0.16455 (9)	0.121 (7)
C135	0.3949 (2)	0.85458 (11)	0.11900 (9)	0.112 (6)
C136	0.3805 (2)	0.78288 (11)	0.13503 (9)	0.067 (4)
C141	0.2996 (2)	0.59025 (11)	0.40914 (9)	0.062 (4)
C142	0.2100 (2)	0.61403 (11)	0.44270 (9)	0.080 (5)
C143	0.2228 (2)	0.62211 (11)	0.49573 (9)	0.093 (5)
C144	0.3252 (2)	0.60642 (11)	0.51521 (9)	0.108 (6)
C145	0.4148 (2)	0.58264 (11)	0.48165 (9)	0.128 (7)
C146	0.4020 (2)	0.57456 (11)	0.42862 (9)	0.105 (5)
C151	0.0543 (2)	0.58072 (11)	0.34741 (9)	0.056 (4)
C152	−0.0293 (2)	0.54663 (11)	0.39985 (9)	0.086 (5)
C153	−0.1083 (2)	0.58519 (11)	0.42426 (9)	0.112 (6)
C154	−0.1038 (2)	0.65784 (11)	0.39623 (9)	0.103 (6)
C155	−0.0202 (2)	0.69193 (11)	0.34380 (9)	0.085 (5)
C156	0.0589 (2)	0.65337 (11)	0.31939 (9)	0.070 (4)
C161	0.2136 (2)	0.39692 (11)	0.42999 (9)	0.070 (4)
C162	0.2981 (2)	0.35956 (11)	0.45873 (9)	0.091 (5)
C163	0.2942 (2)	0.28684 (11)	0.48024 (9)	0.142 (9)
C164	0.2058 (2)	0.25146 (11)	0.47301 (9)	0.143 (9)
C165	0.1212 (2)	0.28881 (11)	0.44426 (9)	0.138 (8)
C166	0.1251 (2)	0.36154 (11)	0.42275 (9)	0.083 (5)
C171	0.0479 (2)	0.46297 (11)	0.24738 (9)	0.050 (3)
C172	0.0611 (2)	0.39123 (11)	0.27354 (9)	0.082 (5)
C173	−0.0328 (2)	0.34663 (11)	0.30997 (9)	0.108 (6)
C174	−0.1398 (2)	0.37377 (11)	0.32023 (9)	0.132 (7)
C175	−0.1531 (2)	0.44551 (11)	0.29407 (9)	0.161 (8)
C176	−0.0592 (2)	0.49012 (11)	0.25764 (9)	0.103 (6)
C181	0.2116 (2)	0.60920 (11)	0.09824 (9)	0.052 (3)
C182	0.2106 (2)	0.58758 (11)	0.05030 (9)	0.073 (4)
C183	0.1338 (2)	0.61523 (11)	0.01231 (9)	0.088 (5)
C184	0.0581 (2)	0.66449 (11)	0.02224 (9)	0.094 (5)
C185	0.0591 (2)	0.68612 (11)	0.07018 (9)	0.084 (5)
C186	0.1359 (2)	0.65847 (11)	0.10818 (9)	0.074 (4)
C191	0.3631 (2)	0.42556 (11)	0.13794 (9)	0.046 (3)

C192	0.4534 (2)	0.46846 (11)	0.09485 (9)	0.058 (4)
C193	0.5480 (2)	0.43903 (11)	0.07026 (9)	0.065 (4)
C194	0.5522 (2)	0.36669 (11)	0.08877 (9)	0.074 (4)
C195	0.4618 (2)	0.32378 (11)	0.13187 (9)	0.087 (5)
C196	0.3673 (2)	0.35322 (11)	0.15645 (9)	0.058 (4)
Mo2	0.73624 (10)	0.06117 (6)	0.25169 (5)	0.0508 (4)
P21	0.8263 (3)	−0.0254 (2)	0.3272 (2)	0.0584 (10)
P22	0.8737 (3)	0.1490 (2)	0.2435 (2)	0.0591 (10)
P23	0.8640 (3)	0.0405 (2)	0.16685 (15)	0.0487 (9)
O21	0.6079 (10)	0.1866 (6)	0.1745 (5)	0.115 (4)
O22	0.5511 (9)	−0.0384 (6)	0.2455 (5)	0.106 (4)
O23	0.5455 (9)	0.0666 (6)	0.3607 (5)	0.121 (4)
O211	0.9449 (7)	−0.0524 (4)	0.3016 (3)	0.059 (2)
O212	0.7596 (9)	−0.0928 (5)	0.3792 (4)	0.091 (3)
O213	0.8642 (8)	0.0037 (5)	0.3747 (4)	0.088 (3)
O221	0.8553 (8)	0.1957 (5)	0.2828 (4)	0.078 (3)
O222	0.9945 (8)	0.1171 (5)	0.2564 (5)	0.097 (3)
O223	0.9065 (9)	0.2073 (5)	0.1768 (4)	0.090 (3)
O231	0.8506 (7)	0.0806 (4)	0.0967 (3)	0.055 (2)
O232	0.9998 (6)	0.0542 (4)	0.1547 (3)	0.050 (2)
O233	0.8539 (7)	−0.0398 (4)	0.1761 (3)	0.058 (2)
C21	0.6564 (14)	0.1401 (8)	0.1987 (7)	0.084 (5)
C22	0.6231 (12)	−0.0047 (7)	0.2488 (6)	0.067 (4)
C23	0.6216 (2)	0.06700 (11)	0.32042 (9)	0.068 (4)
C211	1.0249 (2)	−0.08739 (11)	0.33484 (9)	0.074 (4)
C212	0.9970 (2)	−0.15504 (11)	0.37810 (9)	0.098 (5)
C213	1.0767 (2)	−0.19228 (11)	0.41098 (9)	0.137 (7)
C214	1.1843 (2)	−0.16186 (11)	0.40058 (9)	0.128 (7)
C215	1.2122 (2)	−0.09421 (11)	0.35732 (9)	0.170 (9)
C216	1.1325 (2)	−0.05697 (11)	0.32445 (9)	0.146 (8)
C221	0.6806 (2)	−0.13339 (11)	0.36944 (9)	0.073 (4)
C222	0.5713 (2)	−0.14719 (11)	0.40664 (9)	0.102 (6)
C223	0.4972 (2)	−0.19508 (11)	0.40359 (9)	0.139 (8)
C224	0.5324 (2)	−0.22917 (11)	0.36335 (9)	0.144 (9)
C225	0.6417 (2)	−0.21537 (11)	0.32615 (9)	0.116 (6)
C226	0.7158 (2)	−0.16748 (11)	0.32920 (9)	0.096 (5)
C231	0.8300 (2)	−0.01912 (11)	0.43598 (9)	0.088 (5)
C232	0.7185 (2)	−0.01081 (11)	0.46311 (9)	0.156 (8)
C233	0.6859 (2)	−0.03165 (11)	0.52662 (9)	0.221 (11)
C234	0.7648 (2)	−0.06080 (11)	0.56300 (9)	0.224 (12)
C235	0.8763 (2)	−0.06911 (11)	0.53586 (9)	0.225 (11)
C236	0.9089 (2)	−0.04827 (11)	0.47235 (9)	0.165 (8)
C241	0.7632 (2)	0.19939 (11)	0.32511 (9)	0.086 (5)
C242	0.6687 (2)	0.23547 (11)	0.30671 (9)	0.129 (7)
C243	0.5766 (2)	0.24173 (11)	0.35039 (9)	0.174 (10)
C244	0.5792 (2)	0.21192 (11)	0.41247 (9)	0.188 (11)
C245	0.6738 (2)	0.17585 (11)	0.43087 (9)	0.191 (11)
C246	0.7658 (2)	0.16958 (11)	0.38719 (9)	0.125 (7)
C251	1.0945 (2)	0.14209 (11)	0.25598 (9)	0.076 (4)
C252	1.1915 (2)	0.12876 (11)	0.21911 (9)	0.104 (6)
C253	1.2993 (2)	0.14761 (11)	0.22220 (9)	0.119 (7)
C254	1.3099 (2)	0.17979 (11)	0.26214 (9)	0.121 (7)
C255	1.2129 (2)	0.19313 (11)	0.29901 (9)	0.108 (6)
C256	1.1051 (2)	0.17428 (11)	0.29593 (9)	0.093 (5)
C261	0.9318 (2)	0.27705 (11)	0.15967 (9)	0.067 (4)
C262	1.0453 (2)	0.30085 (11)	0.14409 (9)	0.066 (4)
C263	1.0721 (2)	0.37189 (11)	0.12498 (9)	0.069 (4)
C264	0.9853 (2)	0.41914 (11)	0.12144 (9)	0.066 (4)
C265	0.8718 (2)	0.39534 (11)	0.13702 (9)	0.081 (5)
C266	0.8450 (2)	0.32429 (11)	0.15613 (9)	0.077 (4)
C271	0.7496 (2)	0.10463 (11)	0.07868 (9)	0.059 (4)
C272	0.6578 (2)	0.05850 (11)	0.09159 (9)	0.089 (5)
C273	0.5570 (2)	0.08378 (11)	0.07323 (9)	0.119 (6)
C274	0.5479 (2)	0.15519 (11)	0.04196 (9)	0.116 (7)
C275	0.6397 (2)	0.20132 (11)	0.02905 (9)	0.104 (6)
C276	0.7406 (2)	0.17604 (11)	0.04741 (9)	0.077 (4)
C281	1.0801 (2)	0.08511 (11)	0.10035 (9)	0.054 (3)
C282	1.1626 (2)	0.04511 (11)	0.08002 (9)	0.070 (4)
C283	1.2488 (2)	0.07731 (11)	0.02834 (9)	0.083 (5)
C284	1.2525 (2)	0.14953 (11)	−0.00300 (9)	0.098 (5)
C285	1.1700 (2)	0.18953 (11)	0.01734 (9)	0.084 (5)
C286	1.0838 (2)	0.15733 (11)	0.06902 (9)	0.062 (4)
C291	0.9318 (2)	−0.07405 (11)	0.14770 (9)	0.048 (3)
C292	1.0086 (2)	−0.11567 (11)	0.18025 (9)	0.065 (4)
C293	1.0885 (2)	−0.15142 (11)	0.15302 (9)	0.078 (4)
C294	1.0917 (2)	−0.14553 (11)	0.09324 (9)	0.088 (5)
C295	1.0149 (2)	−0.10390 (11)	0.06069 (9)	0.082 (5)
C296	0.9350 (2)	−0.06816 (11)	0.08792 (9)	0.071 (4)

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

Mo1—P11	2.441 (3)	Mo2—P21	2.425 (4)
Mo1—P12	2.432 (3)	Mo2—P22	2.427 (4)
Mo1—P13	2.444 (3)	Mo2—P23	2.442 (4)
Mo1—C11	2.013 (13)	Mo2—C21	2.00 (2)
Mo1—C12	2.018 (13)	Mo2—C22	1.991 (15)
Mo1—C13	1.944 (2)	Mo2—C23	1.950 (2)
P11—Mo1—P12	85.58 (11)	P21—Mo2—P22	88.80 (14
P11—Mo1—P13	89.35 (11)	P21—Mo2—P23	92.05 (12
P12—Mo1—P13	95.05 (11)	P22—Mo2—P23	89.34 (12
C11—Mo1—P11	176.9 (3)	C21—Mo2—P21	171.0 (4)
C12—Mo1—P11	96.1 (4)	C22—Mo2—P21	96.7 (4)
C13—Mo1—P11	91.32 (10)	C23—Mo2—P21	87.31 (11)
C11—Mo1—P12	96.4 (4)	C21—Mo2—P22	87.0 (5)
C12—Mo1—P12	169.2 (3)	C22—Mo2—P22	173.7 (4)
C13—Mo1—P12	84.72 (11)	C23—Mo2—P22	98.36 (11
C11—Mo1—P13	88.1 (4)	C21—Mo2—P23	95.8 (4)
C12—Mo1—P13	95.6 (3)	C22—Mo2—P23	87.3 (4)
C13—Mo1—P13	179.27 (11)	C23—Mo2—P23	172.25 (12
C12—Mo1—C11	82.4 (5)	C22—Mo2—C21	88.0 (6)
C13—Mo1—C11	91.2 (4)	C23—Mo2—C21	85.5 (4)
C13—Mo1—C12	84.6 (3)	C23—Mo2—C22	85.1 (4)

The crystal diffracted very poorly [only 24% of the reflections had $I > 3\sigma(I)$]; because of the size of the refinement (two independent molecules) and the large number of weak reflections, the phenyl rings were constrained to be planar hexagons with C—C 1.39 Å. Examination of the structure with the *SOLV* option in *PLATON* (Spek, 1994*a*) showed that although there were no solvent-accessible voids in the crystal lattice, there were 13 small cavities (size 8–18 Å^3, sum of cavity volume 160 Å^3) in the unit cell. This undoubtedly contributed to the molecules being loosely held in the lattice and the poor diffraction of the data crystal. We attribute the high *R* factor to these numerous voids; many of the phenyl rings show marked anisotropy and are probably slightly disordered. This is entirely consistent with the small percentage of observed data.

Data collection: *Enraf–Nonius CAD*-4 *Software* (Enraf–Nonius 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD*-4 *Software*. Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *NRCVAX*94, *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*94, *PLATON* (Spek, 1994*a*), *PLUTON* (Spek 1994*b*), *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *NRCVAX*94, *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1001). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Two-Dimensional Iron Phosphate Templated by Ethylenediamine

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Abstract

[Fe(PO$_4$)(OH)].0.5(ethylenediamine) was synthesized hydrothermally at 453 K. It is isotypic with the layered gallophosphate obtained in ethylene glycol by Jones, Thomas, Qisheng, Hursthouse & Chen [*J. Chem. Soc. Chem. Commun.* (1991), pp. 1520–1522].

Comment

We have investigated the system FeO(OH)–H$_3$(PO$_4$)–HF–en–H$_2$O (en = ethylenediamine) in order to synthesize oxyfluorinated iron phosphates with open frameworks. By increasing the amine ratio in the mixture, we succeeded in obtaining five different phases, the structures of two of which [ULM-10 (Cavellec, Riou & Férey, 1994) and ULM-11 (Cavellec, Riou & Férey, 1995)] are published elsewhere. The phase described in this paper does not contain fluorine. It corresponds to the formula 0.5(en).[Fe(PO$_4$)(OH)] and is isotypic with the gallophosphate synthesized in ethylene glycol by Jones, Thomas, Qisheng, Hursthouse & Chen (1991). It presents a two-dimensional structure with alternating inorganic and organic layers (Fig. 1). The inorganic sheets (Fig. 2) are built up from chains of edge-sharing FeO$_5$(OH) octahedra bonded by PO$_4$ tetrahedra. These polyhedra form a chain of a type already encountered in linarite (Bachmann & Zemann, 1961; Hawthorne, 1990, 1994). Octahedra are linked one to another *via* their O-atom vertices O1 and O3, the O1 atoms belonging to hydroxy groups. The O3 apices are bonded to two Fe atoms and one P atom; they ensure the connection between the chains *via* PO$_4$ tetrahedra.

The organic layers are made up of ethylenediamine molecules. Strong hydrogen bonds *via* H atoms of the

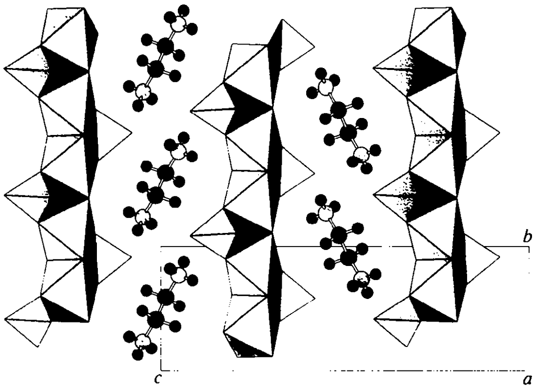

Fig. 1. Projection of the structure along the *a* axis showing its two-dimensional character.

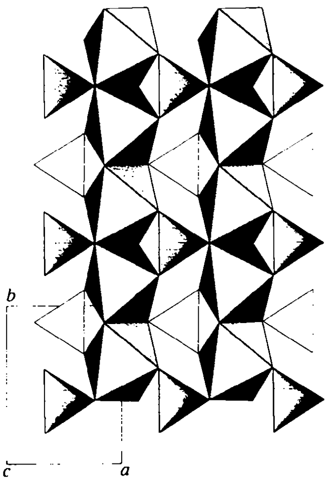

Fig. 2. Projection of an inorganic sheet along the *c* axis.