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Tetracarbonyl-2κ^4C-[μ-5-methyl-1,1,3-triphenyl-2-(propan-2-yl)-2,4-diaza-1,3-diphosphahexan-4-ido-1κN^4:2κP^1,P^3](*N*,*N*,*N*′,*N*′-tetramethylethane-1,2-diamine-1$\kappa^2 N$,*N*′)lithiummolybdenum

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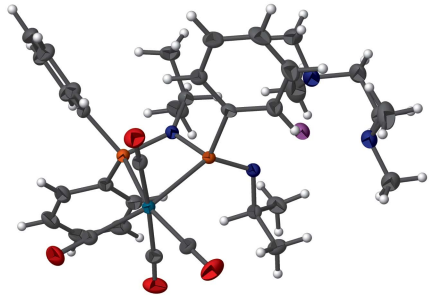
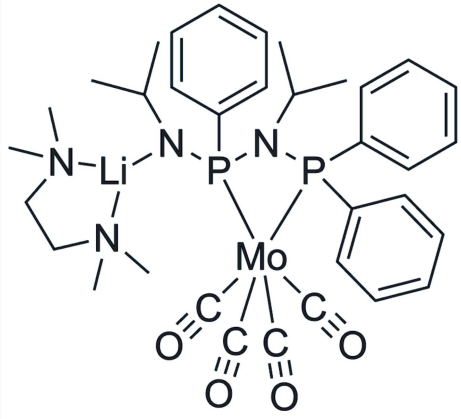
Structural data: full structural data are available from iucrdata.iucr.org

The title complex, [LiMo($C_6H_{16}N_2$)($C_{24}H_{29}N_2P_2$)(CO)$_4$], contains a distorted octahedrally coordinated molybdenum centre bearing a lithiated *P*,*P*′-*cis*-chelating PNPN ligand, which results in a nearly planar four-membered metallacycle. The Li atom is coordinated by one equivalent tetramethylethylenediamine. In the crystal, molecules are linked *via* weak C—H···O interactions, forming a chain along the *b*-axis direction.

3D view



Chemical scheme



Structure description

The title complex is very similar to the compound recently published by Höhne *et al.* (2018). Instead of the terminal amine function of the PNP chelating ligand, the deprotonated N2 atom is attached to a lithium ion coordinated by one tetramethylethylenediamine (tmeda) molecule. An analogous chromium compound was presumably prepared *in situ* by Dulai *et al.* (2011).

The molybdenum atom of the title compound (Fig. 1)exhibits a distorted octahedral geometry and is ligated by four carbonyl groups and the *P*,*P*′-cis-chelating PNPN fragment, which forms a nearly planar four-membered Mo/P/N/P metallacycle. The P—Mo—P bite angle is 65.714 (11)°, similar to those in comparable [Mo(CO)$_4${Ar$_2$PN(*R*)PAr$_2$}] complexes [range from 64.9 (1) to 66.14 (3)°; Al-Masri *et al.*, 2013; Biricik *et al.*, 2003; Gaw *et al.*, 2000, 2002; Majoumo *et al.*, 2004].

The P1—N1—P2 angle is 105.41 (6)°, which is slightly larger than that in the protonated complex [103.06 (7)°] prepared by Höhne *et al.* (2018). Nevertheless, it is obviously larger than in the uncoordinated [Ph$_2$PN(^{i}Pr)P(Ph)N(^{i}Pr)][Li(tmeda)]

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O3^{i}	0.95	2.54	3.4609 (19)	163

Symmetry code: (i) $x, y+1, z$.

[P1—N2—P2 117.77 (7)°; Peitz *et al.*, 2010]. In comparison with the calculated sum of the covalent radii by Pyykkö (2015) [single: Σr_{cov}(P—N) = 1.82 Å, double: Σr_{cov}(P═N) = 1.62 Å], the P—N bond lengths are shortened [range from 1.6142 (12) to 1.7508 (11) Å] and show some multiple-bond character. The central N1 atom is nearly trigonal planar [Σ(∠N1) = 359.96°]. As already observed at the protonated analogue, the Mo—P distances are different [Mo1—P1 2.5074 (3) Å and Mo1—P2 2.5362 (3) Å], which might be a result of the asymmetric character of the *P*,*P*′-cis-chelating PNPN ligand. In the crystal, a weak intermolecular C—H···O interaction is observed (C14—H14···O3^{i}; symmetry code as in Table 1) that links the complex molecules into chains along the *b*-axis direction.

Synthesis and crystallization

$Mo(CO)_6$ (0.99 g, 2.617 mmol) and $Ph_2PN(^iPr)P(Ph)NH(^iPr)$ (1.305 g, 3.193 mmol) were dissolved in CH_2Cl_2 (30 ml) at room temperature. After 2 h of refluxing, 20 ml CH_2Cl_2 were removed under vacuum. Ethanol (15 ml) was added and the solution was cooled down to −78°C. The white solid was washed with *n*-hexane at −78°C and dried under vacuum. Yield 1.45 g (90%). $[Mo(CO)_4\{Ph_2PN(^iPr)P(Ph)NH(^iPr)\}]$ (0.77 g, 1.25 mmol) and tetramethylethylenediamine (0.19 ml, 1.26 mmol) were dissolved in toluene (25 ml). The solution was cooled down to −78°C. *n*-BuLi (0.56 ml, 2.5 *M* in *n*-hexane) was added dropwise without stirring. After defrosting the solution, colourless crystals were obtained. Yield 0.76 g (82%). ^{1}H NMR (300 MHz, CD_2Cl_2, 298 K): δ (p.p.m.) 7.80–7.73 (*m*, 4H, Ar*H*), 7.57–7.46 (*m*, 2H, Ar*H*), 7.40–7.26 (*m*, 9H, Ar*H*), 3.75 (*m*, 1H, C*H*CH$_3$), 3.52 (*m*, 1H, C*H*CH$_3$), 2.14 [*br s*, 4H, $(CH_3)_2N(CH_2)_2N(CH_3)_2$], 1.92 [*br s*, 12H, $(CH_3)_2N(CH_2)_2N(CH_3)_2$], 1.20 (*d*, $^3J_{H,H}$ = 6.1 Hz, 3H, CHC*H*$_3$), 1.11 (*d*, $^3J_{H,H}$ = 6.4 Hz, 3H, CHC*H*$_3$), 1.01 (*d*, $^3J_{H,H}$ = 6.8 Hz, 3H, CHC*H*$_3$), 0.68 (*d*, $^3J_{H,H}$ = 6.7 Hz, 3H, CHC*H*$_3$). ^{13}C NMR (100 MHz, CD_2Cl_2, 298 K): δ (p.p.m.) 220.3 (*m*, *C*O), 213.3 (*m*, *C*O), 133.3, 133.1, 132.3, 132.1, 130.0, 129.8, 129.4, 129.2, 128.9, 128.8, 128.4, 128.2, 128.1, 128.0 (Ar*C*), 56.9 [*br s*, $(CH_3)_2N(CH_2)_2N(CH_3)_2$], 50.6 (*dd*, $^2J_{PC}$ = 8.5 Hz, 2.2 Hz, *C*HCH$_3$), 48.5 (*d*, $^2J_{PC}$ = 8.0 Hz, *C*HCH$_3$), 46.0 [*br s*, $(CH_3)_2N(CH_2)_2N(CH_3)_2$], 29.0, 28.9, 28.8, 28.7, (CHCH$_3$), 25.5, 24.3, (*br s*, CHCH$_3$). ^{31}P NMR (121 MHz, CD_2Cl_2, 298 K): δ = 98.7 (*d*, $^2J_{PP}$ = 8.9 Hz), 81.8 (*d*, $^2J_{PP}$ = 8.9 Hz). Elemental analysis calculated (%) for $C_{34}H_{45}LiMoN_4O_4P_2$ (738.57): C 55.29, H 6.14, N 7.59. Found: C 54.82, H 6.03, N 7.00. IR (CH_2Cl_2, cm^{-1}): ν (CO) 1870, 1896, 1918, 2005. M.p. 155°C (dec.).

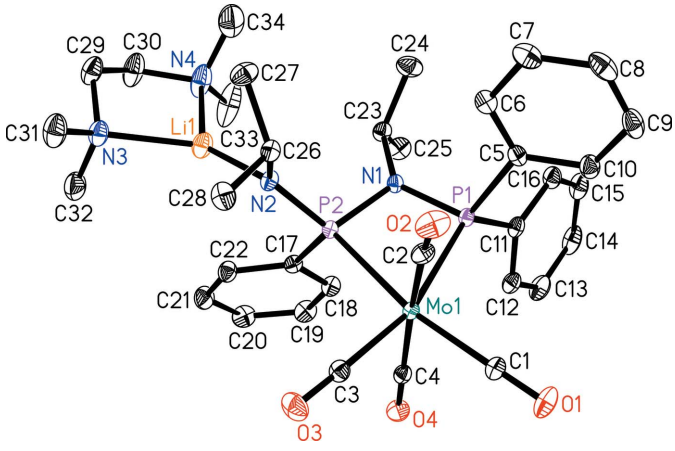


Figure 1
The molecular structure of the title compound with the atom labelling and displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[LiMo(C_6H_{16}N_2)(C_{24}H_{29}N_2P_2)(CO)_4]$
M_r	738.56
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	11.7633 (4), 12.5731 (4), 13.7249 (4)
α, β, γ (°)	87.283 (2), 75.537 (2), 67.450 (2)
V (Å^3)	1812.66 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.49
Crystal size (mm)	0.50 × 0.35 × 0.32
Data collection	
Diffractometer	Stoe IPDS II
Absorption correction	Multi-scan (*LANA*; Stoe & Cie, 2012)
T_{min}, T_{max}	0.79, 0.86
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31426, 8659, 7733
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.023, 0.061, 1.03
No. of reflections	8659
No. of parameters	423
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.33

Computer programs: *X-AREA* (Stoe & Cie, 2005), *SIR2004* (Burla *et al.*, 2005), *SHELXL2014* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2018). **3**, x181149 [https://doi.org/10.1107/S2414314618011495]

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Crystal data

$[LiMo(C_6H_{16}N_2)(C_{24}H_{29}N_2P_2)(CO)_4]$
M_r = 738.56
Triclinic, $P\overline{1}$
a = 11.7633 (4) Å
b = 12.5731 (4) Å
c = 13.7249 (4) Å
α = 87.283 (2)°
β = 75.537 (2)°
γ = 67.450 (2)°
V = 1812.66 (10) Å^3
Z = 2
$F(000)$ = 768
D_x = 1.353 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10549 reflections
θ = 1.7–28.4°
μ = 0.49 mm^{-1}
T = 150 K
Prism, colourless
0.50 × 0.35 × 0.32 mm

Data collection

Stoe IPDS II diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*LANA*; Stoe & Cie, 2012)
T_{min} = 0.79, T_{max} = 0.86
31426 measured reflections
8659 independent reflections
7733 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 27.9°, θ_{min} = 1.8°
h = −15→15
k = −16→16
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.061
S = 1.03
8659 reflections
423 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.02298 (13)	0.88601 (12)	0.32143 (11)	0.0258 (3)
C2	0.16950 (13)	0.72936 (13)	0.17894 (11)	0.0273 (3)
C3	0.13497 (14)	0.65850 (12)	0.37362 (11)	0.0264 (3)
C4	0.12911 (13)	0.85988 (11)	0.45555 (10)	0.0236 (3)
C5	0.25040 (13)	0.94608 (12)	0.09271 (10)	0.0245 (3)
C6	0.32937 (15)	0.86039 (13)	0.01871 (11)	0.0299 (3)
H6	0.3980	0.7971	0.0337	0.036*
C7	0.30915 (18)	0.86617 (16)	−0.07717 (12)	0.0385 (4)
H7	0.3649	0.8076	−0.1276	0.046*
C8	0.20834 (18)	0.95671 (17)	−0.09955 (13)	0.0411 (4)
H8	0.1949	0.9607	−0.1653	0.049*
C9	0.12755 (17)	1.04110 (16)	−0.02614 (14)	0.0408 (4)
H9	0.0575	1.1028	−0.0411	0.049*
C10	0.14768 (15)	1.03667 (14)	0.06965 (12)	0.0325 (3)
H10	0.0915	1.0954	0.1198	0.039*
C11	0.23017 (13)	1.07747 (11)	0.26445 (11)	0.0248 (3)
C12	0.17658 (14)	1.11238 (12)	0.36546 (12)	0.0297 (3)
H12	0.1480	1.0629	0.4105	0.036*
C13	0.16392 (16)	1.21857 (14)	0.40198 (14)	0.0393 (4)
H13	0.1269	1.2414	0.4714	0.047*
C14	0.20546 (16)	1.29100 (13)	0.33669 (16)	0.0432 (4)
H14	0.1983	1.3632	0.3614	0.052*
C15	0.25717 (16)	1.25830 (14)	0.23576 (16)	0.0403 (4)
H15	0.2847	1.3086	0.1911	0.048*
C16	0.26948 (14)	1.15245 (13)	0.19870 (13)	0.0315 (3)
H16	0.3045	1.1310	0.1289	0.038*
C17	0.44782 (12)	0.73527 (12)	0.40823 (10)	0.0227 (2)
C18	0.40542 (14)	0.84035 (13)	0.46161 (11)	0.0281 (3)
H18	0.3537	0.9084	0.4363	0.034*
C19	0.43714 (16)	0.84769 (15)	0.55102 (12)	0.0352 (3)
H19	0.4078	0.9204	0.5860	0.042*
C20	0.51155 (16)	0.74913 (16)	0.58928 (12)	0.0383 (4)
H20	0.5347	0.7541	0.6500	0.046*
C21	0.55215 (16)	0.64334 (15)	0.53884 (12)	0.0385 (4)
H21	0.6023	0.5753	0.5654	0.046*
C22	0.51966 (14)	0.63630 (13)	0.44930 (11)	0.0298 (3)
H22	0.5468	0.5631	0.4157	0.036*
C23	0.53105 (13)	0.86583 (12)	0.20044 (11)	0.0263 (3)
H23	0.5972	0.7921	0.2137	0.032*

C24	0.57085 (16)	0.88029 (17)	0.08935 (13)	0.0406 (4)
H24A	0.5799	0.8120	0.0517	0.061*
H24B	0.6524	0.8897	0.0734	0.061*
H24C	0.5062	0.9487	0.0705	0.061*
C25	0.53846 (16)	0.95936 (15)	0.26222 (13)	0.0364 (3)
H25A	0.4815	1.0352	0.2472	0.055*
H25B	0.6259	0.9558	0.2451	0.055*
H25C	0.5128	0.9473	0.3341	0.055*
C26	0.49594 (13)	0.55535 (12)	0.15393 (10)	0.0251 (3)
H26	0.4295	0.6145	0.1252	0.030*
C27	0.62026 (16)	0.51011 (16)	0.07195 (12)	0.0378 (3)
H27A	0.6428	0.5749	0.0448	0.057*
H27B	0.6098	0.4694	0.0178	0.057*
H27C	0.6880	0.4570	0.1007	0.057*
C28	0.45166 (17)	0.45833 (14)	0.19039 (12)	0.0355 (3)
H28A	0.5145	0.4012	0.2211	0.053*
H28B	0.4422	0.4215	0.1332	0.053*
H28C	0.3697	0.4897	0.2404	0.053*
C29	0.93139 (16)	0.38160 (15)	0.21864 (16)	0.0440 (4)
H29A	0.9368	0.3773	0.1457	0.053*
H29B	1.0011	0.3130	0.2333	0.053*
C30	0.94864 (18)	0.48891 (16)	0.2441 (2)	0.0556 (6)
H30A	0.9509	0.4898	0.3156	0.067*
H30B	1.0310	0.4877	0.2022	0.067*
C31	0.79815 (19)	0.27593 (16)	0.24283 (18)	0.0513 (5)
H31A	0.8679	0.2076	0.2558	0.077*
H31B	0.8027	0.2774	0.1705	0.077*
H31C	0.7166	0.2732	0.2800	0.077*
C32	0.8033 (2)	0.37508 (18)	0.38413 (16)	0.0554 (5)
H32A	0.7266	0.3628	0.4205	0.083*
H32B	0.8008	0.4479	0.4091	0.083*
H32C	0.8787	0.3114	0.3950	0.083*
C33	0.8355 (2)	0.69034 (18)	0.2894 (3)	0.0849 (10)
H33A	0.9188	0.6960	0.2762	0.127*
H33B	0.8080	0.6766	0.3606	0.127*
H33C	0.7734	0.7625	0.2730	0.127*
C34	0.8721 (2)	0.6192 (2)	0.1196 (2)	0.0725 (8)
H34A	0.9527	0.6301	0.1004	0.109*
H34B	0.8034	0.6896	0.1082	0.109*
H34C	0.8780	0.5546	0.0787	0.109*
Li1	0.6848 (3)	0.5480 (2)	0.2588 (2)	0.0360 (6)
Mo1	0.16120 (2)	0.79639 (2)	0.31354 (2)	0.01809 (4)
N1	0.40931 (10)	0.84881 (9)	0.23155 (8)	0.0210 (2)
N2	0.51403 (11)	0.61040 (10)	0.23746 (9)	0.0229 (2)
N3	0.80895 (13)	0.37986 (12)	0.27596 (11)	0.0364 (3)
N4	0.84506 (15)	0.59430 (13)	0.22659 (16)	0.0523 (4)
O1	−0.12821 (11)	0.93646 (11)	0.32469 (10)	0.0424 (3)
O2	0.16212 (12)	0.69253 (12)	0.10722 (9)	0.0442 (3)

O3	0.12213 (13)	0.57696 (10)	0.40721 (10)	0.0433 (3)
O4	0.10544 (11)	0.89290 (10)	0.53707 (8)	0.0340 (2)
P1	0.25981 (3)	0.93050 (3)	0.22417 (2)	0.01956 (7)
P2	0.39924 (3)	0.72621 (3)	0.29287 (2)	0.01817 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0241 (7)	0.0233 (6)	0.0302 (7)	−0.0076 (5)	−0.0086 (5)	−0.0032 (5)
C2	0.0222 (6)	0.0301 (7)	0.0303 (7)	−0.0112 (5)	−0.0049 (5)	−0.0034 (6)
C3	0.0275 (7)	0.0217 (6)	0.0301 (7)	−0.0098 (5)	−0.0064 (6)	−0.0013 (5)
C4	0.0212 (6)	0.0207 (6)	0.0281 (7)	−0.0074 (5)	−0.0057 (5)	0.0000 (5)
C5	0.0262 (6)	0.0266 (6)	0.0268 (6)	−0.0148 (5)	−0.0108 (5)	0.0069 (5)
C6	0.0341 (8)	0.0296 (7)	0.0308 (7)	−0.0156 (6)	−0.0115 (6)	0.0031 (6)
C7	0.0514 (10)	0.0457 (9)	0.0293 (7)	−0.0292 (8)	−0.0123 (7)	0.0021 (7)
C8	0.0559 (11)	0.0576 (11)	0.0333 (8)	−0.0399 (9)	−0.0250 (8)	0.0164 (8)
C9	0.0382 (9)	0.0488 (10)	0.0510 (10)	−0.0258 (8)	−0.0275 (8)	0.0236 (8)
C10	0.0275 (7)	0.0341 (8)	0.0393 (8)	−0.0136 (6)	−0.0131 (6)	0.0096 (6)
C11	0.0204 (6)	0.0185 (6)	0.0361 (7)	−0.0066 (5)	−0.0092 (5)	0.0001 (5)
C12	0.0253 (7)	0.0232 (7)	0.0387 (8)	−0.0067 (5)	−0.0076 (6)	−0.0033 (6)
C13	0.0348 (8)	0.0280 (8)	0.0515 (10)	−0.0053 (6)	−0.0126 (7)	−0.0130 (7)
C14	0.0365 (9)	0.0207 (7)	0.0769 (13)	−0.0079 (6)	−0.0256 (9)	−0.0066 (7)
C15	0.0337 (8)	0.0236 (7)	0.0698 (12)	−0.0145 (6)	−0.0190 (8)	0.0088 (7)
C16	0.0284 (7)	0.0239 (7)	0.0442 (8)	−0.0116 (6)	−0.0107 (6)	0.0059 (6)
C17	0.0200 (6)	0.0268 (7)	0.0234 (6)	−0.0100 (5)	−0.0073 (5)	−0.0003 (5)
C18	0.0286 (7)	0.0272 (7)	0.0295 (7)	−0.0101 (6)	−0.0093 (6)	−0.0021 (5)
C19	0.0365 (8)	0.0385 (8)	0.0340 (8)	−0.0153 (7)	−0.0114 (7)	−0.0082 (6)
C20	0.0370 (8)	0.0540 (10)	0.0285 (7)	−0.0169 (8)	−0.0159 (7)	−0.0027 (7)
C21	0.0385 (9)	0.0426 (9)	0.0312 (8)	−0.0074 (7)	−0.0170 (7)	0.0043 (7)
C22	0.0300 (7)	0.0290 (7)	0.0281 (7)	−0.0066 (6)	−0.0105 (6)	−0.0003 (6)
C23	0.0207 (6)	0.0271 (7)	0.0337 (7)	−0.0117 (5)	−0.0073 (5)	0.0022 (5)
C24	0.0354 (8)	0.0584 (11)	0.0359 (8)	−0.0281 (8)	−0.0076 (7)	0.0083 (8)
C25	0.0326 (8)	0.0400 (9)	0.0458 (9)	−0.0227 (7)	−0.0108 (7)	−0.0015 (7)
C26	0.0261 (7)	0.0238 (6)	0.0251 (6)	−0.0069 (5)	−0.0099 (5)	−0.0018 (5)
C27	0.0332 (8)	0.0479 (9)	0.0291 (7)	−0.0114 (7)	−0.0066 (6)	−0.0105 (7)
C28	0.0460 (9)	0.0290 (7)	0.0377 (8)	−0.0175 (7)	−0.0158 (7)	−0.0006 (6)
C29	0.0286 (8)	0.0331 (8)	0.0678 (12)	−0.0073 (7)	−0.0141 (8)	−0.0023 (8)
C30	0.0299 (9)	0.0364 (9)	0.1060 (18)	−0.0113 (7)	−0.0287 (10)	0.0033 (10)
C31	0.0434 (10)	0.0342 (9)	0.0819 (15)	−0.0159 (8)	−0.0241 (10)	0.0028 (9)
C32	0.0532 (12)	0.0465 (11)	0.0512 (11)	−0.0001 (9)	−0.0184 (9)	0.0083 (9)
C33	0.0559 (13)	0.0360 (11)	0.181 (3)	−0.0120 (10)	−0.0674 (18)	−0.0123 (14)
C34	0.0382 (11)	0.0636 (14)	0.114 (2)	−0.0225 (10)	−0.0164 (12)	0.0302 (14)
Li1	0.0283 (13)	0.0327 (13)	0.0494 (16)	−0.0098 (11)	−0.0169 (12)	0.0011 (12)
Mo1	0.01671 (6)	0.01617 (6)	0.02204 (6)	−0.00650 (4)	−0.00546 (4)	−0.00026 (4)
N1	0.0196 (5)	0.0194 (5)	0.0256 (5)	−0.0083 (4)	−0.0076 (4)	0.0027 (4)
N2	0.0219 (5)	0.0211 (5)	0.0253 (5)	−0.0059 (4)	−0.0083 (4)	−0.0032 (4)
N3	0.0302 (7)	0.0310 (7)	0.0485 (8)	−0.0087 (5)	−0.0158 (6)	0.0029 (6)
N4	0.0319 (8)	0.0330 (8)	0.0991 (14)	−0.0127 (6)	−0.0286 (8)	0.0040 (8)

O1	0.0260 (6)	0.0400 (6)	0.0573 (7)	−0.0026 (5)	−0.0181 (5)	−0.0085 (6)
O2	0.0405 (7)	0.0591 (8)	0.0374 (6)	−0.0233 (6)	−0.0076 (5)	−0.0162 (6)
O3	0.0598 (8)	0.0285 (6)	0.0466 (7)	−0.0248 (6)	−0.0101 (6)	0.0057 (5)
O4	0.0367 (6)	0.0340 (6)	0.0292 (5)	−0.0123 (5)	−0.0052 (4)	−0.0052 (4)
P1	0.01917 (15)	0.01714 (15)	0.02327 (15)	−0.00716 (12)	−0.00659 (12)	0.00109 (12)
P2	0.01789 (15)	0.01743 (15)	0.02011 (15)	−0.00651 (12)	−0.00651 (12)	−0.00051 (11)

Geometric parameters (Å, º)

C1—O1	1.1443 (18)	C24—H24A	0.9800
C1—Mo1	1.9993 (14)	C24—H24B	0.9800
C2—O2	1.1435 (18)	C24—H24C	0.9800
C2—Mo1	2.0315 (14)	C25—H25A	0.9800
C3—O3	1.1513 (18)	C25—H25B	0.9800
C3—Mo1	1.9820 (14)	C25—H25C	0.9800
C4—O4	1.1408 (18)	C26—N2	1.4658 (16)
C4—Mo1	2.0317 (14)	C26—C28	1.520 (2)
C5—C6	1.386 (2)	C26—C27	1.526 (2)
C5—C10	1.399 (2)	C26—H26	1.0000
C5—P1	1.8319 (14)	C27—H27A	0.9800
C6—C7	1.388 (2)	C27—H27B	0.9800
C6—H6	0.9500	C27—H27C	0.9800
C7—C8	1.382 (3)	C28—H28A	0.9800
C7—H7	0.9500	C28—H28B	0.9800
C8—C9	1.376 (3)	C28—H28C	0.9800
C8—H8	0.9500	C29—N3	1.466 (2)
C9—C10	1.388 (2)	C29—C30	1.508 (3)
C9—H9	0.9500	C29—Li1	2.784 (3)
C10—H10	0.9500	C29—H29A	0.9900
C11—C12	1.386 (2)	C29—H29B	0.9900
C11—C16	1.4007 (19)	C30—N4	1.475 (2)
C11—P1	1.8298 (14)	C30—H30A	0.9900
C12—C13	1.389 (2)	C30—H30B	0.9900
C12—H12	0.9500	C31—N3	1.464 (2)
C13—C14	1.384 (3)	C31—H31A	0.9800
C13—H13	0.9500	C31—H31B	0.9800
C14—C15	1.378 (3)	C31—H31C	0.9800
C14—H14	0.9500	C32—N3	1.469 (2)
C15—C16	1.389 (2)	C32—H32A	0.9800
C15—H15	0.9500	C32—H32B	0.9800
C16—H16	0.9500	C32—H32C	0.9800
C17—C18	1.391 (2)	C33—N4	1.469 (3)
C17—C22	1.3948 (19)	C33—H33A	0.9800
C17—P2	1.8344 (13)	C33—H33B	0.9800
C18—C19	1.385 (2)	C33—H33C	0.9800
C18—H18	0.9500	C34—N4	1.469 (3)
C19—C20	1.383 (2)	C34—H34A	0.9800
C19—H19	0.9500	C34—H34B	0.9800

C20—C21	1.383 (2)	C34—H34C	0.9800
C20—H20	0.9500	Li1—N2	1.949 (3)
C21—C22	1.391 (2)	Li1—N3	2.101 (3)
C21—H21	0.9500	Li1—N4	2.119 (3)
C22—H22	0.9500	Mo1—P1	2.5074 (3)
C23—N1	1.4833 (16)	Mo1—P2	2.5362 (3)
C23—C24	1.503 (2)	N1—P1	1.6886 (11)
C23—C25	1.523 (2)	N1—P2	1.7508 (11)
C23—H23	1.0000	N2—P2	1.6142 (12)

O1—C1—Mo1	179.00 (13)	H28B—C28—H28C	109.5
O2—C2—Mo1	173.23 (12)	N3—C29—C30	112.05 (16)
O3—C3—Mo1	178.57 (14)	N3—C29—Li1	47.77 (9)
O4—C4—Mo1	175.82 (12)	C30—C29—Li1	77.15 (11)
C6—C5—C10	118.61 (13)	N3—C29—H29A	109.2
C6—C5—P1	121.50 (10)	C30—C29—H29A	109.2
C10—C5—P1	118.97 (12)	Li1—C29—H29A	92.2
C5—C6—C7	120.63 (15)	N3—C29—H29B	109.2
C5—C6—H6	119.7	C30—C29—H29B	109.2
C7—C6—H6	119.7	Li1—C29—H29B	154.6
C8—C7—C6	120.29 (17)	H29A—C29—H29B	107.9
C8—C7—H7	119.9	N4—C30—C29	111.58 (15)
C6—C7—H7	119.9	N4—C30—H30A	109.3
C9—C8—C7	119.67 (14)	C29—C30—H30A	109.3
C9—C8—H8	120.2	N4—C30—H30B	109.3
C7—C8—H8	120.2	C29—C30—H30B	109.3
C8—C9—C10	120.46 (15)	H30A—C30—H30B	108.0
C8—C9—H9	119.8	N3—C31—H31A	109.5
C10—C9—H9	119.8	N3—C31—H31B	109.5
C9—C10—C5	120.31 (16)	H31A—C31—H31B	109.5
C9—C10—H10	119.8	N3—C31—H31C	109.5
C5—C10—H10	119.8	H31A—C31—H31C	109.5
C12—C11—C16	118.73 (13)	H31B—C31—H31C	109.5
C12—C11—P1	118.68 (10)	N3—C32—H32A	109.5
C16—C11—P1	122.36 (12)	N3—C32—H32B	109.5
C11—C12—C13	121.11 (15)	H32A—C32—H32B	109.5
C11—C12—H12	119.4	N3—C32—H32C	109.5
C13—C12—H12	119.4	H32A—C32—H32C	109.5
C14—C13—C12	119.66 (17)	H32B—C32—H32C	109.5
C14—C13—H13	120.2	N4—C33—H33A	109.5
C12—C13—H13	120.2	N4—C33—H33B	109.5
C15—C14—C13	119.93 (15)	H33A—C33—H33B	109.5
C15—C14—H14	120.0	N4—C33—H33C	109.5
C13—C14—H14	120.0	H33A—C33—H33C	109.5
C14—C15—C16	120.66 (16)	H33B—C33—H33C	109.5
C14—C15—H15	119.7	N4—C34—H34A	109.5
C16—C15—H15	119.7	N4—C34—H34B	109.5
C15—C16—C11	119.88 (16)	H34A—C34—H34B	109.5

C15—C16—H16	120.1	N4—C34—H34C	109.5
C11—C16—H16	120.1	H34A—C34—H34C	109.5
C18—C17—C22	117.96 (12)	H34B—C34—H34C	109.5
C18—C17—P2	120.72 (10)	N2—Li1—N3	132.75 (15)
C22—C17—P2	121.12 (11)	N2—Li1—N4	137.04 (15)
C19—C18—C17	121.32 (14)	N3—Li1—N4	87.21 (11)
C19—C18—H18	119.3	N2—Li1—C29	151.06 (15)
C17—C18—H18	119.3	N3—Li1—C29	31.12 (7)
C20—C19—C18	119.98 (15)	N4—Li1—C29	58.55 (8)
C20—C19—H19	120.0	C3—Mo1—C1	94.46 (6)
C18—C19—H19	120.0	C3—Mo1—C2	86.82 (6)
C21—C20—C19	119.72 (14)	C1—Mo1—C2	86.04 (6)
C21—C20—H20	120.1	C3—Mo1—C4	88.02 (6)
C19—C20—H20	120.1	C1—Mo1—C4	87.95 (5)
C20—C21—C22	120.10 (15)	C2—Mo1—C4	171.75 (5)
C20—C21—H21	119.9	C3—Mo1—P1	163.43 (4)
C22—C21—H21	119.9	C1—Mo1—P1	101.60 (4)
C21—C22—C17	120.86 (14)	C2—Mo1—P1	90.19 (4)
C21—C22—H22	119.6	C4—Mo1—P1	96.55 (4)
C17—C22—H22	119.6	C3—Mo1—P2	98.44 (4)
N1—C23—C24	113.36 (11)	C1—Mo1—P2	166.92 (4)
N1—C23—C25	114.76 (12)	C2—Mo1—P2	96.77 (4)
C24—C23—C25	111.33 (13)	C4—Mo1—P2	90.35 (4)
N1—C23—H23	105.5	P1—Mo1—P2	65.714 (11)
C24—C23—H23	105.5	C23—N1—P1	132.93 (9)
C25—C23—H23	105.5	C23—N1—P2	121.62 (9)
C23—C24—H24A	109.5	P1—N1—P2	105.41 (6)
C23—C24—H24B	109.5	C26—N2—P2	118.00 (9)
H24A—C24—H24B	109.5	C26—N2—Li1	115.88 (12)
C23—C24—H24C	109.5	P2—N2—Li1	125.54 (10)
H24A—C24—H24C	109.5	C31—N3—C29	108.10 (15)
H24B—C24—H24C	109.5	C31—N3—C32	108.49 (16)
C23—C25—H25A	109.5	C29—N3—C32	110.32 (15)
C23—C25—H25B	109.5	C31—N3—Li1	123.53 (13)
H25A—C25—H25B	109.5	C29—N3—Li1	101.10 (13)
C23—C25—H25C	109.5	C32—N3—Li1	104.79 (14)
H25A—C25—H25C	109.5	C34—N4—C33	110.4 (2)
H25B—C25—H25C	109.5	C34—N4—C30	109.63 (19)
N2—C26—C28	110.82 (11)	C33—N4—C30	108.67 (16)
N2—C26—C27	109.31 (11)	C34—N4—Li1	107.85 (15)
C28—C26—C27	110.72 (13)	C33—N4—Li1	116.21 (18)
N2—C26—H26	108.6	C30—N4—Li1	103.82 (13)
C28—C26—H26	108.6	N1—P1—C11	106.08 (6)
C27—C26—H26	108.6	N1—P1—C5	111.02 (6)
C26—C27—H27A	109.5	C11—P1—C5	102.27 (6)
C26—C27—H27B	109.5	N1—P1—Mo1	95.73 (4)
H27A—C27—H27B	109.5	C11—P1—Mo1	128.14 (5)
C26—C27—H27C	109.5	C5—P1—Mo1	112.88 (4)

H27A—C27—H27C	109.5	N2—P2—N1	111.07 (6)
H27B—C27—H27C	109.5	N2—P2—C17	100.63 (6)
C26—C28—H28A	109.5	N1—P2—C17	102.74 (6)
C26—C28—H28B	109.5	N2—P2—Mo1	130.98 (4)
H28A—C28—H28B	109.5	N1—P2—Mo1	93.14 (4)
C26—C28—H28C	109.5	C17—P2—Mo1	115.17 (4)
H28A—C28—H28C	109.5		

C10—C5—C6—C7	1.9 (2)	C29—C30—N4—C34	−82.7 (2)
P1—C5—C6—C7	170.81 (11)	C29—C30—N4—C33	156.6 (2)
C5—C6—C7—C8	−1.1 (2)	C29—C30—N4—Li1	32.3 (2)
C6—C7—C8—C9	−0.3 (2)	C23—N1—P1—C11	−44.51 (14)
C7—C8—C9—C10	0.9 (2)	P2—N1—P1—C11	133.43 (6)
C8—C9—C10—C5	−0.1 (2)	C23—N1—P1—C5	65.84 (14)
C6—C5—C10—C9	−1.3 (2)	P2—N1—P1—C5	−116.22 (6)
P1—C5—C10—C9	−170.48 (11)	C23—N1—P1—Mo1	−176.96 (12)
C16—C11—C12—C13	−1.2 (2)	P2—N1—P1—Mo1	0.98 (5)
P1—C11—C12—C13	173.37 (12)	C12—C11—P1—N1	−91.04 (11)
C11—C12—C13—C14	0.0 (2)	C16—C11—P1—N1	83.37 (12)
C12—C13—C14—C15	1.0 (2)	C12—C11—P1—C5	152.55 (11)
C13—C14—C15—C16	−0.7 (2)	C16—C11—P1—C5	−33.03 (13)
C14—C15—C16—C11	−0.6 (2)	C12—C11—P1—Mo1	19.96 (13)
C12—C11—C16—C15	1.6 (2)	C16—C11—P1—Mo1	−165.62 (9)
P1—C11—C16—C15	−172.85 (11)	C6—C5—P1—N1	30.47 (13)
C22—C17—C18—C19	2.3 (2)	C10—C5—P1—N1	−160.64 (10)
P2—C17—C18—C19	177.34 (12)	C6—C5—P1—C11	143.26 (11)
C17—C18—C19—C20	−0.5 (2)	C10—C5—P1—C11	−47.85 (12)
C18—C19—C20—C21	−1.1 (3)	C6—C5—P1—Mo1	−75.68 (12)
C19—C20—C21—C22	0.8 (3)	C10—C5—P1—Mo1	93.21 (11)
C20—C21—C22—C17	1.0 (3)	C26—N2—P2—N1	−87.20 (10)
C18—C17—C22—C21	−2.6 (2)	Li1—N2—P2—N1	83.66 (13)
P2—C17—C22—C21	−177.56 (12)	C26—N2—P2—C17	164.57 (10)
N3—C29—C30—N4	−57.2 (3)	Li1—N2—P2—C17	−24.57 (14)
Li1—C29—C30—N4	−23.90 (18)	C26—N2—P2—Mo1	27.16 (12)
C24—C23—N1—P1	−60.19 (18)	Li1—N2—P2—Mo1	−161.98 (11)
C25—C23—N1—P1	69.28 (17)	C23—N1—P2—N2	−46.27 (12)
C24—C23—N1—P2	122.14 (13)	P1—N1—P2—N2	135.50 (6)
C25—C23—N1—P2	−108.39 (13)	C23—N1—P2—C17	60.58 (11)
C28—C26—N2—P2	−96.22 (13)	P1—N1—P2—C17	−117.65 (6)
C27—C26—N2—P2	141.48 (11)	C23—N1—P2—Mo1	177.26 (10)
C28—C26—N2—Li1	92.04 (16)	P1—N1—P2—Mo1	−0.97 (5)
C27—C26—N2—Li1	−30.26 (17)	C18—C17—P2—N2	156.25 (11)
C30—C29—N3—C31	177.27 (16)	C22—C17—P2—N2	−28.91 (13)
Li1—C29—N3—C31	131.02 (17)	C18—C17—P2—N1	41.57 (12)
C30—C29—N3—C32	−64.2 (2)	C22—C17—P2—N1	−143.59 (12)
Li1—C29—N3—C32	−110.49 (16)	C18—C17—P2—Mo1	−58.12 (12)
C30—C29—N3—Li1	46.25 (19)	C22—C17—P2—Mo1	116.72 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O3^{i}	0.95	2.54	3.4609 (19)	163

Symmetry code: (i) *x*, *y*+1, *z*.