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Table 1. Crystal data, structure solution and refinement for 2.

Chemical formula	$C_{68}H_{76}Li_2O_4P_4$
Formula weight	1095.05
Temperature	160(2) K
Radiation and wavelength	Synchotron, 0.68750 Å
Crystal system, space group	triclinic, $P\bar{1}$
Unit cell dimensions	a = 11.314(2) Å α = 114.754(4)°
	b = 12.268(3) Å β = 109.517(6)°
	c = 12.857(3) Å γ = 93.354(6)°
Volume	1485.8(6) $Å^3$
Z	1
Density (calculated)	1.224 g/cm^3
Absorption coefficient μ	0.175 mm^{-1}
F(000)	582
Reflections for cell refinement	5896 (θ range 1.83 to 26.92°)
Crystal colour	yellow
Crystal size	0.16 × 0.08 × 0.04 mm
Data collection method	Siemens SMART CCD diffractometer,
	ω rotation with narrow frames
θ range for data collection	1.84 to 22.49°
Index ranges	$-11 \le h \le 11$, $-13 \le k \le 6$, $-13 \le \ell \le 14$
Reflections collected	5024
Independent reflections	3825 (R_{int} = 0.0551)
Reflections with I>2σ(I)	3291
Absorption correction	semi-empirical from ψ-scans
Max. and min. transmission	0.9930 and 0.9725
Structure solution	direct methods
Refinement method	full-matrix least-squares on F^2
Weighting parameters a, b	0.1838, 0.0000
Data / restraints / parameters	3825 / 34 / 352
Final R indices [I>2σ(I)]	R1 = 0.0898, wR2 = 0.2349
R indices (all data)	R1 = 0.0942, wR2 = 0.2407
Goodness-of-fit on F^2	1.040
Largest and mean shift/esd	0.001 and 0.000
Largest diff. peak and hole	0.547 and −0.515 $eÅ^{-3}$

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2**. U(eq) is defined as one third of the trace of the orthogonal U_{ij} tensor.

	x	y	z	U(eq)
Li(1)	4482(6)	1098(5)	3359(5)	31.9(14)
P(1)	2697.4(8)	1875.1(7)	2248.9(7)	21.2(4)
P(2)	2405.1(8)	-578.1(7)	1871.1(8)	23.0(4)
C(1)	1593(3)	468(3)	1510(3)	22.3(8)
C(2)	157(3)	162(3)	680(3)	24.0(8)
C(3)	2627(3)	2499(3)	1153(3)	22.9(8)
C(4)	3068(3)	1860(3)	237(3)	33.5(9)
C(5)	3092(4)	2266(4)	-617(4)	41.2(10)
C(6)	2707(4)	3311(4)	-561(4)	44.2(11)
C(7)	2252(4)	3962(4)	336(4)	46.1(11)
C(8)	2224(4)	3551(3)	1178(3)	34.3(9)
C(9)	2251(3)	3114(3)	3372(3)	26.8(8)
C(10)	3103(4)	4280(3)	4103(4)	38.4(10)
C(11)	2907(5)	5209(4)	5060(4)	47.1(11)
C(12)	1863(5)	4974(4)	5328(4)	50.2(12)
C(13)	1023(4)	3827(4)	4632(4)	42.7(10)
C(14)	1230(4)	2914(3)	3658(3)	32.7(9)
C(15)	1438(3)	-1376(3)	2364(3)	28.0(9)
C(16)	701(3)	-770(4)	2979(3)	37.2(10)
C(17)	24(4)	-1341(4)	3413(4)	49.5(12)
C(18)	102(4)	-2529(4)	3228(4)	56.7(14)
C(19)	850(5)	-3127(4)	2641(5)	55.4(12)
C(20)	1499(4)	-2566(4)	2217(4)	46.3(11)
C(21)	2330(3)	-1961(3)	478(3)	25.6(8)
C(22)	1184(4)	-2728(3)	-507(3)	35.5(9)
C(23)	1188(4)	-3771(3)	-1509(4)	43.6(11)
C(24)	2327(5)	-4085(3)	-1521(4)	44.6(11)
C(25)	3476(4)	-3329(4)	-567(4)	45.5(11)
C(26)	3464(4)	-2270(3)	424(4)	37.4(9)
O(1)	6033(2)	1141(3)	3101(2)	46.8(8)
C(27)	7281(4)	1250(6)	3963(4)	66.0(15)
C(28)	8150(5)	1198(8)	3410(6)	103(3)
C(29)	7581(4)	1388(4)	2320(4)	45.3(11)
C(30)	6163(4)	1189(4)	2046(4)	43.1(10)
O(2)	4853(3)	1736(2)	5103(2)	38.0(7)
C(31)	4121(6)	1220(4)	5563(5)	70.0(16)
C(32)	3662(6)	2242(6)	6315(5)	86.4(19)
C(33)	4716(7)	3324(5)	6849(5)	83.9(19)
C(34)	5315(4)	3038(4)	5944(4)	55.7(13)

Table 3. Bond lengths (Å) and angles (deg.) for **2**.

Li(1)-O(1)	1.894(7)	Li(1)-O(2)	1.918(6)
Li(1)-P(1)	2.542(6)	Li(1)-P(2)	2.559(6)
P(1)-C(1)	1.739(3)	P(1)-C(9)	1.841(3)
P(1)-C(3)	1.848(3)	P(2)-C(1)	1.749(3)
P(2)-C(15)	1.848(3)	P(2)-C(21)	1.852(3)
C(1)-C(2)	1.535(4)	C(2)-C(2#1)	1.527(6)
C(3)-C(4)	1.384(5)	C(3)-C(8)	1.384(5)
C(4)-C(5)	1.389(5)	C(5)-C(6)	1.360(6)
C(6)-C(7)	1.380(6)	C(7)-C(8)	1.382(6)
C(9)-C(14)	1.363(5)	C(9)-C(10)	1.403(5)
C(10)-C(11)	1.378(5)	C(11)-C(12)	1.384(6)
C(12)-C(13)	1.380(6)	C(13)-C(14)	1.388(5)
C(15)-C(16)	1.375(5)	C(15)-C(20)	1.400(5)
C(16)-C(17)	1.392(6)	C(17)-C(18)	1.389(7)
C(18)-C(19)	1.364(7)	C(19)-C(20)	1.353(6)
C(21)-C(26)	1.375(5)	C(21)-C(22)	1.390(5)
C(22)-C(23)	1.387(5)	C(23)-C(24)	1.369(6)
C(24)-C(25)	1.377(6)	C(25)-C(26)	1.392(5)
O(1)-C(27)	1.431(4)	O(1)-C(30)	1.437(4)
C(27)-C(28)	1.383(6)	C(28)-C(29)	1.457(6)
C(29)-C(30)	1.504(6)	O(2)-C(31)	1.420(5)
O(2)-C(34)	1.439(5)	C(31)-C(32)	1.474(7)
C(32)-C(33)	1.470(7)	C(33)-C(34)	1.466(7)

O(1)-Li(1)-O(2)	110.2(3)	O(1)-Li(1)-P(1)	119.0(3)
O(2)-Li(1)-P(1)	113.5(3)	O(1)-Li(1)-P(2)	124.7(3)
O(2)-Li(1)-P(2)	115.9(3)	P(1)-Li(1)-P(2)	67.14(14)
C(1)-P(1)-C(9)	112.26(16)	C(1)-P(1)-C(3)	111.23(15)
C(9)-P(1)-C(3)	99.60(15)	C(1)-P(1)-Li(1)	92.73(17)
C(9)-P(1)-Li(1)	111.02(17)	C(3)-P(1)-Li(1)	130.15(17)
C(1)-P(2)-C(15)	108.41(16)	C(1)-P(2)-C(21)	113.35(15)
C(15)-P(2)-C(21)	97.00(15)	C(1)-P(2)-Li(1)	91.91(17)
C(15)-P(2)-Li(1)	124.30(17)	C(21)-P(2)-Li(1)	121.99(17)
C(2)-C(1)-P(1)	127.3(2)	C(2)-C(1)-P(2)	124.7(2)
P(1)-C(1)-P(2)	107.92(17)	C(2#1)-C(2)-C(1)	115.7(3)
C(4)-C(3)-C(8)	117.1(3)	C(4)-C(3)-P(1)	115.8(3)
C(8)-C(3)-P(1)	127.0(3)	C(3)-C(4)-C(5)	120.7(4)
C(6)-C(5)-C(4)	120.9(4)	C(5)-C(6)-C(7)	119.7(4)
C(6)-C(7)-C(8)	119.1(4)	C(7)-C(8)-C(3)	122.4(4)
C(14)-C(9)-C(10)	117.7(3)	C(14)-C(9)-P(1)	123.2(3)
C(10)-C(9)-P(1)	118.5(3)	C(11)-C(10)-C(9)	121.7(4)
C(10)-C(11)-C(12)	119.1(4)	C(13)-C(12)-C(11)	120.1(4)
C(12)-C(13)-C(14)	119.6(4)	C(9)-C(14)-C(13)	121.8(4)
C(16)-C(15)-C(20)	117.7(3)	C(16)-C(15)-P(2)	120.2(3)
C(20)-C(15)-P(2)	122.0(3)	C(15)-C(16)-C(17)	120.6(4)
C(18)-C(17)-C(16)	119.5(5)	C(19)-C(18)-C(17)	120.3(4)
C(20)-C(19)-C(18)	119.7(4)	C(19)-C(20)-C(15)	122.2(4)
C(26)-C(21)-C(22)	117.8(3)	C(26)-C(21)-P(2)	118.6(3)
C(22)-C(21)-P(2)	123.6(3)	C(23)-C(22)-C(21)	120.9(4)
C(24)-C(23)-C(22)	120.1(4)	C(23)-C(24)-C(25)	120.0(4)
C(24)-C(25)-C(26)	119.4(4)	C(21)-C(26)-C(25)	121.6(4)
C(27)-O(1)-C(30)	108.8(3)	C(27)-O(1)-Li(1)	126.2(3)
C(30)-O(1)-Li(1)	124.8(3)	C(28)-C(27)-O(1)	108.6(4)
C(27)-C(28)-C(29)	109.5(4)	C(28)-C(29)-C(30)	104.5(4)
O(1)-C(30)-C(29)	106.2(3)	C(31)-O(2)-C(34)	107.7(3)
C(31)-O(2)-Li(1)	122.8(3)	C(34)-O(2)-Li(1)	121.3(3)
O(2)-C(31)-C(32)	105.7(4)	C(33)-C(32)-C(31)	103.3(4)
C(34)-C(33)-C(32)	105.4(4)	O(2)-C(34)-C(33)	107.2(4)

Symmetry transformations used to generate equivalent atoms:

#1: -x,-y,-z

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Li(1)	27(3)	39(3)	26(3)	13(3)	9(3)	8(2)
P(1)	16.3(6)	23.2(6)	22.8(6)	10.8(4)	6.0(4)	5.1(4)
P(2)	18.1(6)	24.6(6)	25.0(6)	13.3(4)	4.8(4)	4.9(4)
C(1)	15.2(18)	27.4(17)	24.1(17)	12.7(14)	6.7(14)	5.2(13)
C(2)	14.6(18)	29.0(17)	26.0(18)	13.6(14)	4.7(14)	3.2(13)
C(3)	13.0(17)	27.6(16)	24.4(18)	11.9(14)	3.9(14)	1.0(13)
C(4)	32(2)	39(2)	29.0(19)	14.9(16)	11.3(17)	10.2(16)
C(5)	37(2)	38(2)	35(2)	24(2)	17.4(18)	11.9(19)
C(6)	46(3)	51(2)	34(2)	28(2)	5.9(19)	-4.2(19)
C(7)	58(3)	37(2)	45(2)	26.8(19)	12(2)	11.3(19)
C(8)	41(2)	29.8(18)	33(2)	16.7(16)	13.1(18)	11.4(16)
C(9)	26(2)	28.3(18)	25.2(18)	13.9(15)	6.1(15)	11.0(14)
C(10)	42(2)	28.3(19)	37(2)	8.4(17)	14.7(18)	6.9(16)
C(11)	60(3)	34(2)	34(2)	5.7(18)	16(2)	9.9(19)
C(12)	78(3)	46(2)	32(2)	15.9(19)	28(2)	35(2)
C(13)	51(3)	54(3)	39(2)	25(2)	29(2)	30(2)
C(14)	29(2)	43(2)	32(2)	20.3(17)	14.8(17)	14.1(16)
C(15)	21.2(19)	35.9(18)	25.3(18)	18.2(16)	3.3(15)	0.8(15)
C(16)	29(2)	52(2)	33(2)	26.6(19)	7.7(17)	4.6(17)
C(17)	29(2)	84(3)	39(2)	37(2)	7.3(19)	2(2)
C(18)	43(3)	74(3)	51(3)	48(3)	-2(2)	-19(2)
C(19)	61(3)	48(2)	61(3)	36(2)	18(3)	2(2)
C(20)	53(3)	39(2)	53(3)	30(2)	17(2)	7.0(19)
C(21)	26(2)	22.8(17)	29.6(19)	15.5(15)	8.7(15)	6.7(14)
C(22)	31(2)	27.7(18)	38(2)	13.6(17)	4.5(17)	6.4(15)
C(23)	51(3)	29.0(19)	33(2)	10.5(17)	2.4(19)	-0.8(18)
C(24)	66(3)	31(2)	35(2)	14.2(18)	20(2)	16(2)
C(25)	49(3)	47(2)	50(3)	22(2)	29(2)	25(2)
C(26)	34(2)	37(2)	37(2)	14.6(17)	11.5(18)	8.8(16)
O(1)	18.0(15)	92(2)	40.7(16)	39.7(16)	11.5(12)	15.9(13)
C(27)	25(3)	139(5)	58(3)	67(3)	16(2)	23(3)
C(28)	38(3)	222(8)	123(5)	136(6)	41(3)	50(4)
C(29)	44(3)	49(2)	48(2)	21(2)	28(2)	5.7(19)
C(30)	41(3)	64(3)	38(2)	32(2)	19.2(19)	20(2)
O(2)	39.8(16)	40.5(14)	31.6(14)	17.0(12)	13.0(12)	1.2(11)
C(31)	91(4)	65(3)	49(3)	23(3)	33(3)	-14(3)
C(32)	63(4)	147(6)	66(4)	52(4)	40(3)	36(4)
C(33)	137(6)	62(3)	64(3)	24(3)	58(4)	31(4)
C(34)	53(3)	44(2)	52(3)	18(2)	7(2)	0(2)

Table 5. Hydrogen atom coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for **2**.

	x	y	z	U
H(2A)	-292	-541	702	29
H(2B)	-200	880	1045	29
H(4A)	3357	1137	192	40
H(5A)	3381	1806	-1250	49
H(6A)	2751	3592	-1135	53
H(7A)	1963	4684	373	55
H(8A)	1916	4005	1797	41
H(10A)	3834	4434	3934	46
H(11A)	3481	6001	5529	56
H(12A)	1723	5603	5993	60
H(13A)	308	3662	4817	51
H(14A)	643	2129	3178	39
H(16A)	654	44	3108	45
H(17A)	-488	-920	3832	59
H(18A)	-368	-2927	3512	68
H(19A)	915	-3934	2529	66
H(20A)	2013	-2994	1806	56
H(22A)	387	-2535	-493	43
H(23A)	399	-4270	-2189	52
H(24A)	2324	-4823	-2187	53
H(25A)	4270	-3529	-584	55
H(26A)	4259	-1748	1078	45
H(27A)	7546	2042	4736	79
H(27B)	7259	569	4185	79
H(28A)	8375	385	3158	124
H(28B)	8950	1844	4013	124
H(29A)	7743	789	1606	54
H(29B)	7938	2235	2502	54
H(30A)	5824	1877	1935	52
H(30B)	5681	408	1276	52
H(31A)	4663	884	6083	84
H(31B)	3384	548	4866	84
H(32A)	2848	2334	5788	104
H(32B)	3524	2109	6979	104
H(33A)	4377	4074	6964	101
H(33B)	5348	3461	7666	101
H(34A)	5076	3518	5480	67
H(34B)	6266	3249	6377	67