Crystal structure of tris[(diphenylphosphino)methylene)-diphenylphosphoranylmethyl]yttrium diethyl ether monosolvate, $[Y\{P(C_6H_5)_2CHP(C_6H_5)_2CH_2\}_3] \cdot (C_2H_5)_2O$

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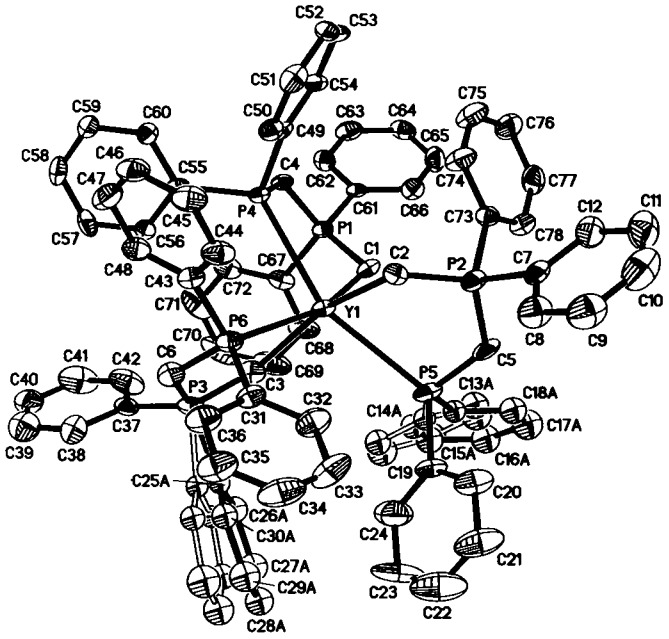

Abstract

$C_{82}H_{79}OP_6Y$, monoclinic, *P*12/*c*1 (no. 13), $a = 24.392(5)$ Å, $b = 12.632(2)$ Å, $c = 23.709(5)$ Å, $\beta = 100.97°$, $V = 7171.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.054$, $wR_{ref}(F^2) = 0.112$, $T = 200$ K.

Source of material

A solution of 1.020 g (2.56 mmol) of the ylide $Ph_2PCH{=}PPh_2Me$ (Ph = C_6H_5, Me = CH_3) in 10 ml of THF was metallated at the Me group by adding 1.6 ml of an 1.6 M solution of LiMe in diethyl ether [1,2]. Adding this solution to a suspension of 0.167 g (0.85 mmol) YCl_3 in 10 ml of THF resulted in a light-yellow solution within a few minutes. The solvent was removed in vacuum and the residue was extracted with 10 ml diethyl ether. From this solution 0.520 g (46 %) light-yellow prismatic crystals could be isolated at 279 K.

Discussion

We investigated the complexation behavior of various α-stabilized phosphorus ylides towards early [3] and late transition metal ions [4]. Now we extend our interest to phosphorus ylides as ligands to form rare earth metal complexes for catalytic applications. The ylide $Ph_2PCH{=}PPh_2Me$ and a nickel complex of the metallated ylide $Ni[CH_2Ph_2PCHPPh_2]_2$ are already described [1,2]. By reaction of the in situ lithiated ligand $Ph_2PCHPPh_2CH_2Li$ with YCl_3 the homoleptic yttrium complex $Y[CH_2Ph_2PCHPPh_2]_3$ was obtained. Its molecular structure was confirmed by the present X-ray diffraction study.

The six-coordinated yttrium atom is surrounded by three chelate ligands. The P—C distances are 1.744(7) Å – 1.751(6) Å for P–CH_2, 1.692(6) Å – 1.713(7) Å and 1.706(7) Å – 1.725(6) Å for P–CH, respectively, comparable with those in $Ni(CH_2Ph_2PCHPPh_2)_2$ [2]. They indicate double bond character and an electron delocalization in the anionic PCPC unit. The bite angle C–Y–P with 73.4(1)° – 75.0(2)° is smaller than in the square planar nickel complex $Ni[CH_2Ph_2PCHPPh_2]_2$ (90.1(2)°). The five membered metallacycles are twisted. The Y—P distances of 2.971(2) Å – 2.983(2) Å are similar to the corresponding bond distances in $YCl[C_5H_4CH_2CH_2PMe_2]_2$ (2.975 Å) [5], but shorter than observed in $Y[OC^tBu_2CH_2PMe_2]_3$ (^{t}Bu = $C(CH_3)_3$, 3.045(2) Å) [6]. Two of the phenyl rings in the molecule are disordered and additional disordered diethyl ether molecules are observed.

Table 1. Data collection and handling.

Crystal:	light-yellow prism, size 0.3 × 0.4 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.90 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, φ
$2\theta_{max}$:	48.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	20771, 10974
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4173
$N(param)_{refined}$:	740
Programs:	SHELXS-97 [7], SHELXL-97 [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(1A)	4*g*		0.1932	0.6922	0.0055	0.047
H(1B)	4*g*		0.2309	0.6509	–0.0381	0.047
H(2A)	4*g*		0.2182	0.5895	0.1619	0.045
H(2B)	4*g*		0.2819	0.5523	0.1789	0.045
H(3A)	4*g*		0.2376	0.3635	–0.0439	0.056
H(3B)	4*g*		0.3008	0.4011	–0.0286	0.056
H(4)	4*g*		0.0598	0.5260	–0.0420	0.039
H(5)	4*g*		0.3545	0.7927	0.1165	0.066
H(6)	4*g*		0.2631	0.1308	0.0768	0.056
H(8)	4*g*		0.3530	0.6744	0.2465	0.091
H(9)	4*g*		0.3898	0.7588	0.3315	0.111
H(10)	4*g*		0.3568	0.9230	0.3511	0.121
H(11)	4*g*		0.2897	1.0096	0.2837	0.109

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Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(12)	4g		0.2508	0.9223	0.1976	0.085
C(13A)	4g	0.741(8)	0.3592(3)	0.7041(6)	0.0018(2)	0.044(2)
C(14A)	4g	0.741	0.3537(3)	0.6466(5)	−0.0490(3)	0.053(3)
H(14A)	4g	0.741	0.3439	0.5738	−0.0495	0.064
C(15A)	4g	0.741	0.3626(3)	0.6957(6)	−0.0990(2)	0.070(4)
H(15A)	4g	0.741	0.3588	0.6564	−0.1337	0.083
C(16A)	4g	0.741	0.3769(3)	0.8023(6)	−0.0982(2)	0.079(4)
H(16A)	4g	0.741	0.3829	0.8358	−0.1323	0.095
C(17A)	4g	0.741	0.3824(3)	0.8597(4)	−0.0474(3)	0.088(4)
H(17A)	4g	0.741	0.3921	0.9326	−0.0469	0.106
C(18A)	4g	0.741	0.3735(3)	0.8107(6)	0.0026(2)	0.064(3)
H(18A)	4g	0.741	0.3772	0.8499	0.0372	0.077
C(13B)	4g	0.259	0.3579(7)	0.650(2)	−0.0049(6)	0.044(2)
C(14B)	4g	0.259	0.3613(7)	0.574(1)	−0.0464(9)	0.060(9)
H(14B)	4g	0.259	0.3632	0.5010	−0.0362	0.072
C(15B)	4g	0.259	0.3618(8)	0.604(2)	−0.1026(8)	0.09(1)
H(15B)	4g	0.259	0.3641	0.5519	−0.1309	0.113
C(16B)	4g	0.259	0.3589(8)	0.711(2)	−0.1175(7)	0.08(1)
H(16B)	4g	0.259	0.3593	0.7313	−0.1559	0.099
C(17B)	4g	0.259	0.3555(8)	0.787(1)	−0.0760(9)	0.088(4)
H(17B)	4g	0.259	0.3536	0.8598	−0.0862	0.106
C(18B)	4g	0.259	0.3550(8)	0.757(2)	−0.0198(8)	0.064(9)
H(18B)	4g	0.259	0.3527	0.8089	0.0085	0.077
H(20)	4g		0.4317	0.6767	0.1669	0.091
H(21)	4g		0.5232	0.6213	0.2049	0.129
H(22)	4g		0.5639	0.4971	0.1557	0.173
H(23)	4g		0.5179	0.4313	0.0692	0.173
H(24)	4g		0.4263	0.4850	0.0322	0.125
C(25A)	4g	0.61(2)	0.3670(2)	0.2510(9)	0.0087(4)	0.037(3)
C(26A)	4g	0.61	0.3862(3)	0.2798(9)	−0.0407(3)	0.062(4)
H(26A)	4g	0.61	0.3604	0.2954	−0.0750	0.074
C(27A)	4g	0.61	0.4432(3)	0.2856(9)	−0.0399(3)	0.068(4)
H(27A)	4g	0.61	0.4564	0.3053	−0.0737	0.081
C(28A)	4g	0.61	0.4811(2)	0.263(1)	0.0102(4)	0.063(4)
H(28A)	4g	0.61	0.5201	0.2667	0.0108	0.076
C(29A)	4g	0.61	0.4619(3)	0.234(1)	0.0596(3)	0.072(4)
H(29A)	4g	0.61	0.4877	0.2184	0.0939	0.086
C(30A)	4g	0.61	0.4048(3)	0.2282(9)	0.0589(3)	0.050(4)
H(30A)	4g	0.61	0.3917	0.2085	0.0926	0.060
C(25B)	4g	0.39	0.3645(4)	0.211(1)	0.0076(5)	0.043(6)
C(26B)	4g	0.39	0.3871(5)	0.236(1)	−0.0403(4)	0.044(6)
H(26B)	4g	0.39	0.3636	0.2621	−0.0741	0.053
C(27B)	4g	0.39	0.4440(5)	0.224(1)	−0.0386(4)	0.055(6)
H(27B)	4g	0.39	0.4594	0.2413	−0.0713	0.066
C(28B)	4g	0.39	0.4783(4)	0.186(2)	0.0109(6)	0.076(7)
H(28B)	4g	0.39	0.5172	0.1779	0.0120	0.091
C(29B)	4g	0.39	0.4557(5)	0.161(2)	0.0588(5)	0.087(8)
H(29B)	4g	0.39	0.4792	0.1352	0.0926	0.105
C(30B)	4g	0.39	0.3988(5)	0.173(1)	0.0571(5)	0.053(6)
H(30B)	4g	0.39	0.3834	0.1559	0.0898	0.064
H(32)	4g		0.3614	0.4335	0.1688	0.080
H(33)	4g		0.4441	0.4172	0.2387	0.110
H(34)	4g		0.4633	0.2572	0.2865	0.109
H(35)	4g		0.4029	0.1168	0.2680	0.094
H(36)	4g		0.3212	0.1350	0.2009	0.076
H(38)	4g		0.2981	0.0156	−0.0009	0.076
H(39)	4g		0.2593	−0.1139	−0.0666	0.097
H(40)	4g		0.2025	−0.0627	−0.1521	0.101
H(41)	4g		0.1831	0.1121	−0.1703	0.117
H(42)	4g		0.2222	0.2415	−0.1065	0.096

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(44)	4g		0.2467	0.3291	0.2369	0.069
H(45)	4g		0.1780	0.2759	0.2864	0.071
H(46)	4g		0.1074	0.1619	0.2441	0.075
H(47)	4g		0.1066	0.0999	0.1515	0.078
H(48)	4g		0.1733	0.1583	0.0998	0.058
H(50)	4g		0.1304	0.3955	0.1518	0.050
H(51)	4g		0.0906	0.4601	0.2282	0.062
H(52)	4g		0.0277	0.5993	0.2120	0.065
H(53)	4g		0.0005	0.6705	0.1213	0.063
H(54)	4g		0.0412	0.6098	0.0455	0.048
H(56)	4g		0.1471	0.2740	−0.0336	0.052
H(57)	4g		0.1049	0.1152	−0.0648	0.068
H(58)	4g		0.0222	0.0636	−0.0346	0.066
H(59)	4g		−0.0128	0.1692	0.0293	0.055
H(60)	4g		0.0301	0.3255	0.0628	0.047
H(62)	4g		0.0432	0.5937	−0.1490	0.051
H(63)	4g		−0.0133	0.7260	−0.1975	0.064
H(64)	4g		0.0123	0.9022	−0.1844	0.069
H(65)	4g		0.0939	0.9460	−0.1205	0.071
H(66)	4g		0.1489	0.8163	−0.0701	0.054
H(68)	4g		0.2356	0.5966	−0.1270	0.073
H(69)	4g		0.2632	0.4902	−0.1968	0.108
H(70)	4g		0.2103	0.3459	−0.2335	0.112
H(71)	4g		0.1299	0.3040	−0.2005	0.098
H(72)	4g		0.1021	0.4076	−0.1290	0.069
H(74)	4g		0.1565	0.7166	0.1458	0.077
H(75)	4g		0.0812	0.8169	0.0995	0.091
H(76)	4g		0.0942	0.9562	0.0407	0.087
H(77)	4g		0.1821	0.9952	0.0241	0.074
H(78)	4g		0.2578	0.8942	0.0687	0.062
O(1)	2e	0.5	0	0.908(1)	¼	0.252(6)
O(2)	2f	0.5	½	0.955(2)	¼	0.311(8)
C(79A)	4g	0.5	0.0372(4)	0.876(1)	0.2136(6)	0.110(6)
H(79A)	4g	0.5	0.0224	0.8990	0.1737	0.132
H(79B)	4g	0.5	0.0406	0.7981	0.2139	0.132
C(80A)	4g	0.5	0.0939(4)	0.925(2)	0.234(1)	0.251(9)
H(80A)	4g	0.5	0.1192	0.9048	0.2086	0.377
H(80B)	4g	0.5	0.1091	0.9003	0.2733	0.377
H(80C)	4g	0.5	0.0904	1.0025	0.2346	0.377
C(79B)	4g	0.5	0.0519(4)	0.858(1)	0.2718(6)	0.121(7)
H(79C)	4g	0.5	0.0457	0.7814	0.2780	0.145
H(79D)	4g	0.5	0.0691	0.8893	0.3092	0.145
C(80B)	4g	0.5	0.0907(5)	0.871(2)	0.230(1)	0.251(9)
H(80D)	4g	0.5	0.1277	0.8434	0.2466	0.377
H(80E)	4g	0.5	0.0935	0.9459	0.2204	0.377
H(80F)	4g	0.5	0.0759	0.8313	0.1944	0.377
C(81A)	4g	0.5	0.4650(9)	0.934(4)	0.1958(4)	0.33(1)
H(81A)	4g	0.5	0.4304	0.9766	0.1919	0.394
H(81B)	4g	0.5	0.4544	0.8583	0.1933	0.394
C(82A)	4g	0.5	0.496(1)	0.962(3)	0.1480(2)	0.27(2)
H(82A)	4g	0.5	0.4710	0.9485	0.1108	0.410
H(82B)	4g	0.5	0.5291	0.9176	0.1513	0.410
H(82C)	4g	0.5	0.5062	1.0364	0.1509	0.410
C(81B)	4g	0.5	0.491(2)	0.900(2)	0.1965(6)	0.33(1)
H(81C)	4g	0.5	0.4715	0.8326	0.2000	0.394
H(81D)	4g	0.5	0.5275	0.8837	0.1858	0.394
C(82B)	4g	0.5	0.457(1)	0.968(3)	0.1505(4)	0.24(2)
H(82D)	4g	0.5	0.4557	0.9353	0.1128	0.358
H(82E)	4g	0.5	0.4733	1.0386	0.1511	0.358
H(82F)	4g	0.5	0.4185	0.9737	0.1577	0.358

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Y(1)	4g	0.24590(2)	0.49686(6)	0.05612(3)	0.0234(3)	0.0484(4)	0.0329(4)	0.0015(4)	0.0068(2)	0.0016(4)
P(1)	4g	0.14288(6)	0.5885(1)	−0.06300(7)	0.0258(9)	0.040(1)	0.026(1)	0.0014(8)	0.0057(8)	0.0032(8)
P(2)	4g	0.27668(7)	0.7217(2)	0.14573(8)	0.038(1)	0.054(1)	0.044(1)	−0.0083(9)	0.0152(9)	0.001(1)
P(3)	4g	0.29107(7)	0.2396(2)	0.00761(7)	0.037(1)	0.062(1)	0.038(1)	0.010(1)	0.0153(9)	0.001(1)
P(4)	4g	0.12381(6)	0.4498(1)	0.03294(7)	0.0239(8)	0.038(1)	0.032(1)	−0.0004(7)	0.0090(8)	−0.0001(8)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(5)	4g	0.34708(6)	0.6320(2)	0.06774(8)	0.027(1)	0.083(2)	0.040(1)	−0.006(1)	0.0095(9)	−0.001(1)
P(6)	4g	0.26909(7)	0.2963(2)	0.12258(8)	0.035(1)	0.057(1)	0.038(1)	0.0030(9)	0.0102(9)	0.004(1)
C(1)	4g	0.2027(2)	0.6289(5)	−0.0154(2)	0.029(3)	0.056(5)	0.031(4)	−0.008(3)	0.004(3)	0.003(3)
C(2)	4g	0.2548(2)	0.5905(4)	0.1495(2)	0.034(4)	0.045(5)	0.031(4)	0.008(3)	0.003(3)	−0.003(3)
C(3)	4g	0.2703(2)	0.3690(5)	−0.0121(3)	0.047(4)	0.055(5)	0.038(5)	0.007(4)	0.006(3)	0.012(4)
C(4)	4g	0.0989(2)	0.5226(4)	−0.0278(2)	0.021(3)	0.041(5)	0.035(4)	0.005(3)	0.003(3)	0.012(3)
C(5)	4g	0.3318(2)	0.7311(5)	0.1113(3)	0.037(4)	0.074(6)	0.062(5)	−0.021(4)	0.030(4)	−0.004(4)
C(6)	4g	0.2720(2)	0.2026(5)	0.0706(2)	0.049(4)	0.054(5)	0.039(5)	−0.001(3)	0.015(3)	0.005(4)
C(7)	4g	0.2996(3)	0.7909(6)	0.2138(3)	0.038(4)	0.056(6)	0.044(5)	−0.014(4)	0.011(4)	0.002(4)
C(8)	4g	0.3400(3)	0.7429(7)	0.2540(4)	0.072(6)	0.101(7)	0.051(7)	−0.012(5)	0.000(5)	−0.021(6)
C(9)	4g	0.3615(4)	0.7922(9)	0.3043(4)	0.080(6)	0.12(1)	0.073(9)	−0.001(7)	0.004(6)	−0.005(7)
C(10)	4g	0.3423(5)	0.890(1)	0.3155(4)	0.107(9)	0.14(1)	0.054(8)	−0.056(8)	0.025(6)	−0.019(8)
C(11)	4g	0.3020(4)	0.9406(8)	0.2760(5)	0.116(8)	0.087(7)	0.080(9)	−0.024(7)	0.046(7)	−0.019(7)
C(12)	4g	0.2795(3)	0.8896(7)	0.2247(3)	0.088(6)	0.074(7)	0.053(7)	−0.016(5)	0.021(5)	−0.013(5)
C(19)	4g	0.4196(2)	0.5860(6)	0.0964(3)	0.028(4)	0.089(6)	0.046(6)	0.005(4)	0.002(4)	0.004(5)
C(20)	4g	0.4489(3)	0.6254(7)	0.1467(4)	0.050(5)	0.119(7)	0.059(7)	0.004(5)	0.011(4)	0.011(5)
C(21)	4g	0.5035(4)	0.5930(9)	0.1697(4)	0.053(6)	0.19(1)	0.071(8)	0.008(7)	−0.004(5)	0.022(7)
C(22)	4g	0.5271(4)	0.521(1)	0.1404(5)	0.075(7)	0.24(2)	0.11(1)	0.062(9)	−0.007(7)	0.05(1)
C(23)	4g	0.4999(4)	0.481(1)	0.0896(5)	0.059(6)	0.23(1)	0.15(1)	0.078(8)	0.041(6)	0.01(1)
C(24)	4g	0.4455(3)	0.5135(8)	0.0675(4)	0.067(6)	0.127(8)	0.125(8)	0.018(6)	0.036(6)	−0.017(7)
C(31)	4g	0.3322(3)	0.2866(6)	0.1793(3)	0.039(4)	0.067(6)	0.032(5)	−0.002(4)	0.009(3)	0.013(4)
C(32)	4g	0.3693(3)	0.3692(6)	0.1896(3)	0.059(5)	0.084(7)	0.057(6)	−0.009(5)	0.008(4)	0.008(5)
C(33)	4g	0.4188(3)	0.3595(8)	0.2306(4)	0.045(5)	0.128(9)	0.093(8)	−0.025(6)	−0.009(5)	0.011(7)
C(34)	4g	0.4298(3)	0.2647(9)	0.2588(4)	0.039(5)	0.16(1)	0.071(7)	0.000(6)	0.001(4)	0.023(7)
C(35)	4g	0.3944(3)	0.1819(7)	0.2482(3)	0.054(6)	0.115(8)	0.065(7)	0.006(5)	0.005(5)	0.034(5)
C(36)	4g	0.3460(3)	0.1934(6)	0.2085(3)	0.043(5)	0.089(7)	0.054(6)	0.006(4)	0.002(4)	0.017(5)
C(37)	4g	0.2642(3)	0.1419(6)	−0.0461(3)	0.043(5)	0.050(5)	0.051(6)	0.008(4)	0.029(4)	−0.001(4)
C(38)	4g	0.2745(3)	0.0364(7)	−0.0358(3)	0.067(5)	0.065(7)	0.059(6)	−0.009(5)	0.015(4)	0.002(5)
C(39)	4g	0.2517(4)	−0.0411(7)	−0.0747(4)	0.088(7)	0.085(8)	0.076(8)	−0.009(6)	0.031(6)	−0.002(6)
C(40)	4g	0.2181(3)	−0.0108(9)	−0.1247(4)	0.069(6)	0.096(9)	0.095(9)	−0.008(6)	0.034(6)	−0.033(7)
C(41)	4g	0.2072(3)	0.092(1)	−0.1356(4)	0.079(6)	0.13(1)	0.071(8)	0.028(7)	−0.009(5)	−0.041(8)
C(42)	4g	0.2299(3)	0.1691(7)	−0.0975(4)	0.081(6)	0.109(8)	0.047(7)	0.038(6)	0.002(5)	−0.020(6)
C(43)	4g	0.2175(2)	0.2482(5)	0.1637(3)	0.034(4)	0.051(5)	0.032(5)	0.002(3)	0.006(3)	0.005(4)
C(44)	4g	0.2179(3)	0.2827(6)	0.2189(3)	0.046(4)	0.091(6)	0.037(5)	0.004(4)	0.013(4)	−0.015(4)
C(45)	4g	0.1771(3)	0.2509(6)	0.2484(3)	0.060(5)	0.081(6)	0.042(5)	0.012(5)	0.024(4)	0.005(4)
C(46)	4g	0.1354(3)	0.1836(6)	0.2237(3)	0.052(5)	0.083(6)	0.061(7)	0.005(5)	0.035(5)	0.009(5)
C(47)	4g	0.1348(3)	0.1480(6)	0.1688(3)	0.053(5)	0.075(6)	0.069(7)	−0.014(4)	0.020(5)	0.003(5)
C(48)	4g	0.1750(3)	0.1814(5)	0.1382(3)	0.046(5)	0.063(5)	0.038(5)	0.005(4)	0.015(4)	−0.002(4)
C(49)	4g	0.0897(2)	0.4965(5)	0.0912(2)	0.022(3)	0.037(4)	0.030(4)	−0.012(4)	0.009(3)	−0.009(4)
C(50)	4g	0.1043(2)	0.4521(5)	0.1454(3)	0.037(4)	0.056(5)	0.032(5)	−0.005(3)	0.009(3)	0.001(4)
C(51)	4g	0.0807(3)	0.4904(6)	0.1910(3)	0.061(4)	0.067(6)	0.029(4)	−0.018(5)	0.012(3)	−0.009(4)
C(52)	4g	0.0431(3)	0.5722(6)	0.1811(3)	0.055(5)	0.055(6)	0.062(6)	−0.012(4)	0.035(4)	−0.027(5)
C(53)	4g	0.0275(2)	0.6154(6)	0.1276(3)	0.034(4)	0.063(5)	0.066(6)	0.001(4)	0.027(4)	−0.013(5)
C(54)	4g	0.0513(2)	0.5786(5)	0.0825(3)	0.028(4)	0.049(5)	0.044(5)	0.000(3)	0.009(3)	−0.004(4)
C(55)	4g	0.0935(2)	0.3190(4)	0.0176(2)	0.019(3)	0.042(4)	0.025(4)	0.004(3)	0.005(3)	0.010(3)
C(56)	4g	0.1143(2)	0.2532(5)	−0.0203(3)	0.043(4)	0.042(5)	0.050(5)	0.000(4)	0.023(4)	−0.009(4)
C(57)	4g	0.0892(3)	0.1589(5)	−0.0394(3)	0.079(6)	0.030(5)	0.059(6)	0.003(4)	0.010(4)	−0.020(4)
C(58)	4g	0.0406(3)	0.1276(5)	−0.0211(3)	0.058(5)	0.035(5)	0.069(6)	−0.016(4)	0.003(4)	0.000(4)
C(59)	4g	0.0201(2)	0.1905(5)	0.0163(3)	0.044(4)	0.041(5)	0.055(6)	−0.010(4)	0.013(4)	−0.004(4)
C(60)	4g	0.0454(2)	0.2840(5)	0.0362(3)	0.038(4)	0.041(5)	0.038(5)	−0.003(3)	0.009(3)	−0.001(4)
C(61)	4g	0.1020(2)	0.6926(5)	−0.1038(2)	0.024(4)	0.036(4)	0.028(4)	0.002(3)	0.012(3)	0.001(3)
C(62)	4g	0.0534(2)	0.6660(5)	−0.1426(3)	0.048(4)	0.036(5)	0.045(5)	0.005(4)	0.010(4)	0.012(4)
C(63)	4g	0.0202(2)	0.7445(7)	−0.1718(3)	0.030(4)	0.077(6)	0.052(5)	0.010(4)	0.003(3)	0.019(5)
C(64)	4g	0.0350(3)	0.8483(6)	−0.1640(3)	0.062(5)	0.048(6)	0.071(6)	0.020(4)	0.031(5)	0.024(5)
C(65)	4g	0.0832(3)	0.8738(6)	−0.1261(3)	0.064(5)	0.041(5)	0.071(6)	−0.006(4)	0.009(4)	0.003(4)
C(66)	4g	0.1160(2)	0.7969(6)	−0.0963(3)	0.040(4)	0.051(5)	0.041(5)	0.001(4)	0.001(3)	0.001(4)
C(67)	4g	0.1665(2)	0.5111(6)	−0.1195(2)	0.040(4)	0.060(5)	0.020(4)	0.015(4)	−0.001(3)	0.008(4)
C(68)	4g	0.2141(3)	0.5362(6)	−0.1411(3)	0.057(5)	0.088(7)	0.046(5)	0.015(4)	0.032(4)	0.011(4)
C(69)	4g	0.2302(4)	0.4735(8)	−0.1830(4)	0.089(7)	0.13(1)	0.069(8)	0.054(7)	0.053(6)	0.037(7)
C(70)	4g	0.1990(5)	0.3882(9)	−0.2046(4)	0.14(1)	0.110(9)	0.037(6)	0.065(8)	0.041(7)	0.014(6)
C(71)	4g	0.1517(4)	0.3633(7)	−0.1851(3)	0.103(7)	0.091(7)	0.043(6)	0.032(6)	−0.008(5)	−0.022(5)
C(72)	4g	0.1353(3)	0.4251(6)	−0.1424(3)	0.055(5)	0.074(6)	0.041(5)	0.017(4)	0.004(4)	−0.012(4)
C(73)	4g	0.2155(2)	0.7979(5)	0.1120(3)	0.037(4)	0.045(5)	0.041(5)	0.000(3)	0.013(3)	0.003(4)
C(74)	4g	0.1621(3)	0.7733(6)	0.1212(3)	0.039(4)	0.076(6)	0.078(6)	0.001(4)	0.010(4)	0.024(5)
C(75)	4g	0.1178(3)	0.8331(7)	0.0938(4)	0.038(5)	0.091(7)	0.098(7)	−0.007(5)	0.015(5)	0.023(6)
C(76)	4g	0.1254(3)	0.9152(6)	0.0586(4)	0.052(5)	0.066(6)	0.092(7)	−0.002(5)	−0.002(5)	0.006(5)
C(77)	4g	0.1768(3)	0.9386(5)	0.0489(3)	0.066(5)	0.047(5)	0.069(6)	−0.007(5)	0.006(5)	0.011(4)
C(78)	4g	0.2216(3)	0.8786(6)	0.0758(3)	0.040(4)	0.060(5)	0.058(6)	−0.008(4)	0.017(4)	0.003(4)

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References

1. Schmidbaur, H.; Deschler, U.: Die Atomsequenz PCPCP als Grundstrukturelement für Phosphoniumsalze, Ylide und deren Alkalikomplexe. Chem. Ber. **116** (1983) 1386-1392.
2. Schmidbaur, H.; Deschler, U.; Milewski-Mahrla, B.: Ein neuer ylidischer Organophosphor-Ligand. Angew. Chem. **93** (1981) 598-599.
3. Spannenberg, A.; Baumann, W.; Rosenthal, U.: Early transition-metal complexes of α-keto-stabilized phosphorus ylides. Appl. Organomet. Chem. **14** (2000) 611-615.
4. Spannenberg, A.; Baumann, W.; Rosenthal, U.: Palladium(II) Complexes of α-Stabilized Phosphorus Ylides. Organometallics **19** (2000) 3991-3993.
5. Karsch, H. H.; Graf, V. W.; Scherer, W.: Phosphane coordination to rare earth metal centers: monomeric, solvent-free complexes of type Cp'_2LnX with phosphanoethyl substituted cyclopentadienyl ligands. J. Organomet. Chem. **604** (2000) 72-82.
6. Hitchcock, P. B.; Lappert, M. F.; MacKinnon, I. A.: Use of a Highly Hindered Phosphino-alkoxide Ligand in the Formation of Monomeric Homoleptic Lanthanoid Metal Complexes: X-Ray Structures of $[Ln(OCBu^t_2CH_2PMe_2)_3]$ (Ln = Y or Nd). J. Chem. Soc., Chem. Commun. (1988) 1557-1558.
7. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
8. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.