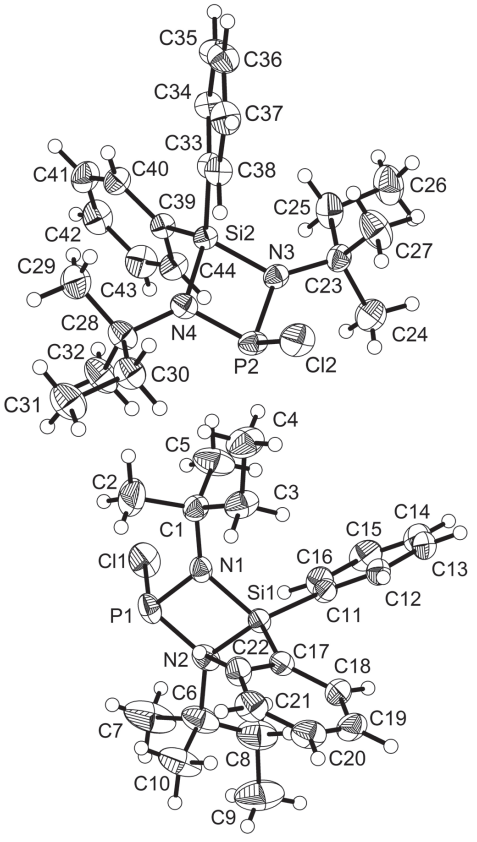
Dennis Mo, Marcel Serio and Walter Frank*

Crystal structure of 2-chloro-1,3-di-*tert*-pentyl-4,4-diphenyl-1,3,2λ^3,4-diazaphosphasiletidine, $C_{22}H_{32}ClN_2PSi$



https://doi.org/10.1515/ncrs-2017-0226
Received August 3, 2017; accepted October 16, 2017; available online November 8, 2017

Abstract

$C_{22}H_{32}ClN_2PSi$, triclinic, $P\bar{1}$ (no. 2), $a = 11.8482(3)$ Å, $b = 12.8044(3)$ Å, $c = 15.6562(4)$ Å, $\alpha = 77.694(2)°$, $\beta = 84.144(2)°$, $\gamma = 89.029(2)°$, $V = 2308.48(10)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0527$, $wR_{ref}(F^2) = 0.1148$, $T = 173(2)$ K.

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CCDC no.: 1580185

The major component of the asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless cube
Size:	0.50 × 0.49 × 0.49 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.0 cm^{-1}
Diffractometer, scan mode:	STOE IPDS, ω scans
$2\theta_{max}$, completeness:	58.6°, 98.8%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	62121, 12461, 0.053
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 11305
$N(param)_{refined}$:	568
Programs:	SHELX [1], DIAMOND [2]

Source of materials

The title compound was prepared according to standard procedures in argon atmosphere in oven-dried glassware using Schlenk-techniques [3–8]. 5.4 g (15.2 mmol) *N*,*N*′-di(ᵗpentyl)-*Si*,*Si*-diphenylsilanediamine were dissolved in 15 mL *n*-pentane and 10 mL diethylether. 19.0 mL of a *n*-butyllithium solution (c = 1.6 mol/L in *n*-hexane, 30.4 mmol) were added at −20 °C. The reaction mixture was stirred for 24 h at room temperature. Cooling to −78 °C and addition of 2.1 g (15.2 mmol) PCl_3 yielded a yellow suspension. The reaction mixture was stirred for 1 h. After filtration and removal of the solvent under reduced pressure the crude product was obtained as viscous oil. The oil was allowed to stand at room temperature. Isometric colourless crystals were obtained within seven days. **^{1}H NMR** (300 MHz, $CDCl_3$, 25 °C): δ (p.p.m.) 0.90 (m, 6H, $C(CH_3)_2)CH_2C\mathbf{H}_3$), 1.09 (s, 6H, $C(C\mathbf{H}_3)_2)CH_2CH_3$), 1.13 (s, 6H, $C(C\mathbf{H}_3)_2)CH_2CH_3$), 1.51 (m, 4H, $C(CH_3)_2)C\mathbf{H}_2CH_3$), 7.48 (m, 6H, *m*-, *p*-C**H**), 7.86 (m, 2H, *o*-C**H**), 8.09 (m, 2H, *o*-C**H**). **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$, 25 °C): δ (p.p.m.) 9.2 (d, $^4J(P,C) = 3.2$ Hz, 2 C, $C(CH_3)_2)CH_2\mathbf{C}H_3$), 28.6 (d, $^3J(P,C) = 7.5$ Hz, 2 C, $C(\mathbf{C}H_3)_2)CH_2CH_3$), 29.2 (d, $^3J(P,C) = 6.6$ Hz, 2 C, $C(\mathbf{C}H_3)_2)CH_2CH_3$), 36.9 (d, $^3J(P,C) = 6.8$ Hz, 2 C, $C(CH_3)_2)\mathbf{C}H_2CH_3$), 55.9 (d, $^2J(P,C) = 6.5$ Hz, 2 C,

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.40851(4)	0.92813(5)	0.73670(4)	0.05338(14)
Cl2	1.08565(4)	0.54349(5)	0.26905(4)	0.05019(13)
P1	0.57620(4)	0.99404(4)	0.68008(4)	0.03739(11)
P2	0.91626(4)	0.58286(4)	0.32422(3)	0.03386(10)
Si1	0.73086(4)	0.87843(4)	0.75278(3)	0.02756(10)
Si2	0.76401(4)	0.50553(4)	0.25180(3)	0.02541(9)
N1	0.65354(12)	0.88669(11)	0.66294(10)	0.0303(3)
N2	0.64615(13)	0.98601(12)	0.76999(11)	0.0365(3)
N3	0.84147(12)	0.46741(12)	0.34184(9)	0.0292(3)
N4	0.84621(12)	0.62118(11)	0.23403(10)	0.0313(3)
C1	0.64194(15)	0.81907(15)	0.59858(13)	0.0357(4)
C2[a]	0.6073(4)	0.8866(3)	0.5143(2)	0.0805(13)
H21[a]	0.6655	0.9412	0.4896	0.121*
H22[a]	0.5348	0.9214	0.5263	0.121*
H23[a]	0.5990	0.8412	0.4722	0.121*
C3[a]	0.7570(2)	0.7663(2)	0.5839(2)	0.0472(7)
H31[a]	0.8145	0.8232	0.5607	0.057*
H32[a]	0.7783	0.7267	0.6415	0.057*
C4[a]	0.7617(3)	0.6895(3)	0.52129(19)	0.0603(8)
H41[a]	0.8387	0.6611	0.5147	0.090*
H42[a]	0.7411	0.7276	0.4638	0.090*
H43[a]	0.7082	0.6304	0.5451	0.090*
C5[a]	0.5530(2)	0.7308(3)	0.6391(3)	0.0607(9)
H51[a]	0.4796	0.7638	0.6514	0.091*
H52[a]	0.5773	0.6884	0.6939	0.091*
H53[a]	0.5458	0.6843	0.5977	0.091*
C2A[b]	0.7310(16)	0.7386(19)	0.608(2)	0.0472(7)
H24[b]	0.7382	0.7055	0.5567	0.071*
H25[b]	0.7114	0.6836	0.6613	0.071*
H26[b]	0.8031	0.7724	0.6124	0.071*
C3A[b]	0.669(3)	0.899(2)	0.5067(12)	0.0805(13)
H33[b]	0.6227	0.9646	0.5013	0.097*
H34[b]	0.7508	0.9183	0.4941	0.097*
C4A[b]	0.632(2)	0.821(2)	0.4483(16)	0.0603(8)
H44[b]	0.6094	0.8636	0.3927	0.090*
H45[b]	0.5674	0.7775	0.4798	0.090*
H46[b]	0.6953	0.7749	0.4361	0.090*
C5A[b]	0.5245(12)	0.7775(13)	0.5994(11)	0.031(4)
H54[b]	0.5255	0.7271	0.5603	0.047*
H55[b]	0.4741	0.8372	0.5793	0.047*
H56[b]	0.4969	0.7410	0.6593	0.047*
C6	0.65451(19)	1.06929(17)	0.82212(17)	0.0525(6)
C7[c]	0.5349(6)	1.1237(7)	0.8256(7)	0.088(3)
H71[c]	0.5181	1.1567	0.7659	0.131*
H72[c]	0.5350	1.1787	0.8606	0.131*
H73[c]	0.4770	1.0697	0.8527	0.131*
C8[c]	0.6775(6)	1.0153(6)	0.9109(5)	0.0652(14)
H81[c]	0.7502	0.9766	0.9067	0.078*
H82[c]	0.6172	0.9613	0.9347	0.078*
C9[c]	0.6842(5)	1.0870(4)	0.9758(4)	0.0891(17)
H91[c]	0.7084	1.0450	1.0308	0.134*
H92[c]	0.6093	1.1178	0.9875	0.134*
H93[c]	0.7390	1.1447	0.9510	0.134*
C7A[d]	0.7191(11)	1.0067(12)	0.9071(9)	0.061(3)
H74[d]	0.7920	0.9789	0.8862	0.092*
H75[d]	0.7322	1.0567	0.9446	0.092*
H76[d]	0.6715	0.9473	0.9410	0.092*
C8A[d]	0.5455(16)	1.113(2)	0.8486(16)	0.0891(17)
H83[d]	0.4931	1.0522	0.8746	0.107*
H84[d]	0.5141	1.1536	0.7953	0.107*
C9A[d]	0.5474(7)	1.1859(7)	0.9151(6)	0.0652(14)
H94[d]	0.4712	1.2143	0.9261	0.098*
H95[d]	0.6005	1.2453	0.8911	0.098*
H96[d]	0.5717	1.1447	0.9704	0.098*
C10	0.7413(2)	1.1540(2)	0.7740(2)	0.0670(8)
H101	0.8162	1.1210	0.7683	0.101*
H102	0.7447	1.2103	0.8074	0.101*
H103	0.7188	1.1851	0.7155	0.101*
C11	0.70864(15)	0.75330(14)	0.83810(11)	0.0310(3)
C12	0.78538(17)	0.66858(14)	0.84524(12)	0.0361(4)
H121	0.8549	0.6765	0.8084	0.043*
C13	0.7618(2)	0.57305(16)	0.90532(14)	0.0444(5)
H131	0.8146	0.5160	0.9090	0.053*
C14	0.6618(2)	0.56101(17)	0.95957(14)	0.0498(5)
H141	0.6463	0.4960	1.0013	0.060*
C15	0.5841(2)	0.64305(19)	0.95348(14)	0.0497(5)
H151	0.5150	0.6343	0.9908	0.060*
C16	0.60672(17)	0.73838(16)	0.89280(13)	0.0400(4)
H161	0.5523	0.7941	0.8884	0.048*
C17	0.88367(14)	0.91430(13)	0.72332(12)	0.0300(3)
C18	0.96758(15)	0.88290(15)	0.77996(13)	0.0363(4)
H181	0.9480	0.8375	0.8358	0.044*
C19	1.07943(16)	0.91710(17)	0.75594(15)	0.0450(5)
H191	1.1355	0.8945	0.7951	0.054*
C20	1.10913(17)	0.98360(18)	0.67561(16)	0.0472(5)
H201	1.1856	1.0067	0.6594	0.057*
C21	1.02780(17)	1.01677(17)	0.61865(15)	0.0459(5)
H211	1.0482	1.0630	0.5633	0.055*
C22	0.91616(16)	0.98254(15)	0.64218(13)	0.0379(4)
H221	0.8608	1.0058	0.6025	0.045*
C23	0.85553(15)	0.36590(15)	0.40588(12)	0.0337(4)
C24[e]	0.8852(5)	0.3928(3)	0.4910(3)	0.0689(11)
H241[e]	0.9562	0.4338	0.4799	0.103*
H242[e]	0.8942	0.3266	0.5345	0.103*
H243[e]	0.8241	0.4354	0.5135	0.103*
C25[e]	0.7410(3)	0.3080(4)	0.4219(3)	0.0463(9)
H251[e]	0.6832	0.3543	0.4452	0.056*
H252[e]	0.7190	0.2981	0.3648	0.056*
C26[e]	0.7384(3)	0.1996(2)	0.4850(2)	0.0608(8)
H261[e]	0.6615	0.1697	0.4932	0.091*
H262[e]	0.7607	0.2081	0.5417	0.091*
H263[e]	0.7913	0.1512	0.4606	0.091*
C24A[f]	0.752(3)	0.299(4)	0.405(3)	0.0463(9)
H244[f]	0.7600	0.2685	0.3524	0.069*
H245[f]	0.6843	0.3439	0.4046	0.069*
H246[f]	0.7450	0.2411	0.4575	0.069*
C25A[f]	0.903(4)	0.376(2)	0.4914(16)	0.0689(11)
H253[f]	0.9865	0.3864	0.4801	0.083*
H254[f]	0.8707	0.4407	0.5100	0.083*
C26A[f]	0.8770(19)	0.2790(17)	0.5656(11)	0.0608(8)
H264[f]	0.9305	0.2763	0.6098	0.091*
H265[f]	0.8843	0.2140	0.5419	0.091*
H266[f]	0.7994	0.2841	0.5927	0.091*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C27	0.9473(2)	0.29849(18)	0.36811(18)	0.0572(6)
H271	1.0198	0.3372	0.3575	0.086*
H272	0.9263	0.2845	0.3126	0.086*
H273	0.9548	0.2305	0.4100	0.086*
C28	0.83491(16)	0.73172(14)	0.18230(13)	0.0371(4)
C29[g]	0.7766(5)	0.7243(8)	0.1006(3)	0.0551(13)
H291[g]	0.7657	0.7964	0.0659	0.083*
H292[g]	0.7028	0.6888	0.1189	0.083*
H293[g]	0.8241	0.6830	0.0650	0.083*
C30[g]	0.9534(2)	0.7787(2)	0.1507(2)	0.0431(6)
H301[g]	0.9974	0.7278	0.1212	0.052*
H302[g]	0.9916	0.7845	0.2029	0.052*
C31[g]	0.9584(3)	0.8882(2)	0.0879(2)	0.0588(8)
H311[g]	1.0369	0.9143	0.0768	0.088*
H312[g]	0.9107	0.9387	0.1143	0.088*
H313[g]	0.9307	0.8819	0.0323	0.088*
C29A[h]	0.9579(13)	0.7806(15)	0.1894(14)	0.0431(6)
H294[h]	1.0176	0.7361	0.1678	0.065*
H295[h]	0.9659	0.7817	0.2509	0.065*
H296[h]	0.9646	0.8536	0.1539	0.065*
C30A[h]	0.758(4)	0.720(6)	0.113(3)	0.0551(13)
H303[h]	0.6768	0.7206	0.1347	0.066*
H304[h]	0.7762	0.6566	0.0880	0.066*
C31A[h]	0.800(2)	0.8317(16)	0.0459(13)	0.074(7)
H314[h]	0.7888	0.8270	−0.0144	0.112*
H315[h]	0.8804	0.8438	0.0499	0.112*
H316[h]	0.7552	0.8912	0.0617	0.112*
C32	0.7643(2)	0.79763(17)	0.23868(17)	0.0526(6)
H321	0.8031	0.8009	0.2904	0.079*
H322	0.6896	0.7641	0.2575	0.079*
H323	0.7550	0.8701	0.2042	0.079*
C33	0.78979(14)	0.42232(13)	0.16785(10)	0.0271(3)
C34	0.71330(15)	0.34506(14)	0.15749(11)	0.0324(3)
H341	0.6421	0.3363	0.1922	0.039*
C35	0.74005(17)	0.28107(16)	0.09721(13)	0.0397(4)
H351	0.6871	0.2292	0.0905	0.048*
C36	0.84394(18)	0.29270(16)	0.04674(13)	0.0418(4)
H361	0.8618	0.2494	0.0049	0.050*
C37	0.92155(17)	0.36705(16)	0.05714(13)	0.0403(4)
H371	0.9933	0.3741	0.0232	0.048*
C38	0.89504(15)	0.43145(15)	0.11700(11)	0.0333(4)
H381	0.9489	0.4825	0.1237	0.040*
C39	0.61046(13)	0.52868(13)	0.27989(11)	0.0275(3)
C40	0.52760(15)	0.52649(15)	0.22260(12)	0.0346(4)
H401	0.5487	0.5086	0.1673	0.041*
C41	0.41503(16)	0.55002(17)	0.24537(14)	0.0430(4)
H411	0.3599	0.5474	0.2059	0.052*
C42	0.38312(16)	0.57701(17)	0.32476(15)	0.0453(5)
H421	0.3061	0.5932	0.3401	0.054*
C43	0.46351(17)	0.58050(18)	0.38220(14)	0.0447(5)
H431	0.4417	0.5993	0.4371	0.054*
C44	0.57602(15)	0.55667(15)	0.35993(12)	0.0358(4)
H441	0.6305	0.5595	0.3999	0.043*

[a]Occupancy: 0.892(4); [b]Occupancy: 0.108(4); [c]Occupancy: 0.665(5); [d]Occupancy: 0.335(5); [e]Occupancy: 0.883(4); [f]Occupancy: 0.117(4); [g]Occupancy: 0.863(5); [h]Occupancy: 0.137(5).

C$(CH_3)_2)CH_2CH_3$), 128.3–136.3 (12 C, Ar-**C**) **$^{31}P\{^1H\}$ NMR** (121 MHz, $CDCl_3$, 25 °C): δ (p.p.m.) 216.2 (*s*). EI–MS spectra were obtained using a Finnigan TSQ 7000 instrument. **EI–MS**: m/*z* (%) 418(1) [M^+], 389(100) [M–C_2H_5]. IR spectroscopic data were measured using a Bio-Rad Excalibur FTS 3500 FT–IR spectrometer with ATR-unit. **IR** 4000-560 cm^{-1}: 3069(*w*), 3049(*w*), 2966(*vs*), 2927(*m*), 2878(*m*), 1966(*vw*), 1904(*vw*), 1872(*vw*), 1777(*vw*), 1589(*m*), 1461(*m*), 1427(*s*), 1171(*s*), 1115(*vs*), 1046(*s*), 979(*w*), 922(*m*), 880(*vs*), 814(*w*), 782(*vw*), 738(*s*), 715(*m*), 697(*s*), 632(*w*), 562(*w*).

Experimental details

In the refinement the riding model was applied using idealized C—H bond lengths as well as H—C—H, C—C—H angles. In addition, the H atoms of the CH_3 groups were allowed to rotate around the neighbouring C—C bonds. The U_{iso} values were set to $1.5U_{eq}(C_{methyl})$ and $1.2U_{eq}(C_{ar}, C_{methylene})$, respectively. To account for residual electron density in the regions of all the *tert*-pentyl groups and for elongated anisotropic displacement ellipsoids of several carbon-atoms that did not appear to be physically meaningful, a two-position disorder for each *tert*-pentyl group was introduced with partial occupation sites for all carbon atoms but the tertiary ones C1, C6, C23 and C28 and the primary ones C10, C27 and C32. (Disorder ratio 0.892(4)/0.108(4) (group containing C1), 0.665(5)/0.335(5) ratio (C6), 0.883(4)/0.117(4) ratio (C23) and 0.863(5)/0.137(5) ratio (C28); disorder is omitted in the figure for clarity). Appropriate same distance and anisotropic displacement restraints and some equivalent anisotropic displacement parameters had to be applied to stabilize the geometry of the minor occupied parts of the partial occupation site models.

Comment

Diazaphosphasiletidines are heterocyclic compounds that contain a SiN_2P four-membered ring as central building block. The first synthesis was described in the year 1963 and the compounds of the class have attracted considerable attention in phosphorus chemistry [3–8]. Crystals of the first structurally characterised chlorosubstituted diazaphosphasiletidine, 2-chloro-1,3-bis(2,4,6-trimethylphenyl)-4,4-dimethyl-1,3,2λ^3,4-diazaphosphasiletidine [7] contained approximately 12% of a second compound, namely 2-chloro-1,3-bis(2,4,6-trimethylphenyl)-4-chloro-4-methyl-1,3,2λ^3,4-diazaphosphasiletidine. With respect to this impurity a *Si*,*Si*-diphenylsubstituted diazaphosphasiletidine, the title compound, has been introduced to preparative chemistry to avoid problems related to the content of *Si*,*P*-bis(chloro)functionalized species always present in samples of the *Si*,*Si*-dimethylderivative.

The asymmetric unit of the crystal structure of the phenyl derivative contains two crystallographically independent

molecules, which do not differ significantly in bond lengths and angles. The central moiety of the title compound, the SiN_2P four-membered ring, is almost planar. The nitrogen atoms exhibit a trigonal planar coordination sphere (sums of bond angles 359.52(11)° (N1) and 356.88(13)° (N2) (molecule no. 1), 359.39(11)° (N3) and 356.52(11)° (N4) (molecule no. 2). Phosphorus and silicon atoms bear the main ring strain (N1—Si1—N2 82.89(8)°; N3—Si2—N4 82.76(7)° and N1—P1—N2 85.90(7)°; N3—P2—N4 85.75(7)°). The Si—N bond lengths (Si1—N1 1.7392(15) Å, Si1—N2 1.7418(16) Å; Si2—N3 1.7387(15) Å, Si2—N4 1.7427(15) Å) exceed the expected lenght of a Si—N single bond (1.724(4) Å [9]) but correspond to those in related cyclosilazanes [7, 10–13]. In contrast, the P—N distances are shorter (P1—N1 1.6928(15) Å, P1—N2 1.6889(18) Å; P2—N3 1.6932(15) Å, P2—N4 1.6891(16) Å) than reported for a typical single bond (1.704(4) Å [9]), but they also correspond to those in the first structurally characterized chlorosubstituted diazaphosphasiletidine [7]. The P—Cl bonds of the title compound are remarkably elongated (P1—Cl1 2.1963(7) Å, P2—Cl2 2.1967(7) Å) compared to the P—Cl distance in PCl_3 (2.034 Å [14]) and exceed the sum of the covalence radii [15]. A comparison of the average Si—N, P—N and P—Cl distances in the title compound and the analogous distances of the formerly published dimethylsilanederivative [7] does not give evidence for substitution effects [16] and only shows a small difference (numerically significant but chemically not relevant) in the case of the P—Cl bond: Si—N 1.7406(15) Å average (in the title compound) *vs.* 1.7441(17) Å average (in the mentioned dimethylderivative); P—N 1.6910(16) Å average *vs.* 1.6856(17) Å average; P—Cl 2.1965(7) Å average *vs.* 2.1813(7) Å). The *tert*-pentyl groups in the solid of the title compound are disordered to give two positions of the ethyl moiety in each case (Exptl. det.). Comparing the solid state packings of the title compound and of related higher congeners of group 15 the increasing tendency to association of the molecules *via* El—Cl bridging bonds from P to Bi becomes apparent. Whereas the title compound consists of isolated molecules, $Me_2Si(N^tBu)_2AsCl$ contains dimers and in the antimony and the bismuth compound the molecules are connected to chains *via* bridging Cl atoms [17, 18].

Acknowledgements: We thank E. Hammes and P. Roloff for technical support.

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