Crystal structure of tris(*tert*-butyl-dimethylsilyl)phosphine, $((C_4H_9)(CH_3)_2Si)_3P$

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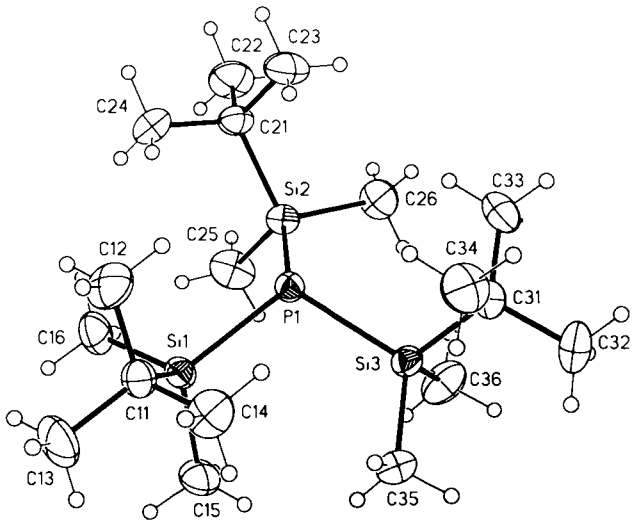

Source of material: Single crystals of the title compound (see ref. 2) were obtained by crystallization from diethylether (see ref. 6). Compared to the parent system $(H_3Si)_3P$ (see ref. 1) and the tris(trimethylsilyl)phosphine (see ref. 2) the Si–P–Si angles are widened (96° vs. 106° vs. 110°). Due to the bulky substituents the phosphorus atom is less pyramidalized. The same effect is observed in the structures of trialkylphosphines (see refs. 3-5).

$C_{18}H_{45}PSi_3$, monoclinic, $P12_1/n1$ (No. 14), a =10.979(1) Å, b =17.078(3) Å, c =13.797(2) Å, β =110.42(1)°, V =2424.4 Å^3, Z =4, $R(F)$ =0.042, $R_w(F^2$, all data) =0.117.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless plate, size 0.08 x 0.20 x 0.40 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	23.82 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	2θ/ω
$T_{measurement}$:	193 K
$2\theta_{max}$:	112°
N(hkl)$_{unique}$:	3143
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
N($param$)$_{refined}$:	199
Programs:	SHELXS-86, SHELXL-93

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

Atom	Site	x	y	z	U_{iso}
H(12A)	4e	0.1256(4)	0.2368(2)	0.7148(2)	0.076
H(12B)	4e	0.2616(4)	0.2127(2)	0.7046(2)	0.076
H(12C)	4e	0.2019(4)	0.1611(2)	0.7739(2)	0.076
H(13A)	4e	–0.0601(4)	0.0754(2)	0.5707(3)	0.087
H(13B)	4e	–0.0696(4)	0.1507(2)	0.6366(3)	0.087
H(13C)	4e	0.0120(4)	0.0759(2)	0.6934(3)	0.087
H(14A)	4e	0.1552(3)	0.0405(2)	0.5535(3)	0.071
H(14B)	4e	0.2197(3)	0.0430(2)	0.6770(3)	0.071
H(14C)	4e	0.2794(3)	0.0947(2)	0.6076(3)	0.071
H(15A)	4e	0.0052(3)	0.1023(2)	0.3671(2)	0.068
H(15B)	4e	–0.0762(3)	0.1795(2)	0.3195(2)	0.068
H(15C)	4e	–0.1197(3)	0.1247(2)	0.3957(2)	0.068
H(16A)	4e	0.0044(3)	0.3166(2)	0.5706(3)	0.068
H(16B)	4e	–0.1201(3)	0.2611(2)	0.5253(3)	0.068
H(16C)	4e	–0.0765(3)	0.3159(2)	0.4491(3)	0.068
H(22A)	4e	0.2968(4)	0.5386(2)	0.4476(3)	0.087
H(22B)	4e	0.2855(4)	0.5643(2)	0.5557(3)	0.087
H(22C)	4e	0.1581(4)	0.5389(2)	0.4616(3)	0.087
H(23A)	4e	0.4666(3)	0.4381(2)	0.5463(3)	0.071
H(23B)	4e	0.4318(3)	0.3765(2)	0.6201(3)	0.071
H(23C)	4e	0.4483(3)	0.4673(2)	0.6505(3)	0.071
H(24A)	4e	0.1064(3)	0.4381(2)	0.5736(3)	0.079
H(24B)	4e	0.2315(3)	0.4673(2)	0.6670(3)	0.079
H(24C)	4e	0.2151(3)	0.3765(2)	0.6365(3)	0.079
H(25A)	4e	–0.0151(3)	0.3888(2)	0.3728(3)	0.076
H(25B)	4e	0.0176(3)	0.3668(2)	0.2718(3)	0.076
H(25C)	4e	0.0382(3)	0.4546(2)	0.3151(3)	0.076
H(26A)	4e	0.4078(3)	0.3845(2)	0.3770(3)	0.068
H(26B)	4e	0.3080(3)	0.4518(2)	0.3179(3)	0.068
H(26C)	4e	0.2875(3)	0.3640(2)	0.2746(3)	0.068
H(32A)	4e	0.5172(4)	0.1382(2)	0.2893(3)	0.096
H(32B)	4e	0.5056(4)	0.0615(2)	0.3521(3)	0.096
H(32C)	4e	0.6381(4)	0.1106(2)	0.3874(3)	0.096
H(33A)	4e	0.5341(3)	0.2712(2)	0.3737(3)	0.078
H(33B)	4e	0.6538(3)	0.2388(2)	0.4692(3)	0.078
H(33C)	4e	0.5317(3)	0.2776(2)	0.4888(3)	0.078
H(34A)	4e	0.4938(3)	0.0743(2)	0.5302(3)	0.080
H(34B)	4e	0.5073(3)	0.1587(2)	0.5831(3)	0.080
H(34C)	4e	0.6294(3)	0.1200(2)	0.5635(3)	0.080
H(35A)	4e	0.2677(3)	0.0491(2)	0.4351(3)	0.066
H(35B)	4e	0.2807(3)	0.0378(2)	0.3239(3)	0.066
H(35C)	4e	0.1490(3)	0.0717(2)	0.3326(3)	0.066
H(36A)	4e	0.2886(4)	0.2602(2)	0.2323(2)	0.078
H(36B)	4e	0.1622(4)	0.2059(2)	0.2036(2)	0.078
H(36C)	4e	0.2939(4)	0.1720(2)	0.1949(2)	0.078

Table 3. Final 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}
P(1)	4*e*	0.24613(6)	0.25031(4)	0.47934(5)	0.0262(4)	0.0238(4)	0.0283(4)	–0.0007(3)	0.0119(3)	0.0001(3)
Si(1)	4*e*	0.06020(7)	0.20372(4)	0.49400(6)	0.0243(4)	0.0318(4)	0.0277(4)	–0.0028(3)	0.0092(3)	–0.0004(3)
C(11)	4*e*	0.1092(3)	0.1421(2)	0.6165(2)	0.040(2)	0.037(2)	0.029(2)	–0.006(1)	0.017(1)	0.002(1)
C(12)	4*e*	0.1811(4)	0.1927(2)	0.7110(2)	0.066(2)	0.058(2)	0.028(2)	–0.004(2)	0.016(2)	–0.001(2)
C(13)	4*e*	–0.0132(4)	0.1079(2)	0.6306(3)	0.062(2)	0.065(2)	0.060(2)	–0.014(2)	0.038(2)	0.013(2)
C(14)	4*e*	0.1990(3)	0.0739(2)	0.6134(3)	0.064(2)	0.040(2)	0.038(2)	0.005(2)	0.020(2)	0.011(2)
C(15)	4*e*	–0.0452(3)	0.1456(2)	0.3806(2)	0.035(2)	0.058(2)	0.038(2)	–0.012(2)	0.006(2)	–0.008(2)
C(16)	4*e*	–0.0456(3)	0.2838(2)	0.5118(3)	0.033(2)	0.049(2)	0.059(2)	0.005(2)	0.022(2)	0.003(2)
Si(2)	4*e*	0.21453(7)	0.37455(4)	0.41774(6)	0.0295(4)	0.0265(4)	0.0273(4)	0.0001(3)	0.0068(3)	0.0027(3)
C(21)	4*e*	0.2721(3)	0.4432(2)	0.5336(2)	0.035(2)	0.027(2)	0.039(2)	–0.003(1)	0.010(1)	–0.005(1)
C(22)	4*e*	0.2512(4)	0.5291(2)	0.4962(3)	0.062(2)	0.026(2)	0.084(3)	0.000(2)	0.022(2)	–0.007(2)
C(23)	4*e*	0.4180(3)	0.4301(2)	0.5930(3)	0.039(2)	0.046(2)	0.047(2)	–0.008(2)	0.003(2)	–0.008(2)
C(24)	4*e*	0.1997(3)	0.4301(2)	0.6096(3)	0.058(2)	0.055(2)	0.049(2)	–0.010(2)	0.025(2)	–0.024(2)
C(25)	4*e*	0.0434(3)	0.3991(2)	0.3344(3)	0.043(2)	0.048(2)	0.048(2)	0.004(2)	–0.002(2)	0.015(2)
C(26)	4*e*	0.3166(3)	0.3963(2)	0.3372(3)	0.057(2)	0.040(2)	0.043(2)	–0.003(2)	0.023(2)	0.010(2)
Si(3)	4*e*	0.30905(7)	0.17250(4)	0.37184(6)	0.0286(4)	0.0299(4)	0.0244(4)	–0.0004(3)	0.0084(3)	–0.0033(3)
C(31)	4*e*	0.4933(3)	0.1646(2)	0.4291(2)	0.031(2)	0.037(2)	0.043(2)	0.003(1)	0.016(1)	–0.004(1)
C(32)	4*e*	0.5431(4)	0.1141(2)	0.3580(3)	0.053(2)	0.073(3)	0.079(3)	0.009(2)	0.039(2)	–0.018(2)
C(33)	4*e*	0.5593(3)	0.2454(2)	0.4413(3)	0.030(2)	0.057(2)	0.069(2)	–0.007(2)	0.019(2)	–0.007(2)
C(34)	4*e*	0.5347(3)	0.1259(2)	0.5362(3)	0.039(2)	0.057(2)	0.053(2)	0.012(2)	0.003(2)	0.006(2)
C(35)	4*e*	0.2438(3)	0.0707(2)	0.3651(3)	0.050(2)	0.035(2)	0.049(2)	–0.007(2)	0.020(2)	–0.013(2)
C(36)	4*e*	0.2572(4)	0.2067(2)	0.2342(2)	0.074(2)	0.055(2)	0.024(2)	0.007(2)	0.014(2)	–0.002(2)

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