Crystal structure of trimethylsilyl-triphenylphosphane-iminium-dichloroiodate(I), $(C_6H_5)_3PNHSi(CH_3)_3ICl_2$

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Source of material: The compound is produced by reaction of $Me_3SiNPPH_3$ with ICl in the presence HCl in dichloro methane solution and subsequent addition of *n*-hexane at 293 K.

Average bond lengths in the two symmetry independent cations are: $d(P-N) = 162.2(4)$ pm; $d(Si-N) = 176.5(4)$ pm; $d(N-H) = 72(5)$ pm; $d(H(1) \cdots Cl(1)) = 267(5)$ pm. Cation bond angles are (in average): $(Si-N-P) = 132.6(3)^\circ$; $(N-H \cdots Cl) = 160(5)^\circ$. The anions lie in general positions with $d(I(1)-Cl) = 251.5(2)$ pm and $256.7(2)$ pm and are disordered around centres of symmetry with $d(I(2)-Cl) = 252(1)$ pm and $256(1)$ pm and $d(I(3)-Cl) = 247(1)$ pm and $252.2(4)$ pm. The bond angle of anion 1 ($Cl(1)-I(1)-Cl(2)$) is $178.17(5)^\circ$, whereas the other species are linear. Details of the bonding in the ICl_2^- -ions are comparable with other compounds with the dichloroiodate(I)-ion (see ref. 1).

The observed bond angle $SiNP$ is somewhat different from that in $[Me_3SiN(H)PMe_3]_2[Se_2Cl_6]$ ($150.6(6)^\circ$, see ref. 2).

$C_{21}H_{25}Cl_2INPSi$, triclinic, $P\bar{1}$ (No. 2), $a = 10.836(1)$ Å, $b = 14.035(1)$ Å, $c = 16.324(1)$ Å, $\alpha = 88.062(4)^\circ$, $\beta = 84.880(5)^\circ$, $\gamma = 79.806(5)^\circ$, $V = 2433.2$ Å³, $Z = 4$, $R(F) = 0.034$, $R_w(F^2) = 0.072$.

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

Crystal:	orange prism, size 0.12 x 0.17 x 0.38 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	16.57 cm ⁻¹
Diffractometer:	Enraf Nonius CAD4
Scan mode:	ω
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	49°
$N(hkl)_{\text{unique}}$:	8240
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	476
Program:	SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(1)	2i	0.023(5)	0.482(3)	0.198(3)	0.06(2)
H(2)	2i	0.667(4)	0.047(3)	0.086(2)	0.03(1)
H(12)	2i	-0.3237(4)	0.6612(3)	0.3033(2)	0.087(7)
H(13)	2i	-0.3742(4)	0.7818(3)	0.4037(3)	0.087(7)
H(14)	2i	-0.2554(5)	0.7739(3)	0.5184(2)	0.087(7)
H(15)	2i	-0.0861(5)	0.6455(4)	0.5329(2)	0.087(7)
H(16)	2i	-0.0357(4)	0.5249(3)	0.4326(3)	0.087(7)
H(22)	2i	-0.2311(3)	0.4005(3)	0.4220(3)	0.083(7)
H(23)	2i	-0.1847(4)	0.2449(3)	0.4824(2)	0.083(7)
H(24)	2i	-0.0180(5)	0.1300(2)	0.4236(3)	0.083(7)
H(25)	2i	0.1021(4)	0.1707(3)	0.3043(3)	0.083(7)
H(26)	2i	0.0557(4)	0.3262(3)	0.2439(2)	0.083(7)
H(32)	2i	-0.1584(3)	0.5361(3)	0.1100(2)	0.071(6)
H(33)	2i	-0.3260(4)	0.5425(3)	0.0264(1)	0.071(6)
H(34)	2i	-0.5212(3)	0.5087(3)	0.0836(2)	0.071(6)
H(35)	2i	-0.5490(3)	0.4685(4)	0.2244(3)	0.071(6)
H(36)	2i	-0.3814(4)	0.4622(3)	0.3081(1)	0.071(6)
H(411)	2i	-0.028(7)	0.785(3)	0.173(4)	0.21(2)
H(412)	2i	-0.136(4)	0.734(5)	0.216(3)	0.21(2)
H(413)	2i	-0.090(7)	0.715(6)	0.123(2)	0.21(2)
H(421)	2i	0.232(5)	0.646(3)	0.134(4)	0.15(2)
H(422)	2i	0.160(6)	0.596(4)	0.073(2)	0.15(2)
H(423)	2i	0.240(5)	0.533(2)	0.138(4)	0.15(2)
H(431)	2i	0.181(5)	0.706(4)	0.294(5)	0.20(2)
H(432)	2i	0.161(6)	0.598(2)	0.305(5)	0.20(2)
H(433)	2i	0.069(6)	0.681(5)	0.354(3)	0.20(2)
H(52)	2i	0.7015(3)	-0.1359(3)	0.1158(2)	0.075(7)
H(53)	2i	0.6018(4)	-0.2724(2)	0.1239(2)	0.075(7)
H(54)	2i	0.4328(4)	-0.2806(2)	0.2236(3)	0.075(7)
H(55)	2i	0.3635(3)	-0.1523(3)	0.3152(2)	0.075(7)
H(56)	2i	0.4633(4)	-0.0158(2)	0.3071(2)	0.075(7)
H(62)	2i	0.9343(4)	0.0306(3)	0.1487(2)	0.088(8)
H(63)	2i	1.1260(3)	-0.0202(3)	0.2080(3)	0.088(8)
H(64)	2i	1.1260(3)	-0.0839(3)	0.3433(3)	0.088(8)
H(65)	2i	0.9343(4)	-0.0969(3)	0.4194(2)	0.088(8)
H(66)	2i	0.7426(3)	-0.0461(3)	0.3601(2)	0.088(8)
H(72)	2i	0.7015(3)	0.1402(3)	0.3528(2)	0.078(7)
H(73)	2i	0.5752(4)	0.2727(3)	0.4212(2)	0.078(7)
H(74)	2i	0.3822(4)	0.3443(3)	0.3717(3)	0.078(7)
H(75)	2i	0.3153(3)	0.2835(3)	0.2536(3)	0.078(7)
H(76)	2i	0.4416(4)	0.1510(3)	0.1851(2)	0.078(7)
H(811)	2i	0.830(3)	0.297(2)	0.114(2)	0.10(1)
H(812)	2i	0.854(3)	0.206(3)	0.173(2)	0.10(1)
H(813)	2i	0.722(3)	0.274(3)	0.177(2)	0.10(1)
H(821)	2i	0.611(5)	0.300(2)	-0.013(3)	0.14(2)
H(822)	2i	0.532(4)	0.261(3)	0.062(2)	0.14(2)
H(823)	2i	0.572(5)	0.198(3)	-0.017(2)	0.14(2)
H(831)	2i	0.904(5)	0.189(3)	-0.041(4)	0.17(2)
H(832)	2i	0.826(6)	0.110(4)	-0.060(3)	0.17(2)
H(833)	2i	0.930(4)	0.087(3)	0.002(4)	0.17(2)

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
I(1)	2i		0.20551(3)	0.20853(3)	0.06754(2)	0.0568(2)	0.0770(2)	0.0487(2)	-0.0118(2)	0.0010(1)	-0.0184(2)
Cl(1)	2i		0.0715(2)	0.3784(1)	0.06141(9)	0.100(1)	0.080(1)	0.079(1)	0.0063(9)	0.0222(8)	-0.0117(8)
Cl(2)	2i		0.3403(2)	0.0431(1)	0.0692(1)	0.105(1)	0.082(1)	0.085(1)	0.0073(9)	-0.0321(9)	-0.0273(8)
I(2)	1f		1/2	0	1/2	0.0741(3)	0.0686(3)	0.0441(2)	-0.0067(2)	0.0067(2)	-0.0122(2)
Cl(3A)	2i	0.51(6)	0.285(1)	0.014(2)	0.449(1)	0.066(4)	0.114(8)	0.080(5)	0.004(4)	0.003(3)	-0.037(4)
Cl(3B)	2i	0.49(6)	0.282(2)	-0.022(3)	0.462(1)	0.091(4)	0.17(1)	0.066(4)	-0.044(6)	0.002(3)	-0.031(7)
I(3)	1h		1/2	1/2	1/2	0.0612(3)	0.0778(3)	0.0543(3)	-0.0123(2)	0.0095(2)	-0.0206(2)
Cl(4A)	2i	0.32(3)	0.370(3)	0.591(3)	0.3967(9)	0.16(1)	0.26(3)	0.108(6)	0.10(1)	-0.018(7)	-0.03(1)
Cl(4B)	2i	0.68(3)	0.3966(4)	0.6403(5)	0.4151(4)	0.083(2)	0.109(4)	0.073(3)	-0.008(2)	-0.002(2)	0.009(2)
P(1)	2i		0.1277(1)	0.50489(8)	0.71987(6)	0.0376(6)	0.0426(6)	0.0407(6)	-0.0011(5)	-0.0035(5)	-0.0052(5)
P(2)	2i		0.6738(1)	0.03606(8)	0.20523(6)	0.0441(6)	0.0423(6)	0.0355(6)	-0.0088(5)	-0.0012(5)	-0.0093(5)
Si(1)	2i		0.0598(1)	0.6275(1)	0.2102(1)	0.0518(9)	0.079(1)	0.097(1)	-0.0257(8)	0.0031(8)	-0.0104(9)
Si(2)	2i		0.7375(1)	0.1751(1)	0.06282(8)	0.0629(9)	0.0532(8)	0.0473(7)	-0.0175(7)	0.0004(6)	-0.0007(6)
N(1)	2i		-0.0039(4)	0.5206(3)	0.2260(3)	0.045(2)	0.045(3)	0.060(3)	-0.002(2)	0.004(2)	-0.005(2)
N(2)	2i		0.6966(4)	0.0683(3)	0.1098(2)	0.060(3)	0.051(3)	0.039(2)	-0.017(2)	-0.007(2)	-0.012(2)
C(12)	2i		-0.2750(3)	0.6580(2)	0.3504(2)	0.050(3)	0.053(3)	0.048(3)	-0.001(2)	-0.004(2)	-0.005(2)
C(13)	2i		-0.3048(3)	0.7291(2)	0.4096(2)	0.068(3)	0.048(3)	0.069(3)	0.009(3)	-0.003(3)	-0.012(2)
C(14)	2i		-0.2347(3)	0.7245(2)	0.4773(2)	0.089(4)	0.057(3)	0.056(3)	-0.013(3)	0.003(3)	-0.019(3)
C(15)	2i		-0.1348(3)	0.6487(2)	0.4859(2)	0.109(5)	0.086(4)	0.059(3)	0.005(4)	-0.034(3)	-0.030(3)
C(16)	2i		-0.1050(3)	0.5776(2)	0.4266(2)	0.086(4)	0.061(3)	0.070(3)	0.014(3)	-0.034(3)	-0.019(3)
C(11)	2i		-0.1751(3)	0.5822(2)	0.3589(2)	0.044(2)	0.043(3)	0.039(2)	-0.004(2)	-0.006(2)	-0.007(2)
C(22)	2i		-0.1628(2)	0.3534(2)	0.3979(2)	0.052(3)	0.059(3)	0.065(3)	-0.002(2)	-0.004(2)	0.008(3)
C(23)	2i		-0.1354(3)	0.2616(2)	0.4335(2)	0.074(4)	0.075(4)	0.065(3)	-0.019(3)	-0.005(3)	0.020(3)
C(24)	2i		-0.0371(3)	0.1938(2)	0.3988(2)	0.093(4)	0.046(3)	0.079(4)	-0.016(3)	-0.030(3)	0.005(3)
C(25)	2i		0.0339(3)	0.2178(2)	0.3284(2)	0.083(4)	0.046(3)	0.078(4)	0.009(3)	-0.012(3)	-0.015(3)
C(26)	2i		0.0065(3)	0.3096(2)	0.2928(2)	0.066(3)	0.046(3)	0.055(3)	0.005(2)	-0.007(2)	-0.008(2)
C(21)	2i		-0.0919(3)	0.3774(2)	0.3275(2)	0.042(2)	0.045(3)	0.043(2)	-0.006(2)	-0.010(2)	-0.004(2)
C(32)	2i		-0.2385(2)	0.5223(2)	0.1335(2)	0.046(3)	0.049(3)	0.042(2)	0.000(2)	-0.002(2)	-0.008(2)
C(33)	2i		-0.3373(3)	0.5260(2)	0.0841(1)	0.066(3)	0.062(3)	0.040(2)	-0.002(3)	-0.008(2)	-0.011(2)
C(34)	2i		-0.4526(2)	0.5061(2)	0.1178(2)	0.060(3)	0.070(3)	0.060(3)	-0.005(3)	-0.023(3)	-0.010(3)
C(35)	2i		-0.4690(2)	0.4824(2)	0.2010(2)	0.050(3)	0.087(4)	0.067(3)	-0.017(3)	-0.009(3)	-0.002(3)
C(36)	2i		-0.3701(3)	0.4786(2)	0.2504(1)	0.051(3)	0.074(3)	0.046(3)	-0.010(3)	-0.007(2)	-0.002(2)
C(31)	2i		-0.2548(2)	0.4986(2)	0.2166(1)	0.042(2)	0.042(2)	0.034(2)	0.000(2)	-0.003(2)	-0.005(2)
C(41)	2i		-0.0643(6)	0.7268(4)	0.1761(4)	0.085(5)	0.061(4)	0.127(6)	-0.005(3)	0.020(4)	0.006(4)
C(42)	2i		0.1864(6)	0.5939(6)	0.1281(6)	0.074(5)	0.099(5)	0.192(8)	-0.019(4)	0.054(5)	-0.001(5)
C(43)	2i		0.119(1)	0.6647(8)	0.3027(6)	0.20(1)	0.19(1)	0.18(1)	-0.125(9)	-0.074(8)	-0.024(8)
C(52)	2i		0.6323(2)	-0.1393(2)	0.1567(2)	0.055(3)	0.045(3)	0.048(3)	-0.006(2)	-0.004(2)	-0.004(2)
C(53)	2i		0.5734(3)	-0.2198(2)	0.1614(2)	0.076(4)	0.044(3)	0.063(3)	-0.005(3)	-0.019(3)	-0.010(2)
C(54)	2i		0.4737(3)	-0.2247(2)	0.2203(2)	0.068(3)	0.050(3)	0.084(4)	-0.022(3)	-0.024(3)	0.006(3)
C(55)	2i		0.4328(2)	-0.1489(2)	0.2744(2)	0.062(3)	0.065(3)	0.069(3)	-0.023(3)	0.004(3)	-0.001(3)
C(56)	2i		0.4917(3)	-0.0684(2)	0.2696(2)	0.054(3)	0.057(3)	0.045(3)	-0.012(2)	-0.002(2)	-0.006(2)
C(51)	2i		0.5914(2)	-0.0635(2)	0.2107(2)	0.045(2)	0.042(2)	0.036(2)	-0.006(2)	-0.005(2)	-0.003(2)
C(62)	2i		0.9343(3)	0.0045(2)	0.2042(2)	0.051(3)	0.054(3)	0.062(3)	-0.009(2)	0.003(2)	-0.016(2)
C(63)	2i		1.0474(2)	-0.0255(2)	0.2392(2)	0.044(3)	0.064(3)	0.091(4)	-0.004(2)	0.000(3)	-0.022(3)
C(64)	2i		1.0474(2)	-0.0631(2)	0.3190(2)	0.055(3)	0.068(4)	0.088(4)	0.005(3)	-0.025(3)	-0.022(3)
C(65)	2i		0.9343(3)	-0.0708(2)	0.3639(2)	0.066(4)	0.083(4)	0.060(3)	0.004(3)	-0.021(3)	-0.011(3)
C(66)	2i		0.8211(2)	-0.0408(2)	0.3289(2)	0.051(3)	0.066(3)	0.045(3)	-0.005(2)	-0.006(2)	-0.003(2)
C(61)	2i		0.8211(2)	-0.0032(2)	0.2490(2)	0.044(3)	0.044(3)	0.046(2)	-0.006(2)	-0.002(2)	-0.015(2)
C(72)	2i		0.6224(2)	0.1695(2)	0.3324(2)	0.060(3)	0.054(3)	0.052(3)	-0.009(2)	-0.008(2)	-0.012(2)
C(73)	2i		0.5479(3)	0.2477(2)	0.3729(2)	0.079(4)	0.061(3)	0.050(3)	-0.012(3)	0.001(3)	-0.021(2)
C(74)	2i		0.4339(3)	0.2900(2)	0.3436(2)	0.064(3)	0.056(3)	0.071(3)	-0.006(3)	0.019(3)	-0.023(3)
C(75)	2i		0.3945(2)	0.2541(2)	0.2739(2)	0.051(3)	0.066(3)	0.084(4)	0.003(3)	-0.003(3)	-0.019(3)
C(76)	2i		0.4690(3)	0.1759(2)	0.2335(2)	0.054(3)	0.057(3)	0.062(3)	-0.003(2)	-0.008(2)	-0.020(2)
C(71)	2i		0.5830(2)	0.1336(2)	0.2628(2)	0.045(3)	0.042(3)	0.044(2)	-0.010(2)	-0.001(2)	-0.009(2)
C(81)	2i		0.7929(5)	0.2460(4)	0.1412(3)	0.080(4)	0.051(3)	0.079(4)	-0.023(3)	-0.004(3)	-0.007(3)
C(82)	2i		0.5972(6)	0.2434(5)	0.0185(4)	0.093(5)	0.090(5)	0.102(5)	-0.025(4)	-0.030(4)	0.039(4)
C(83)	2i		0.8660(6)	0.1358(5)	-0.0185(4)	0.107(5)	0.096(5)	0.073(4)	-0.039(4)	0.029(4)	-0.006(3)

References

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