Crystal structure of tetraphenylphosphonium hexachlorogermanate bis(dichloromethane) solvate, $(\text{P}(\text{C}_6\text{H}_5)_4)_2\text{GeCl}_6 \cdot 2\text{CH}_2\text{Cl}_2$

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Source of material: Reaction of germanium tetrachloride with tetraphenylphosphonium chloride in dichloromethane in the presence of sodium tetrasulfide; the latter probably is not essential for the reaction.

$(\text{PPh}_4)_2[\text{GeCl}_6] \cdot 2\text{CH}_2\text{Cl}_2$ crystallizes with the structure type of $\beta\text{-(PPh}_4)_2[\text{UCl}_6] \cdot 2\text{CH}_2\text{Cl}_2$ (see refs. 1, 2). To our knowledge, the only other structurally characterized hexachlorogermanate is Cs_2GeCl_6 (see ref. 3).

$\text{C}_{50}\text{H}_{44}\text{Cl}_{10}\text{GeP}_2$, triclinic, $\bar{P}1$ (No. 2), $a = 10.110(4)$ Å, $b = 11.07(1)$ Å, $c = 12.350(8)$ Å, $\alpha = 70.68(7)^\circ$, $\beta = 80.18(4)^\circ$, $\gamma = 80.47(5)^\circ$, $V = 1276.3$ Å³, $Z = 1$, $R(F) = 0.033$, $R_w(F^2) = 0.086$.

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

Crystal:	light yellow platelet, size 0.12 x 0.45 x 0.51 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ :	12.21 cm ⁻¹
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω
$T_{\text{measurement}}$:	305 K
$2\theta_{\text{max}}$:	48°
$N(hkl)_{\text{unique}}$:	3894
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	286
Programs:	SHELXS-86, SHELXL-93, ZORTEP

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

Atom	Site	x	y	z	U_{iso}
H(2)	2i	0.6210(3)	0.4958(3)	0.3632(2)	0.041
H(3)	2i	0.6393(3)	0.7083(3)	0.2515(3)	0.048
H(4)	2i	0.5358(3)	0.7952(3)	0.0860(2)	0.045
H(5)	2i	0.4149(3)	0.6721(3)	0.0291(2)	0.044
H(6)	2i	0.3988(3)	0.4585(2)	0.1383(2)	0.036
H(8)	2i	0.2556(2)	0.2596(2)	0.2744(2)	0.035
H(9)	2i	0.2122(3)	0.1451(3)	0.1611(2)	0.039
H(10)	2i	0.3828(3)	0.0090(2)	0.0985(2)	0.038
H(11)	2i	0.5993(3)	-0.0059(2)	0.1397(2)	0.039
H(12)	2i	0.6464(3)	0.1107(2)	0.2511(2)	0.033
H(14)	2i	0.5517(3)	0.1912(3)	0.5881(2)	0.046
H(15)	2i	0.7519(4)	0.1240(3)	0.6703(3)	0.064
H(16)	2i	0.9553(3)	0.1196(3)	0.5550(3)	0.064
H(17)	2i	0.9635(3)	0.1911(3)	0.3556(3)	0.056
H(18)	2i	0.7654(3)	0.2684(3)	0.2720(2)	0.041
H(20)	2i	0.3012(3)	0.4947(3)	0.4173(2)	0.036
H(21)	2i	0.1138(3)	0.4955(3)	0.5555(2)	0.045
H(22)	2i	0.0465(3)	0.3037(3)	0.6810(2)	0.047
H(23)	2i	0.1614(3)	0.1098(3)	0.6684(2)	0.044
H(24)	2i	0.3449(3)	0.1067(3)	0.5292(2)	0.038
H(25)	2i	0.1318(3)	0.6599(3)	0.0762(3)	0.052
H(26)	2i	0.0094(3)	0.6266(3)	0.0328(3)	0.052

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ge	1a	0	0	0	0.0211(2)	0.0229(2)	0.0192(2)	-0.0081(1)	0.0006(1)	-0.0070(1)
Cl(1)	2i	0.15401(6)	0.14468(6)	-0.07826(5)	0.0273(3)	0.0295(3)	0.0297(3)	-0.0141(2)	0.0035(2)	-0.0081(3)
Cl(2)	2i	0.08357(7)	-0.10371(6)	-0.13507(6)	0.0407(4)	0.0357(3)	0.0314(4)	-0.0146(3)	0.0103(3)	-0.0182(3)
Cl(3)	2i	0.14917(7)	-0.13108(6)	0.12245(6)	0.0380(4)	0.0292(3)	0.0429(4)	-0.0072(3)	-0.0184(3)	-0.0059(3)
P	2i	0.48715(6)	0.29599(6)	0.35399(5)	0.0251(3)	0.0212(3)	0.0186(3)	-0.0071(2)	-0.0019(2)	-0.0055(2)
C(1)	2i	0.5071(2)	0.4568(2)	0.2618(2)	0.025(1)	0.022(1)	0.022(1)	-0.0086(9)	0.002(1)	-0.005(1)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(2)	2i	0.5798(3)	0.5311(3)	0.2960(2)	0.044(2)	0.033(1)	0.028(1)	-0.016(1)	-0.006(1)	-0.008(1)
C(3)	2i	0.5905(3)	0.6581(3)	0.2293(3)	0.053(2)	0.031(2)	0.041(2)	-0.021(1)	-0.002(1)	-0.011(1)
C(4)	2i	0.5288(3)	0.7098(3)	0.1303(2)	0.050(2)	0.024(1)	0.035(2)	-0.013(1)	0.004(1)	-0.002(1)
C(5)	2i	0.4566(3)	0.6362(3)	0.0960(2)	0.045(2)	0.030(1)	0.029(1)	-0.006(1)	-0.006(1)	0.001(1)
C(6)	2i	0.4463(3)	0.5088(2)	0.1616(2)	0.036(1)	0.026(1)	0.029(1)	-0.009(1)	-0.006(1)	-0.005(1)
C(7)	2i	0.4558(2)	0.1976(2)	0.2735(2)	0.027(1)	0.019(1)	0.019(1)	-0.0077(9)	-0.002(1)	-0.003(1)
C(8)	2i	0.3251(2)	0.2067(2)	0.2470(2)	0.026(1)	0.029(1)	0.032(1)	-0.004(1)	-0.004(1)	-0.010(1)
C(9)	2i	0.2990(3)	0.1375(3)	0.1802(2)	0.031(1)	0.036(1)	0.034(2)	-0.011(1)	-0.010(1)	-0.010(1)
C(10)	2i	0.4013(3)	0.0572(2)	0.1419(2)	0.043(2)	0.030(1)	0.027(1)	-0.014(1)	-0.003(1)	-0.012(1)
C(11)	2i	0.5306(3)	0.0476(2)	0.1672(2)	0.039(2)	0.029(1)	0.032(2)	-0.005(1)	0.000(1)	-0.014(1)
C(12)	2i	0.5590(3)	0.1175(2)	0.2336(2)	0.026(1)	0.028(1)	0.029(1)	-0.005(1)	-0.005(1)	-0.007(1)
C(13)	2i	0.6390(2)	0.2345(2)	0.4223(2)	0.031(1)	0.025(1)	0.025(1)	-0.008(1)	-0.008(1)	-0.007(1)
C(14)	2i	0.6342(3)	0.1921(3)	0.5414(2)	0.044(2)	0.042(2)	0.028(2)	-0.016(1)	-0.008(1)	-0.003(1)
C(15)	2i	0.7540(4)	0.1509(3)	0.5903(3)	0.066(2)	0.056(2)	0.037(2)	-0.020(2)	-0.027(2)	0.006(2)
C(16)	2i	0.8757(3)	0.1495(3)	0.5214(3)	0.045(2)	0.051(2)	0.066(2)	-0.010(2)	-0.032(2)	-0.007(2)
C(17)	2i	0.8808(3)	0.1924(3)	0.4019(3)	0.033(2)	0.051(2)	0.061(2)	-0.007(1)	-0.011(1)	-0.021(2)
C(18)	2i	0.7625(3)	0.2370(3)	0.3521(2)	0.031(1)	0.042(2)	0.034(2)	-0.007(1)	-0.005(1)	-0.015(1)
C(19)	2i	0.3429(2)	0.3008(2)	0.4600(2)	0.027(1)	0.029(1)	0.020(1)	-0.009(1)	-0.002(1)	-0.007(1)
C(20)	2i	0.2733(3)	0.4174(3)	0.4674(2)	0.033(1)	0.031(1)	0.027(1)	-0.007(1)	-0.003(1)	-0.009(1)
C(21)	2i	0.1613(3)	0.4177(3)	0.5504(2)	0.034(2)	0.045(2)	0.036(2)	-0.000(1)	-0.001(1)	-0.018(1)
C(22)	2i	0.1207(3)	0.3028(3)	0.6252(2)	0.028(1)	0.062(2)	0.027(1)	-0.010(1)	0.003(1)	-0.016(1)
C(23)	2i	0.1894(3)	0.1868(3)	0.6177(2)	0.038(2)	0.047(2)	0.024(1)	-0.019(1)	0.000(1)	-0.004(1)
C(24)	2i	0.2995(3)	0.1849(3)	0.5351(2)	0.035(1)	0.032(1)	0.028(1)	-0.010(1)	-0.003(1)	-0.007(1)
C	2i	0.0913(3)	0.5900(3)	0.0685(3)	0.048(2)	0.032(2)	0.051(2)	-0.004(1)	-0.003(1)	-0.015(1)
Cl(4)	2i	0.20430(8)	0.51381(9)	-0.02053(7)	0.0464(4)	0.0732(6)	0.0495(5)	0.0015(4)	-0.0085(4)	-0.0322(4)
Cl(5)	2i	0.0506(1)	0.48103(9)	0.20628(8)	0.0750(6)	0.0549(5)	0.0494(5)	0.0129(4)	0.0049(4)	-0.0064(4)

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