Crystal structure of the new modification of tetraphenylphosphonium chloride, β -P(C₆H₅)₄Cl

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C₂₄H₂₀ClP, triclinic, $P\bar{1}$ (No. 2),
 $a = 9.387(1)$ Å, $b = 10.201(1)$ Å,
 $c = 11.248(1)$ Å, $\alpha = 75.83(1)^\circ$,
 $\beta = 71.42(1)^\circ$, $\gamma = 78.46(1)^\circ$, $V = 981.1$ Å³,
 $Z = 2$, $R(F) = 0.036$, $R_w(F^2) = 0.092$.

Source of material: A solution of 1.66 g PPh₄Cl in 70 ml of dry acetonitrile was stirred for 15 h together with 0.32 g Na₂S₄ and 0.16 g CoCl₂, filtered and evaporated to 10 ml. β -PPh₄Cl crystallized at 253 K.

In the new modification β -PPh₄Cl the tetraphenylphosphonium ions are arranged to zigzag chains, in a similar way as in β -(PPh₄)₂[UCl₆]·2CH₂Cl₂ (see refs. 1, 2). The known monoclinic modification α -PPh₄Cl (see ref. 3) as well as PPh₄Br (see ref. 4) and PPh₄I (see refs. 5, 6) crystallize with different packings.

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

Crystal:	colorless, compact, size 0.25 x 0.31 x 0.42 mm
Wavelength:	Mo $K\alpha$ radiation (0.71069 Å)
μ :	2.81 cm ⁻¹
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω
$T_{\text{measurement}}$:	297 K
$2\theta_{\text{max}}$:	42°
$N(hkl)_{\text{unique}}$:	1778
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	238
Programs:	SHELXS-86, SHELXL-93, SCHAKAL

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	2i	-0.2170(4)	0.7945(4)	0.3258(3)	0.069
H(3)	2i	-0.4632(5)	0.7468(5)	0.3944(4)	0.088
H(4)	2i	-0.5148(5)	0.5466(5)	0.3713(4)	0.093
H(5)	2i	-0.3230(5)	0.3882(4)	0.2876(4)	0.092
H(6)	2i	-0.0737(4)	0.4307(4)	0.2228(3)	0.069
H(8)	2i	-0.0478(4)	0.6858(4)	0.4630(3)	0.067
H(9)	2i	0.0186(5)	0.7941(4)	0.5908(4)	0.084
H(10)	2i	0.2210(5)	0.9162(4)	0.5090(4)	0.076
H(11)	2i	0.3549(4)	0.9331(3)	0.2971(4)	0.066
H(12)	2i	0.2887(4)	0.8298(3)	0.1665(3)	0.057
H(14)	2i	0.2211(4)	0.4754(3)	0.3642(3)	0.059
H(15)	2i	0.3662(4)	0.2624(4)	0.3608(4)	0.073
H(16)	2i	0.4342(4)	0.1713(4)	0.1795(4)	0.078
H(17)	2i	0.3645(4)	0.2902(4)	0.0015(4)	0.082
H(18)	2i	0.2133(4)	0.4990(4)	0.0051(4)	0.072
H(20)	2i	0.3074(4)	0.7614(4)	-0.0215(3)	0.073
H(21)	2i	0.3129(5)	0.8925(4)	-0.2225(3)	0.079
H(22)	2i	0.0922(5)	0.9685(4)	-0.2790(4)	0.074
H(23)	2i	-0.1353(5)	0.9188(4)	-0.1372(4)	0.079
H(24)	2i	-0.1446(4)	0.7915(3)	0.0642(3)	0.065

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Cl(1)	1f	1/2	0	1/2	0.072(1)	0.0633(9)	0.0668(9)	-0.0178(7)	-0.0360(7)	-0.0099(7)
Cl(2)	1d	1/2	0	0	0.099(1)	0.069(1)	0.084(1)	-0.0355(9)	0.0378(9)	-0.0403(8)
P	2i	0.06948(9)	0.66085(8)	0.19876(8)	0.0400(6)	0.0414(5)	0.0392(5)	-0.0018(4)	-0.0101(4)	-0.0074(4)
C(1)	2i	-0.1188(4)	0.6177(3)	0.2660(3)	0.040(2)	0.042(2)	0.044(2)	-0.006(2)	-0.014(2)	-0.007(2)
C(2)	2i	-0.2375(4)	0.7119(4)	0.3182(3)	0.046(3)	0.056(2)	0.070(3)	-0.002(2)	-0.016(2)	-0.015(2)
C(3)	2i	-0.3847(5)	0.6840(5)	0.3585(4)	0.046(3)	0.089(3)	0.079(3)	0.000(2)	-0.012(2)	-0.021(2)
C(4)	2i	-0.4150(5)	0.5645(5)	0.3456(4)	0.047(3)	0.107(4)	0.080(3)	-0.028(3)	-0.009(2)	-0.019(3)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(5)	2i	-0.3006(5)	0.4700(4)	0.2953(4)	0.073(3)	0.080(3)	0.084(3)	-0.036(3)	-0.012(3)	-0.021(2)
C(6)	2i	-0.1515(4)	0.4956(4)	0.2559(3)	0.054(3)	0.057(2)	0.060(2)	-0.013(2)	-0.010(2)	-0.012(2)
C(7)	2i	0.1129(3)	0.7482(3)	0.3002(3)	0.040(2)	0.037(2)	0.038(2)	-0.001(2)	-0.012(2)	-0.004(2)
C(8)	2i	0.0329(4)	0.7366(4)	0.4286(3)	0.059(2)	0.065(2)	0.045(2)	-0.022(2)	-0.009(2)	-0.011(2)
C(9)	2i	0.0736(5)	0.8007(4)	0.5051(4)	0.081(3)	0.090(3)	0.043(2)	-0.026(3)	-0.011(2)	-0.018(2)
C(10)	2i	0.1937(5)	0.8738(4)	0.4567(4)	0.074(3)	0.066(3)	0.061(3)	-0.013(2)	-0.029(2)	-0.017(2)
C(11)	2i	0.2730(4)	0.8837(3)	0.3305(4)	0.052(2)	0.051(2)	0.066(3)	-0.012(2)	-0.022(2)	-0.006(2)
C(12)	2i	0.2336(4)	0.8219(3)	0.2522(3)	0.043(2)	0.050(2)	0.044(2)	-0.007(2)	-0.008(2)	-0.005(2)
C(13)	2i	0.2001(3)	0.5072(3)	0.1863(3)	0.038(2)	0.043(2)	0.044(2)	-0.001(2)	-0.009(2)	-0.010(2)
C(14)	2i	0.2478(4)	0.4373(3)	0.2916(3)	0.044(2)	0.049(2)	0.049(2)	-0.004(2)	-0.010(2)	-0.007(2)
C(15)	2i	0.3355(4)	0.3103(4)	0.2895(4)	0.053(2)	0.048(2)	0.069(3)	0.001(2)	-0.019(2)	0.004(2)
C(16)	2i	0.3764(4)	0.2567(4)	0.1810(4)	0.052(2)	0.045(2)	0.091(3)	0.004(2)	-0.014(2)	-0.018(3)
C(17)	2i	0.3334(4)	0.3269(4)	0.0751(4)	0.064(3)	0.063(3)	0.081(3)	0.011(2)	-0.019(2)	-0.037(2)
C(18)	2i	0.2439(4)	0.4521(4)	0.0768(4)	0.063(3)	0.060(3)	0.059(2)	0.006(2)	-0.024(2)	-0.021(2)
C(19)	2i	0.0800(4)	0.7623(3)	0.0422(3)	0.048(2)	0.041(2)	0.041(2)	0.000(2)	-0.015(2)	-0.008(2)
C(20)	2i	0.2178(4)	0.7930(4)	-0.0442(3)	0.049(2)	0.077(3)	0.045(2)	-0.003(2)	-0.009(2)	-0.004(2)
C(21)	2i	0.2210(5)	0.8711(4)	-0.1644(3)	0.072(3)	0.071(3)	0.041(2)	-0.011(2)	-0.002(2)	-0.004(2)
C(22)	2i	0.0892(5)	0.9168(4)	-0.1979(4)	0.093(3)	0.047(2)	0.047(2)	-0.003(2)	-0.029(3)	-0.006(2)
C(23)	2i	-0.0463(5)	0.8872(4)	-0.1136(4)	0.072(3)	0.059(3)	0.069(3)	-0.007(2)	-0.036(2)	0.006(2)
C(24)	2i	-0.0517(4)	0.8107(3)	0.0066(3)	0.057(3)	0.051(2)	0.053(2)	-0.009(2)	-0.022(2)	0.001(2)

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