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Crystal structure of 1,2-vinylenbis(triphenylphosphonium) dibromide dihydrate, $[Ph_3P–CH=CH–PPh_3][Br]_2 \cdot 2H_2O$

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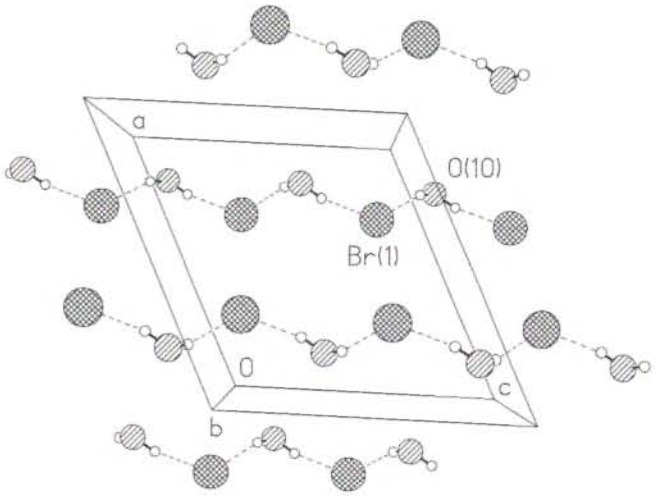

Abstract

$C_{19}H_{18}BrOP$, monoclinic, $P12_1/c1$ (No. 14), $a = 11.716(3)$ Å, $b = 14.145(4)$ Å, $c = 11.250(3)$ Å, $\beta = 115.39(2)°$, $V = 1684.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.045$, $wR_{ref}(F^2) = 0.112$, $T = 293$ K.

Source of material

During attempts to prepare $[Ph_3P–CH=CH–PPh_3][CoBr_4]$ a fine crystalline white precipitate was observed. Single crystals of $[Ph_3P–CH=CH–PPh_3][Br]_2 \cdot 2H_2O$ were obtained by slow evaporation of water from an aqueous solution of this precipitate in a desiccator.

Discussion

Bromide anions and water molecules are connected by hydrogen bonds and form infinitive zig-zag chains running along [001]. Bond distances and bond angles in the cation agree well with the data of Stang and Zhdankin [1]. The compound was investigated with a differential scanning calorimeter. Between 153 K and 423 K no phase transition was detected.

Table 1. Data collection and handling.

Crystal:	transparent block, size 0.21 × 0.43 × 0.62 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	25.35 cm^{-1}
Diffractometer, scan mode:	Siemens P4, 2θ/ω
$2\theta_{max}$:	44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2669, 2055
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1606
$N(param)_{refined}$:	201
Program:	SHELXLTL [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.9116	0.4292	0.9503	0.059(4)
H(11)	4e	0.974	0.6656	1.1917	0.059(4)
H(12)	4e	1.0607	0.8150	1.2584	0.059(4)
H(13)	4e	1.0281	0.9301	1.1056	0.059(4)
H(14)	4e	0.9121	0.8997	0.8849	0.059(4)
H(15)	4e	0.8266	0.7518	0.8170	0.059(4)
H(21)	4e	0.6742	0.6862	1.0457	0.059(4)
H(22)	4e	0.5586	0.6510	1.1614	0.059(4)
H(23)	4e	0.5585	0.4996	1.2362	0.059(4)
H(24)	4e	0.6693	0.3817	1.1914	0.059(4)
H(25)	4e	0.7742	0.4132	1.0635	0.059(4)
H(31)	4e	0.5794	0.6619	0.7897	0.059(4)
H(32)	4e	0.4594	0.6787	0.5670	0.059(4)
H(33)	4e	0.5237	0.6144	0.4206	0.059(4)
H(34)	4e	0.7108	0.5313	0.4926	0.059(4)
H(35)	4e	0.8326	0.5116	0.7140	0.059(4)
H(101)	4e	0.7610	0.1776	1.1373	0.059(4)
H(102)	4e	0.8010	0.2174	1.0184	0.059(4)

Table 3. 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}
Br(1)	4e	0.69041(5)	0.30742(4)	0.81035(6)	0.0438(4)	0.0474(4)	0.0529(4)	–0.0008(3)	0.0229(3)	–0.0034(3)
P(1)	4e	0.8212(1)	0.58076(9)	0.9472(1)	0.0156(6)	0.0260(7)	0.0310(8)	0.0025(5)	0.0089(5)	0.0013(6)
C(1)	4e	0.9385(4)	0.4911(4)	0.9741(5)	0.021(2)	0.025(3)	0.037(3)	0.004(2)	0.011(2)	–0.001(2)
C(10)	4e	0.8924(4)	0.6944(4)	0.9971(5)	0.021(2)	0.027(3)	0.041(3)	0.000(2)	0.015(2)	–0.002(3)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(11)	4*e*	0.9619(5)	0.7127(4)	1.1297(6)	0.031(3)	0.035(3)	0.052(4)	0.001(2)	0.015(3)	0.000(3)
C(12)	4*e*	1.0132(6)	0.8021(4)	1.1695(7)	0.041(3)	0.044(4)	0.061(4)	–0.007(3)	0.013(3)	–0.021(4)
C(13)	4*e*	0.9939(6)	0.8702(4)	1.0786(8)	0.054(4)	0.035(4)	0.092(6)	–0.015(3)	0.034(4)	–0.014(4)
C(14)	4*e*	0.9245(7)	0.8522(5)	0.9465(8)	0.075(5)	0.042(4)	0.071(5)	–0.013(4)	0.030(4)	0.007(4)
C(15)	4*e*	0.8737(6)	0.7642(4)	0.9061(6)	0.048(3)	0.034(3)	0.047(4)	–0.006(3)	0.020(3)	0.003(3)
C(20)	4*e*	0.7384(4)	0.5527(3)	1.0449(5)	0.020(2)	0.029(3)	0.028(3)	0.000(2)	0.008(2)	0.000(2)
C(21)	4*e*	0.6721(5)	0.6247(4)	1.0738(6)	0.036(3)	0.032(3)	0.052(4)	–0.002(2)	0.024(3)	0.001(3)
C(22)	4*e*	0.6043(5)	0.6038(4)	1.1436(6)	0.041(3)	0.054(4)	0.052(4)	–0.006(3)	0.034(3)	–0.013(3)
C(23)	4*e*	0.6036(5)	0.5131(5)	1.1875(6)	0.045(3)	0.066(4)	0.041(4)	–0.017(3)	0.028(3)	–0.011(3)
C(24)	4*e*	0.6689(6)	0.4426(4)	1.1599(6)	0.055(4)	0.042(4)	0.047(4)	–0.011(3)	0.021(3)	0.010(3)
C(25)	4*e*	0.7336(5)	0.4617(4)	1.0860(5)	0.033(3)	0.035(3)	0.043(3)	0.000(2)	0.018(3)	0.002(3)
C(30)	4*e*	0.7210(4)	0.5844(3)	0.7759(5)	0.019(2)	0.029(3)	0.040(3)	–0.003(2)	0.014(2)	0.000(2)
C(31)	4*e*	0.6062(5)	0.6354(4)	0.7303(6)	0.024(3)	0.039(3)	0.043(4)	0.003(2)	0.010(3)	0.002(3)
C(32)	4*e*	0.5350(5)	0.6453(4)	0.5976(6)	0.029(3)	0.043(3)	0.054(4)	0.003(3)	0.002(3)	0.007(3)
C(33)	4*e*	0.5733(6)	0.6070(5)	0.5103(6)	0.048(4)	0.053(4)	0.036(4)	–0.010(3)	0.004(3)	0.008(3)
C(34)	4*e*	0.6850(5)	0.5571(5)	0.5531(6)	0.042(3)	0.070(5)	0.035(4)	–0.003(3)	0.013(3)	–0.007(3)
C(35)	4*e*	0.7577(5)	0.5457(4)	0.6852(5)	0.029(3)	0.051(4)	0.032(3)	0.004(3)	0.006(3)	–0.003(3)
O(10)	4*e*	0.8130(4)	0.1592(4)	1.0812(5)	0.055(3)	0.124(5)	0.069(3)	0.006(3)	0.024(3)	0.021(3)

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