Crystal structure of (2*R*,4*S*,5*R*)-2-(4-benzyloxy-5-benzyloxymethyl-tetrahydrofuran-2-yl)triphenylphosphonium tetrafluoroborate tetrachloromethane solvate, C₃₇H₃₆PBF₄O₃ · CCl₄

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Source of material: The title compound (see ref. 1 and 2) was prepared by reaction of methyl 3,5-di-*O*-benzyl-2,3-dideoxy-D-ribofuranoside (see ref. 3) with hydrogentriphenylphosphonium tetrafluoroborate in acetonitrile. The precipitated phosphonium salt was recrystallized from tetrachloromethane/dichloromethane. One tetrachloromethane solvent molecule per asymmetric unit was found. This solvent molecule was strongly disordered.

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

Crystal:	colorless needle, size 0.20 x 0.25 x 2.0 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.11 cm ⁻¹
Diffractometer:	Nicolet P3
Scan mode:	Wyckoff
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	50°
$N(hkl)_{\text{unique}}$:	3398
Criterion for I_0 :	$I_0 > 2 \sigma(I_0)$
$N(\text{param})_{\text{refined}}$:	491
Programs:	SHELXS-86, SHELXL-93

C₃₈H₃₆BCl₄F₄O₃P, orthorhombic, $P2_12_12_1$ (No. 19), $a = 9.975(5)$ Å, $b = 18.234(7)$ Å, $c = 20.820(7)$ Å, $V = 3786.8$ Å³, $Z = 4$, $R(F) = 0.080$, $R_w(F^2) = 0.173$.

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

Atom	Site	x	y	z	U_{iso}
H(1)	4a	0.8489(8)	0.5695(4)	0.8765(3)	0.052
H(2A)	4a	0.7821(9)	0.5848(4)	0.7724(3)	0.055
H(2B)	4a	0.6783(9)	0.6471(4)	0.7902(3)	0.055
H(3)	4a	0.8510(9)	0.7167(4)	0.7550(3)	0.053
H(4)	4a	0.9623(9)	0.7425(4)	0.8652(3)	0.053
H(5A)	4a	0.807(1)	0.8241(4)	0.8176(4)	0.061
H(5B)	4a	0.688(1)	0.7737(4)	0.8399(4)	0.061
H(6A)	4a	0.618(1)	0.8856(5)	0.8821(4)	0.069
H(6B)	4a	0.755(1)	0.9284(5)	0.8780(4)	0.069
H(8)	4a	0.664(1)	1.0325(5)	0.9344(5)	0.083
H(9)	4a	0.598(1)	1.0859(6)	1.0313(6)	0.100
H(10)	4a	0.567(1)	1.0144(7)	1.1211(5)	0.092
H(11)	4a	0.6135(9)	0.8914(6)	1.1175(4)	0.074
H(12)	4a	0.6824(9)	0.8377(5)	1.0220(4)	0.061
H(13A)	4a	0.999(1)	0.6652(7)	0.6875(4)	0.097
H(13B)	4a	0.977(1)	0.5841(7)	0.7106(4)	0.097
H(15)	4a	1.182(1)	0.5115(5)	0.7441(4)	0.079
H(16)	4a	1.406(1)	0.4931(6)	0.7249(5)	0.089
H(17)	4a	1.537(1)	0.5853(8)	0.6834(5)	0.104
H(18)	4a	1.438(1)	0.6919(7)	0.6530(5)	0.097
H(19)	4a	1.214(1)	0.7132(6)	0.6699(5)	0.091
H(21)	4a	0.3785(9)	0.5076(4)	0.9115(4)	0.065
H(22)	4a	0.293(1)	0.4005(5)	0.8666(5)	0.080
H(23)	4a	0.430(1)	0.3288(5)	0.8052(5)	0.085
H(24)	4a	0.649(1)	0.3608(5)	0.7869(5)	0.088
H(25)	4a	0.733(1)	0.4686(5)	0.8307(5)	0.078
H(27)	4a	0.8577(9)	0.5465(5)	0.9853(4)	0.058
H(28)	4a	0.895(1)	0.5189(5)	1.0932(4)	0.071
H(29)	4a	0.725(1)	0.5273(5)	1.1656(4)	0.075
H(30)	4a	0.512(1)	0.5524(6)	1.1330(4)	0.088
H(31)	4a	0.4700(9)	0.5827(6)	1.0265(4)	0.072
H(33)	4a	0.4153(9)	0.6211(4)	0.8237(4)	0.059
H(34)	4a	0.272(1)	0.7192(5)	0.8134(5)	0.073
H(35)	4a	0.272(1)	0.8113(5)	0.8897(5)	0.071
H(36)	4a	0.422(1)	0.8062(5)	0.9745(4)	0.074
H(37)	4a	0.5710(9)	0.7103(4)	0.9834(4)	0.062

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
P(1)	4a		0.6246(2)	0.5815(1)	0.9114(1)	0.041(1)	0.0345(9)	0.041(1)	−0.001(1)	0.005(1)	−0.0022(9)
O(1)	4a		0.8248(5)	0.6738(3)	0.9023(2)	0.059(3)	0.040(3)	0.029(3)	−0.012(3)	0.000(2)	0.000(2)
C(1)	4a		0.7814(8)	0.6081(4)	0.8711(3)	0.046(4)	0.042(4)	0.042(4)	−0.006(4)	0.006(4)	−0.005(3)
O(2)	4a		0.7735(6)	0.8279(3)	0.9110(2)	0.073(4)	0.044(3)	0.034(3)	0.013(3)	0.002(3)	0.000(2)
C(2)	4a		0.7664(9)	0.6272(4)	0.7995(3)	0.057(5)	0.052(5)	0.028(4)	−0.007(4)	0.000(4)	−0.007(3)
O(3)	4a		1.0026(6)	0.6543(3)	0.7823(2)	0.053(3)	0.054(3)	0.027(2)	−0.002(3)	0.011(2)	−0.005(2)
C(3)	4a		0.8736(9)	0.6839(4)	0.7907(3)	0.056(5)	0.048(4)	0.028(4)	0.002(5)	0.007(4)	−0.002(3)
C(4)	4a		0.8716(9)	0.7258(4)	0.8544(3)	0.056(5)	0.041(4)	0.035(4)	−0.002(4)	0.008(4)	−0.001(3)
C(5)	4a		0.777(1)	0.7906(4)	0.8509(4)	0.077(6)	0.047(4)	0.029(4)	−0.001(5)	0.008(4)	0.002(3)
C(6)	4a		0.703(1)	0.8948(5)	0.9041(4)	0.066(6)	0.052(5)	0.055(5)	0.010(5)	−0.001(5)	0.002(4)
C(7)	4a		0.6744(8)	0.9297(4)	0.9687(4)	0.044(4)	0.040(5)	0.053(5)	0.005(4)	−0.006(4)	−0.011(4)
C(8)	4a		0.653(1)	1.0038(5)	0.9709(5)	0.089(8)	0.050(5)	0.069(6)	0.000(5)	−0.027(6)	−0.008(5)
C(9)	4a		0.613(1)	1.0356(6)	1.0291(6)	0.090(8)	0.058(6)	0.101(8)	0.010(6)	−0.032(8)	−0.037(6)
C(10)	4a		0.596(1)	0.9931(7)	1.0830(5)	0.054(6)	0.100(8)	0.075(7)	0.010(6)	−0.007(6)	−0.043(7)
C(11)	4a		0.6226(9)	0.9198(6)	1.0807(4)	0.053(5)	0.085(7)	0.047(5)	−0.004(6)	0.008(5)	−0.014(5)
C(12)	4a		0.6631(9)	0.8876(5)	1.0234(4)	0.056(5)	0.046(4)	0.051(5)	0.000(4)	0.001(4)	−0.007(4)
C(13)	4a		1.028(1)	0.6286(7)	0.7183(4)	0.078(7)	0.13(1)	0.040(5)	0.008(7)	0.005(5)	−0.028(6)
C(14)	4a		1.170(1)	0.6141(5)	0.7095(4)	0.065(6)	0.066(6)	0.037(4)	0.003(5)	0.016(4)	−0.020(4)
C(15)	4a		1.232(1)	0.5489(5)	0.7255(4)	0.097(8)	0.053(5)	0.046(5)	−0.011(6)	0.008(5)	−0.015(4)
C(16)	4a		1.367(1)	0.5380(6)	0.7146(5)	0.089(8)	0.071(7)	0.062(6)	0.021(7)	−0.013(6)	−0.022(5)
C(17)	4a		1.445(1)	0.5921(8)	0.6890(5)	0.062(7)	0.13(1)	0.064(7)	0.010(8)	0.007(6)	−0.044(8)
C(18)	4a		1.386(1)	0.6555(7)	0.6720(5)	0.081(8)	0.093(8)	0.067(7)	−0.021(8)	0.027(7)	−0.013(6)
C(19)	4a		1.251(1)	0.6684(6)	0.6817(5)	0.100(9)	0.062(6)	0.066(6)	0.000(7)	0.026(6)	−0.008(5)
C(20)	4a		0.5613(8)	0.4993(4)	0.8752(4)	0.037(4)	0.039(4)	0.050(4)	0.003(4)	0.004(4)	−0.006(4)
C(21)	4a		0.4334(9)	0.4785(4)	0.8859(4)	0.046(5)	0.044(5)	0.074(6)	0.001(4)	0.005(5)	−0.010(4)
C(22)	4a		0.382(1)	0.4140(5)	0.8594(5)	0.057(5)	0.046(5)	0.097(7)	−0.007(5)	0.000(6)	−0.007(5)
C(23)	4a		0.463(1)	0.3717(5)	0.8230(5)	0.072(7)	0.049(5)	0.092(8)	−0.007(5)	−0.008(6)	−0.015(5)
C(24)	4a		0.594(1)	0.3908(5)	0.8118(5)	0.077(7)	0.047(5)	0.096(8)	0.004(5)	0.013(6)	−0.025(5)
C(25)	4a		0.644(1)	0.4551(5)	0.8380(5)	0.063(6)	0.052(5)	0.080(6)	−0.002(5)	0.013(6)	−0.011(5)
C(26)	4a		0.6598(8)	0.5655(4)	0.9952(4)	0.048(5)	0.037(4)	0.051(5)	−0.002(4)	0.013(4)	0.002(3)
C(27)	4a		0.7875(9)	0.5479(5)	1.0147(4)	0.049(5)	0.056(5)	0.040(4)	−0.006(4)	0.008(4)	0.008(4)
C(28)	4a		0.810(1)	0.5322(5)	1.0794(4)	0.055(5)	0.059(5)	0.064(6)	0.007(5)	−0.009(5)	0.013(5)
C(29)	4a		0.708(1)	0.5360(5)	1.1224(4)	0.061(6)	0.078(7)	0.048(5)	0.003(6)	0.004(5)	0.013(5)
C(30)	4a		0.581(1)	0.5524(6)	1.1033(4)	0.080(7)	0.097(8)	0.042(5)	0.002(6)	0.024(5)	0.006(5)
C(31)	4a		0.5557(9)	0.5692(6)	1.0395(4)	0.045(5)	0.087(7)	0.048(5)	−0.001(5)	0.007(4)	0.014(5)
C(32)	4a		0.5067(8)	0.6546(4)	0.9046(4)	0.042(4)	0.035(4)	0.045(4)	−0.007(3)	0.005(4)	−0.004(3)
C(33)	4a		0.4172(9)	0.6585(4)	0.8540(4)	0.065(6)	0.041(4)	0.041(4)	0.001(4)	−0.007(4)	−0.014(4)
C(34)	4a		0.331(1)	0.7169(5)	0.8480(5)	0.069(6)	0.057(5)	0.058(5)	0.021(5)	−0.017(5)	0.006(5)
C(35)	4a		0.331(1)	0.7722(5)	0.8932(5)	0.063(6)	0.043(5)	0.072(6)	0.006(4)	−0.010(5)	−0.007(4)
C(36)	4a		0.421(1)	0.7689(5)	0.9440(4)	0.072(6)	0.051(5)	0.062(6)	0.015(5)	0.000(5)	−0.026(4)
C(37)	4a		0.5097(9)	0.7114(4)	0.9497(4)	0.058(5)	0.042(4)	0.055(5)	0.007(4)	−0.003(4)	−0.011(4)
B(1)	4a		−0.030(1)	0.3728(7)	0.7822(6)	0.077(9)	0.061(7)	0.064(7)	0.004(6)	0.005(7)	−0.021(6)
F(1)	4a		−0.0862(8)	0.3312(4)	0.7361(4)	0.141(7)	0.092(5)	0.122(6)	−0.020(5)	−0.032(6)	−0.039(4)
F(2)	4a		0.1036(7)	0.3655(4)	0.7829(4)	0.068(4)	0.142(6)	0.118(5)	0.015(4)	0.001(4)	−0.050(5)
F(3)	4a		−0.0650(7)	0.4460(3)	0.7707(3)	0.123(5)	0.062(3)	0.092(4)	−0.001(4)	0.005(4)	−0.001(3)
F(4)	4a		−0.0801(8)	0.3550(4)	0.8407(3)	0.138(7)	0.134(6)	0.087(5)	0.016(6)	0.041(5)	0.042(5)
C(38)	4a		0.183(2)	0.1965(8)	0.9685(8)	0.19(2)	0.068(8)	0.13(1)	0.02(1)	0.02(1)	0.006(9)
Cl(1)	4a	0.54(2)	0.113(1)	0.2600(3)	0.9119(4)	0.25(1)	0.066(4)	0.100(5)	−0.021(5)	−0.050(7)	0.006(3)
Cl(1')	4a	0.46	0.123(3)	0.274(1)	0.983(2)	0.42(2)	0.27(2)	0.37(3)	0.08(2)	−0.02(2)	0.01(2)
Cl(2)	4a		0.0538(8)	0.1304(3)	0.9708(4)	0.252(8)	0.154(5)	0.240(7)	−0.023(5)	0.044(6)	0.067(5)
Cl(3)	4a	0.35(3)	0.261(4)	0.198(3)	0.9049(9)	0.23(3)	0.50(6)	0.10(1)	−0.17(4)	0.05(1)	0.02(2)
Cl(3')	4a	0.65	0.320(2)	0.1616(8)	0.937(2)	0.28(2)	0.18(1)	0.49(4)	0.08(1)	0.18(2)	−0.07(2)
Cl(4)	4a	0.58(2)	0.207(2)	0.2509(8)	1.0344(5)	0.37(2)	0.25(2)	0.110(7)	0.08(1)	−0.092(9)	−0.057(8)
Cl(4')	4a	0.42	0.261(2)	0.1627(8)	1.0374(6)	0.28(2)	0.16(1)	0.122(9)	−0.01(1)	−0.05(1)	0.036(8)

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