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[4-(Methoxycarbonyl)benzyl]triphenylphosphonium bromide hemihydrate

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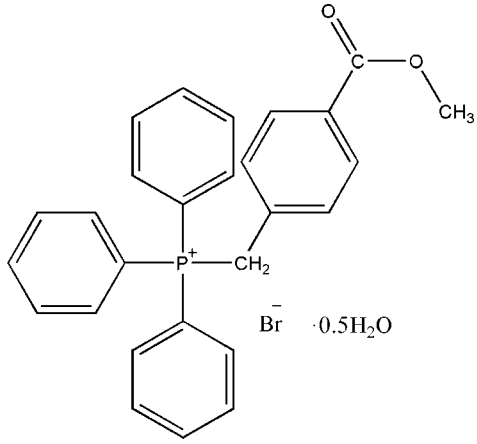
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.080; data-to-parameter ratio = 18.7.

In the crystal structure of the title compound, $C_{27}H_{24}O_2P^+\cdot Br^-\cdot 0.5H_2O$, there are intermolecular O—H···Br hydrogen bonds between the H atoms of the water of crystallization and the bromide anions. The three phenyl rings of the triphenylphosphonium moiety are at angles of 59.73 (15), 79.15 (14) and 82.81 (17)° with the C/P/C planes.

Related literature

For related literature, see: Ahmed *et al.* (1996); Harcken & Martin (2001); Kojima *et al.* (2002); McDonald & Campbell (1959); Nassar *et al.* (2004); Phillips *et al.* (2002); Tanaka *et al.* (2003); Wittig & Schöllkopf (1954).



Experimental

Crystal data

$C_{27}H_{24}O_2P^+\cdot Br^-\cdot 0.5H_2O$
M_r = 500.35
Monoclinic, $C2/c$
a = 21.017 (8) Å
b = 14.045 (5) Å
c = 19.868 (7) Å
β = 126.107 (4)°
V = 4738 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.83 mm^{-1}
T = 123 (2) K
0.40 × 0.31 × 0.22 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
Absorption correction: integration (*ABSCOR*; Higashi, 1999)
T_{min} = 0.404, T_{max} = 0.520
18847 measured reflections
5425 independent reflections
5022 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.079
S = 1.15
5425 reflections
290 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*O*···Br1	1.03 (4)	2.22 (4)	3.2308 (17)	169 (3)

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *TEXSAN*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2361).

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supplementary materials

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[4-(Methoxycarbonyl)benzyl]triphenylphosphonium bromide hemihydrate

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Comment

One of the most useful methods for the synthesis of alkenes with control over the stereoselectivity is the well known Wittig reaction (Wittig & Schöllkopf, 1954). It is mainly due to the stereoselectivity of the Wittig reagent that not only its use in the synthesis (Kojima *et al.*, 2002; Phillips *et al.*, 2002; Harcken & Martin, 2001) has not seen a decline but new and improved methods for its synthesis are being constantly developed (Nassar *et al.*, 2004; Tanaka *et al.*, 2003; Kojima *et al.*, 2002). The title compound, (I), is an intermediate in the synthesis of (*E*)-hydroxyalkyl 4-(4-substituted styryl)benzoate as a part of the project to synthesize ligands for polymeric liquid crystals. Here, we are going to present the crystal structure of the title compound (I). All the geometric parameters are in agreement with the the similar type of studies made by (Ahmed *et al.*, 1996) There are standard electrostatic interactions between the triphenyl-(4-methylcarboxy)benzylphosphonium cations and the bromide anions. It is confirmed that the compound (I) is an ion pair (Fig. 1), with a distance of 4.382 (2)Å between the P^+ and Br^- centres.

Experimental

The triphenyl-(4-methylcarboxy)benzylphosphonium bromide (I) was synthesized following a method reported in the literature (Mcdonald & Campbell, 1959)·A mixture of methyl 4-(bromomethyl)benzoate 2.5 g, 0.01 mol) and triphenylphosphine (2.43 g, 0.01 mol) in 40 ml of toluene was heated under reflux for 3 hr. After cooling to room temperature the salt was filtered, washed with ether and dried under reduced pressure·Yield: 81%, m.p: 245–248°C, R_f = 0.11 (n-Hexane: ethylacetate 7:3). IR (KBr, ν_{max}, cm^{-1}): 3010, 2926, 2830, 1719, 1605, 786. ^{1}H-NMR ($CDCl_3$): δ 3.87 (3*H*, s), 5.65 (2*H*, d, *J* = 15 Hz), 7.70 (17*H*, m), 8.04 (2*H*, d, *J* = 8.1 Hz). ^{13}C-NMR (75 MHz, $CDCl_3$):δ 52.23, 30.58 (d, *J* = 186 Hz), 117.56 (d, *J* = 342 Hz),129.70 (d, *J* = 12 Hz), 129.87 (d, *J* = 18 Hz), 130.165 (d, *J* = 51 Hz), 131.73 (d, *J* = 21 Hz), 132.80 (d, *J* = 33 Hz), 134.45 (d, *J* = 36 Hz), 166.54.

Refinement

H atom on the N atom was refined isotropically. Other H atoms were placed in idealized positions and treated as riding atoms with C—H distance in the range 0.95–0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C).

Figures

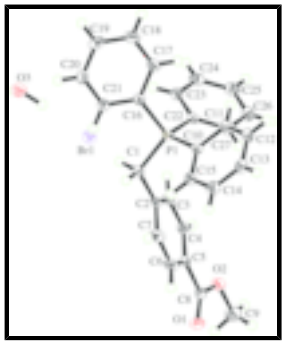


Fig. 1. Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 30% probability level.

[4-(Methoxycarbonyl)benzyl]triphenylphosphonium bromide hemihydrate

Crystal data

$C_{27}H_{24}O_2P^+·Br^-·0.5H_2O$	F_{000} = 2056
M_r = 500.35	D_x = 1.403 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71070 Å
Hall symbol: -C 2yc	Cell parameters from 7012 reflections
a = 21.017 (8) Å	θ = 3.1–27.5º
b = 14.045 (5) Å	μ = 1.83 mm^{-1}
c = 19.868 (7) Å	*T* = 123 (2) K
β = 126.107 (4)º	Block, colorless
V = 4738 (3) Å^3	0.40 × 0.31 × 0.22 mm
Z = 8	

Data collection

Rigaku/MSC Mercury CCD diffractometer	5022 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 123(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.2º
Absorption correction: integration (ABSCOR; Higashi, 1999)	*h* = −27→25
T_{min} = 0.404, T_{max} = 0.520	*k* = −18→17
18847 measured reflections	*l* = −18→25
5425 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H atoms treated by a mixture of

	independent and constrained refinement
$wR(F^2) = 0.079$	$w = 1/[\sigma^2(F_o^2) + (0.0133P)^2 + 10.1118P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} = 0.001$
5425 reflections	$\Delta\rho_{max} = 0.51$ e Å^{-3}
290 parameters	$\Delta\rho_{min} = -0.53$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.31029 (3)	0.44471 (4)	0.32117 (3)	0.01545 (12)
C1	0.41411 (12)	0.41713 (15)	0.38823 (13)	0.0174 (4)
H1A	0.4424	0.4655	0.3789	0.021*
H1B	0.4324	0.4234	0.4468	0.021*
C2	0.43759 (12)	0.32001 (16)	0.37752 (12)	0.0181 (4)
C3	0.42000 (13)	0.23839 (16)	0.40364 (13)	0.0206 (5)
H3	0.3909	0.2438	0.4261	0.025*
C4	0.44456 (13)	0.14960 (17)	0.39714 (14)	0.0222 (5)
H4	0.4314	0.0945	0.4143	0.027*
C5	0.48845 (12)	0.14061 (16)	0.36557 (13)	0.0205 (5)
C6	0.50870 (13)	0.22217 (18)	0.34252 (14)	0.0249 (5)
H6	0.5408	0.2170	0.3235	0.030*
C7	0.48255 (13)	0.31103 (18)	0.34700 (14)	0.0241 (5)
H7	0.4953	0.3660	0.3292	0.029*
C8	0.51658 (13)	0.04628 (18)	0.35787 (14)	0.0260 (5)
O1	0.56546 (10)	0.03619 (14)	0.34485 (11)	0.0341 (4)
O2	0.48183 (10)	−0.02626 (12)	0.36867 (12)	0.0343 (4)
C9	0.50683 (18)	−0.1213 (2)	0.3647 (2)	0.0495 (8)
H9A	0.4922	−0.1329	0.3085	0.074*
H9B	0.4811	−0.1685	0.3776	0.074*
H9C	0.5641	−0.1266	0.4052	0.074*
C10	0.27533 (12)	0.45605 (15)	0.21423 (13)	0.0167 (4)
C11	0.19410 (13)	0.45949 (17)	0.15136 (13)	0.0217 (5)
H11	0.1578	0.4523	0.1648	0.026*

C12	0.16704 (14)	0.47353 (17)	0.06923 (14)	0.0252 (5)
H12	0.1120	0.4762	0.0265	0.030*
C13	0.21980 (14)	0.48367 (16)	0.04915 (14)	0.0240 (5)
H13	0.2008	0.4929	−0.0072	0.029*
C14	0.30000 (14)	0.48039 (17)	0.11111 (14)	0.0243 (5)
H14	0.3361	0.4875	0.0974	0.029*
C15	0.32787 (13)	0.46655 (16)	0.19402 (13)	0.0212 (5)
H15	0.3830	0.4643	0.2366	0.025*
C16	0.29724 (13)	0.55555 (15)	0.35705 (13)	0.0182 (4)
C17	0.22172 (14)	0.58176 (18)	0.33081 (17)	0.0311 (6)
H17	0.1780	0.5419	0.2939	0.037*
C18	0.21096 (16)	0.66619 (19)	0.35887 (18)	0.0363 (6)
H18	0.1596	0.6842	0.3409	0.044*
C19	0.27431 (15)	0.72433 (17)	0.41278 (15)	0.0280 (5)
H19	0.2665	0.7816	0.4325	0.034*
C20	0.34886 (14)	0.69949 (17)	0.43800 (14)	0.0253 (5)
H20	0.3921	0.7402	0.4744	0.030*
C21	0.36103 (13)	0.61531 (17)	0.41050 (13)	0.0221 (5)
H21	0.4124	0.5984	0.4279	0.027*
C22	0.25177 (12)	0.35735 (15)	0.32766 (13)	0.0171 (4)
C23	0.24544 (13)	0.36231 (17)	0.39383 (14)	0.0223 (5)
H23	0.2734	0.4097	0.4356	0.027*
C24	0.19816 (14)	0.29762 (18)	0.39790 (15)	0.0265 (5)
H24	0.1933	0.3011	0.4424	0.032*
C25	0.15793 (14)	0.22790 (18)	0.33766 (16)	0.0281 (5)
H25	0.1248	0.1846	0.3404	0.034*
C26	0.16576 (15)	0.22106 (18)	0.27322 (16)	0.0287 (5)
H26	0.1388	0.1725	0.2324	0.034*
C27	0.21322 (13)	0.28556 (16)	0.26844 (14)	0.0230 (5)
H27	0.2193	0.2806	0.2248	0.028*
Br1	0.456153 (14)	0.381335 (18)	0.588418 (14)	0.02564 (7)
O3	0.5000	0.49982 (19)	0.7500	0.0300 (5)
H3O	0.485 (2)	0.455 (3)	0.702 (2)	0.090 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0136 (2)	0.0170 (3)	0.0155 (2)	−0.0005 (2)	0.0084 (2)	0.0006 (2)
C1	0.0142 (9)	0.0186 (10)	0.0169 (10)	−0.0006 (8)	0.0077 (8)	0.0010 (8)
C2	0.0129 (9)	0.0227 (11)	0.0153 (9)	0.0020 (8)	0.0064 (8)	0.0026 (8)
C3	0.0206 (11)	0.0213 (11)	0.0230 (11)	−0.0003 (9)	0.0146 (9)	0.0007 (9)
C4	0.0208 (11)	0.0208 (11)	0.0260 (11)	−0.0012 (9)	0.0144 (10)	−0.0006 (9)
C5	0.0146 (10)	0.0261 (12)	0.0147 (10)	0.0029 (8)	0.0053 (8)	−0.0020 (8)
C6	0.0200 (11)	0.0386 (14)	0.0201 (10)	0.0080 (10)	0.0140 (9)	0.0060 (10)
C7	0.0208 (11)	0.0294 (13)	0.0227 (11)	0.0054 (10)	0.0132 (9)	0.0101 (10)
C8	0.0173 (11)	0.0318 (13)	0.0184 (11)	0.0062 (10)	0.0047 (9)	−0.0048 (9)
O1	0.0263 (9)	0.0428 (11)	0.0333 (9)	0.0090 (8)	0.0176 (8)	−0.0058 (8)
O2	0.0256 (9)	0.0222 (9)	0.0494 (11)	0.0018 (7)	0.0189 (9)	−0.0084 (8)

C9	0.0380 (16)	0.0257 (14)	0.078 (2)	0.0020 (12)	0.0304 (16)	−0.0167 (15)
C10	0.0177 (10)	0.0144 (10)	0.0174 (10)	0.0010 (8)	0.0100 (9)	0.0024 (8)
C11	0.0197 (11)	0.0266 (12)	0.0195 (10)	0.0039 (9)	0.0121 (9)	0.0022 (9)
C12	0.0204 (11)	0.0298 (13)	0.0185 (10)	0.0031 (10)	0.0077 (9)	0.0019 (9)
C13	0.0305 (12)	0.0228 (12)	0.0183 (10)	0.0019 (10)	0.0143 (10)	0.0027 (9)
C14	0.0291 (12)	0.0274 (12)	0.0231 (11)	−0.0023 (10)	0.0190 (10)	0.0021 (9)
C15	0.0185 (10)	0.0236 (12)	0.0204 (10)	−0.0018 (9)	0.0109 (9)	0.0011 (9)
C16	0.0186 (10)	0.0171 (10)	0.0188 (10)	0.0007 (8)	0.0110 (9)	0.0012 (8)
C17	0.0184 (11)	0.0266 (13)	0.0435 (15)	−0.0029 (10)	0.0156 (11)	−0.0082 (11)
C18	0.0274 (13)	0.0282 (14)	0.0588 (18)	0.0024 (11)	0.0283 (13)	−0.0050 (12)
C19	0.0387 (14)	0.0191 (12)	0.0342 (13)	0.0025 (10)	0.0258 (12)	−0.0011 (10)
C20	0.0260 (12)	0.0220 (12)	0.0203 (11)	−0.0020 (9)	0.0094 (10)	−0.0039 (9)
C21	0.0181 (10)	0.0233 (11)	0.0205 (10)	0.0016 (9)	0.0088 (9)	−0.0008 (9)
C22	0.0144 (10)	0.0180 (11)	0.0182 (10)	−0.0003 (8)	0.0092 (8)	0.0013 (8)
C23	0.0215 (11)	0.0256 (12)	0.0212 (11)	−0.0001 (9)	0.0134 (9)	0.0005 (9)
C24	0.0284 (12)	0.0319 (13)	0.0268 (12)	0.0044 (10)	0.0205 (10)	0.0072 (10)
C25	0.0282 (12)	0.0236 (12)	0.0413 (14)	−0.0011 (10)	0.0255 (12)	0.0058 (10)
C26	0.0308 (13)	0.0243 (12)	0.0350 (13)	−0.0084 (10)	0.0217 (11)	−0.0059 (10)
C27	0.0258 (12)	0.0223 (12)	0.0238 (11)	−0.0034 (9)	0.0162 (10)	−0.0020 (9)
Br1	0.02436 (12)	0.03282 (14)	0.02411 (12)	−0.00150 (10)	0.01670 (10)	0.00167 (10)
O3	0.0276 (13)	0.0313 (14)	0.0331 (13)	0.000	0.0190 (11)	0.000

Geometric parameters (Å, °)

P1—C22	1.795 (2)	C12—H12	0.9500
P1—C16	1.799 (2)	C13—C14	1.383 (3)
P1—C10	1.800 (2)	C13—H13	0.9500
P1—C1	1.806 (2)	C14—C15	1.399 (3)
C1—C2	1.508 (3)	C14—H14	0.9500
C1—H1A	0.9900	C15—H15	0.9500
C1—H1B	0.9900	C16—C17	1.397 (3)
C2—C3	1.395 (3)	C16—C21	1.397 (3)
C2—C7	1.397 (3)	C17—C18	1.384 (4)
C3—C4	1.385 (3)	C17—H17	0.9500
C3—H3	0.9500	C18—C19	1.381 (4)
C4—C5	1.393 (3)	C18—H18	0.9500
C4—H4	0.9500	C19—C20	1.379 (4)
C5—C6	1.390 (3)	C19—H19	0.9500
C5—C8	1.495 (3)	C20—C21	1.388 (3)
C6—C7	1.388 (3)	C20—H20	0.9500
C6—H6	0.9500	C21—H21	0.9500
C7—H7	0.9500	C22—C27	1.391 (3)
C8—O1	1.207 (3)	C22—C23	1.399 (3)
C8—O2	1.343 (3)	C23—C24	1.384 (3)
O2—C9	1.454 (3)	C23—H23	0.9500
C9—H9A	0.9800	C24—C25	1.383 (4)
C9—H9B	0.9800	C24—H24	0.9500
C9—H9C	0.9800	C25—C26	1.387 (4)
C10—C15	1.388 (3)	C25—H25	0.9500

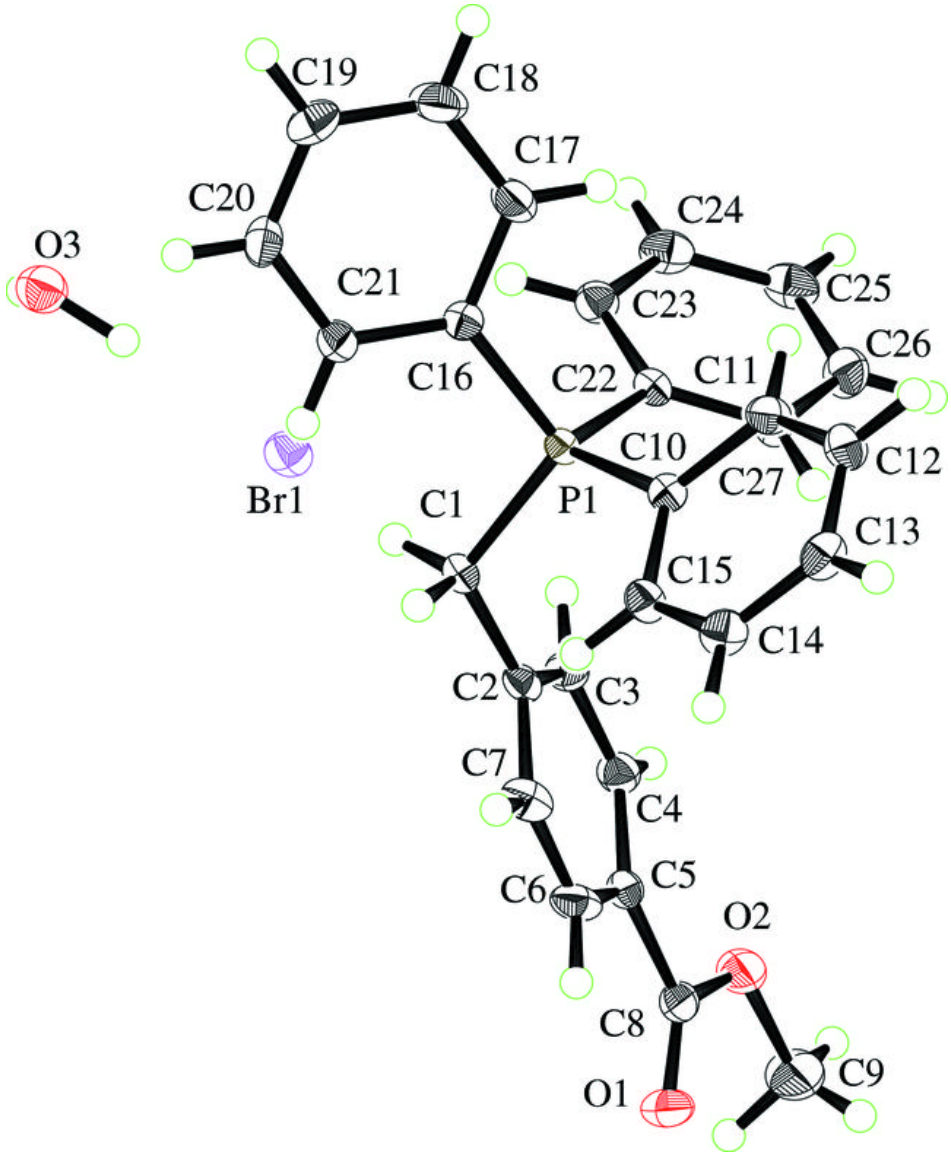
C10—C11	1.401 (3)	C26—C27	1.392 (3)
C11—C12	1.390 (3)	C26—H26	0.9500
C11—H11	0.9500	C27—H27	0.9500
C12—C13	1.389 (3)	O3—H3O	1.03 (4)
C22—P1—C16	107.00 (11)	C13—C12—H12	119.7
C22—P1—C10	108.96 (10)	C11—C12—H12	119.7
C16—P1—C10	109.85 (10)	C14—C13—C12	120.1 (2)
C22—P1—C1	112.43 (10)	C14—C13—H13	120.0
C16—P1—C1	107.06 (10)	C12—C13—H13	120.0
C10—P1—C1	111.41 (10)	C13—C14—C15	119.8 (2)
C2—C1—P1	116.34 (15)	C13—C14—H14	120.1
C2—C1—H1A	108.2	C15—C14—H14	120.1
P1—C1—H1A	108.2	C10—C15—C14	120.2 (2)
C2—C1—H1B	108.2	C10—C15—H15	119.9
P1—C1—H1B	108.2	C14—C15—H15	119.9
H1A—C1—H1B	107.4	C17—C16—C21	119.7 (2)
C3—C2—C7	118.9 (2)	C17—C16—P1	119.02 (17)
C3—C2—C1	120.7 (2)	C21—C16—P1	121.27 (17)
C7—C2—C1	120.3 (2)	C18—C17—C16	119.7 (2)
C4—C3—C2	120.7 (2)	C18—C17—H17	120.2
C4—C3—H3	119.7	C16—C17—H17	120.2
C2—C3—H3	119.7	C19—C18—C17	120.5 (2)
C3—C4—C5	120.4 (2)	C19—C18—H18	119.8
C3—C4—H4	119.8	C17—C18—H18	119.8
C5—C4—H4	119.8	C20—C19—C18	120.1 (2)
C6—C5—C4	119.1 (2)	C20—C19—H19	119.9
C6—C5—C8	118.7 (2)	C18—C19—H19	119.9
C4—C5—C8	122.2 (2)	C19—C20—C21	120.4 (2)
C7—C6—C5	120.7 (2)	C19—C20—H20	119.8
C7—C6—H6	119.7	C21—C20—H20	119.8
C5—C6—H6	119.7	C20—C21—C16	119.6 (2)
C6—C7—C2	120.2 (2)	C20—C21—H21	120.2
C6—C7—H7	119.9	C16—C21—H21	120.2
C2—C7—H7	119.9	C27—C22—C23	120.0 (2)
O1—C8—O2	123.9 (2)	C27—C22—P1	121.14 (17)
O1—C8—C5	124.3 (2)	C23—C22—P1	118.82 (16)
O2—C8—C5	111.8 (2)	C24—C23—C22	119.4 (2)
C8—O2—C9	116.1 (2)	C24—C23—H23	120.3
O2—C9—H9A	109.5	C22—C23—H23	120.3
O2—C9—H9B	109.5	C25—C24—C23	120.5 (2)
H9A—C9—H9B	109.5	C25—C24—H24	119.7
O2—C9—H9C	109.5	C23—C24—H24	119.7
H9A—C9—H9C	109.5	C24—C25—C26	120.2 (2)
H9B—C9—H9C	109.5	C24—C25—H25	119.9
C15—C10—C11	119.9 (2)	C26—C25—H25	119.9
C15—C10—P1	120.68 (16)	C25—C26—C27	119.8 (2)
C11—C10—P1	119.38 (17)	C25—C26—H26	120.1
C12—C11—C10	119.5 (2)	C27—C26—H26	120.1
C12—C11—H11	120.3	C22—C27—C26	119.9 (2)

C10—C11—H11	120.3	C22—C27—H27	120.1
C13—C12—C11	120.5 (2)	C26—C27—H27	120.1
C22—P1—C1—C2	54.37 (19)	C11—C10—C15—C14	−0.1 (3)
C16—P1—C1—C2	171.59 (16)	P1—C10—C15—C14	−176.59 (18)
C10—P1—C1—C2	−68.29 (19)	C13—C14—C15—C10	0.0 (4)
P1—C1—C2—C3	−71.4 (2)	C22—P1—C16—C17	−43.1 (2)
P1—C1—C2—C7	113.4 (2)	C10—P1—C16—C17	75.1 (2)
C7—C2—C3—C4	−1.9 (3)	C1—P1—C16—C17	−163.83 (19)
C1—C2—C3—C4	−177.2 (2)	C22—P1—C16—C21	136.78 (19)
C2—C3—C4—C5	1.1 (3)	C10—P1—C16—C21	−105.09 (19)
C3—C4—C5—C6	1.5 (3)	C1—P1—C16—C21	16.0 (2)
C3—C4—C5—C8	179.7 (2)	C21—C16—C17—C18	−0.8 (4)
C4—C5—C6—C7	−3.1 (3)	P1—C16—C17—C18	179.1 (2)
C8—C5—C6—C7	178.7 (2)	C16—C17—C18—C19	−0.3 (4)
C5—C6—C7—C2	2.2 (3)	C17—C18—C19—C20	1.2 (4)
C3—C2—C7—C6	0.3 (3)	C18—C19—C20—C21	−1.0 (4)
C1—C2—C7—C6	175.6 (2)	C19—C20—C21—C16	−0.1 (4)
C6—C5—C8—O1	11.6 (3)	C17—C16—C21—C20	1.0 (3)
C4—C5—C8—O1	−166.6 (2)	P1—C16—C21—C20	−178.90 (17)
C6—C5—C8—O2	−169.47 (19)	C16—P1—C22—C27	144.04 (18)
C4—C5—C8—O2	12.3 (3)	C10—P1—C22—C27	25.3 (2)
O1—C8—O2—C9	0.5 (3)	C1—P1—C22—C27	−98.69 (19)
C5—C8—O2—C9	−178.4 (2)	C16—P1—C22—C23	−36.1 (2)
C22—P1—C10—C15	−140.05 (18)	C10—P1—C22—C23	−154.78 (17)
C16—P1—C10—C15	103.04 (19)	C1—P1—C22—C23	81.2 (2)
C1—P1—C10—C15	−15.4 (2)	C27—C22—C23—C24	−2.5 (3)
C22—P1—C10—C11	43.5 (2)	P1—C22—C23—C24	177.58 (17)
C16—P1—C10—C11	−73.5 (2)	C22—C23—C24—C25	0.6 (4)
C1—P1—C10—C11	168.08 (17)	C23—C24—C25—C26	1.2 (4)
C15—C10—C11—C12	−0.1 (3)	C24—C25—C26—C27	−1.1 (4)
P1—C10—C11—C12	176.46 (18)	C23—C22—C27—C26	2.6 (3)
C10—C11—C12—C13	0.3 (4)	P1—C22—C27—C26	−177.47 (18)
C11—C12—C13—C14	−0.4 (4)	C25—C26—C27—C22	−0.8 (4)
C12—C13—C14—C15	0.2 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3O···Br1	1.03 (4)	2.22 (4)	3.2308 (17)	169 (3)

Fig. 1


C19
C18
C17
C20
C24
O3
C21
C23
C25
C26
C16
C22
C11
C10
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Br1
C1
P1
C27
C13
C15
C14
C2
C3
C7
C4
C5
C6
O2
C8
C9
O1