organic compounds

(4-Carboxybutyl)triphenylphosphonium bromide

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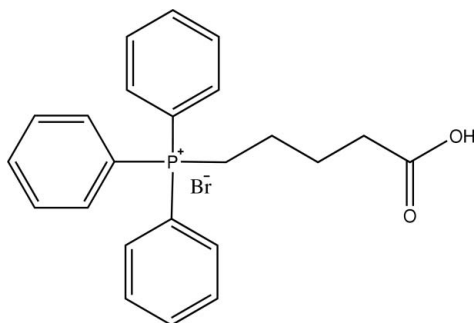Correspondence e-mail: fangshi.li@njut.edu.cn

The asymmetric unit of the title triphenylphosphonium salt, $C_{23}H_{24}O_2P^+\cdot Br^-$, contains two cations and two anions. In the crystal structure, O—H···Br, C—H···O and C—H···Br hydrogen bonds link the ions.

Related literature

For related literature, see: Mashraqui *et al.* (2004); Ishikawa & Manabe (2006). For bond-length data, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_{23}H_{24}O_2P^+\cdot Br^-$	γ = 81.07 (3)°
M_r = 443.29	V = 2135.1 (8) Å³
Triclinic, $P\bar{1}$	Z = 4
a = 9.2690 (19) Å	Mo $K\alpha$ radiation
b = 13.674 (3) Å	μ = 2.02 mm⁻¹
c = 17.075 (3) Å	T = 294 (2) K
α = 88.91 (3)°	0.30 × 0.20 × 0.10 mm
β = 87.15 (3)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	8367 independent reflections
Absorption correction: ψ scan (North *et al.*, 1968)	3700 reflections with $I > 2\sigma(I)$
T_{min} = 0.583, T_{max} = 0.824	R_{int} = 0.055
8913 measured reflections	3 standard reflections frequency: 120 min intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.084	121 restraints
$wR(F^2)$ = 0.196	H-atom parameters constrained
S = 0.99	$\Delta\rho_{max}$ = 0.72 e Å⁻³
8367 reflections	$\Delta\rho_{min}$ = −0.60 e Å⁻³
475 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···Br1[i]	0.82	2.35	3.148 (7)	165
O4—H4B···Br2[ii]	0.82	2.41	3.199 (7)	163
C14—H14A···O1[iii]	0.93	2.58	3.357 (10)	141
C17—H17A···Br2[iv]	0.93	2.77	3.691 (7)	169
C19—H19A···Br2[iv]	0.97	2.88	3.790 (7)	157
C19—H19B···O1[i]	0.97	2.41	3.351 (10)	163
C33—H33A···Br2	0.93	2.79	3.623 (9)	150
C37—H37A···Br1[v]	0.93	2.82	3.574 (8)	139

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $-x, -y+1, -z+1$; (iii) $-x+3, -y+2, -z+1$; (iv) $x+1, y+1, z$; (v) $x-1, y, z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2349).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Ishikawa, S. & Manabe, K. (2006). *J. Chem. Soc. Chem. Commun.* pp. 2589–2591.
Mashraqui, S. H., Kenny, R. S., Ghadigaonkar, S. G., Krishnan, A. & Bhattacharya, M. (2004). *Opt. Mater.* **27**, 257–260.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1996). *SHELXTL*. Version 5.06. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

(4-Carboxybutyl)triphenylphosphonium bromide

D.-Y. Wu, F.-S. Li, J.-Y. Xia, N.-W. Mao and H.-L. Yao

Comment

Triphenylphosphoniums are an important class of isoaromatic compounds and have widespread applications in pharmaceuticals (Mashraqui *et al.*, 2004; Ishikawa & Manabe, 2006). As part of our ongoing studies in this area, we report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The asymmetric unit contains two independent molecules. Rings A (C1–C6), B (C7–C12), C (C13–C18), D (C24–C29), E (C30–C35) and F (C36–C41) are, of course, planar and the dihedral angles between them are A/B = 79.30 (3)°, A/C = 87.03 (3)°, B/C = 63.74 (2)°, D/E = 61.30 (2)°, D/F = 70.17 (3)° and E/F = 84.32 (3)°.

In the crystal structure, O—H···Br, C—H···O and C—H···Br hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they seem to be effective in the stabilization of the structure.

Experimental

For the preparation of the title copound, (I), a mixture of 5-bromovaleric acid (2.4613 g, 9 mmol) and triphenylphosphine (3.6076 g, 9 mmol) was heated under reflux in acetonitrile (100 ml) for 24 h. The resulting salt was recrystallized from acetonitrile, washed with ether, and dried in vacuum to give the title compound (yield; 2.5994 g). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Refinement

H atoms were positioned geometrically, with O—H = 0.82 Å (for OH) and C—H = 0.93 and 0.97 Å for aromatic and methylene H, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,O)$, where x = 1.5 for OH H, and x = 1.2 for all other H atoms.
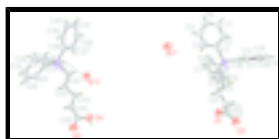
Figures

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines.

supplementary materials
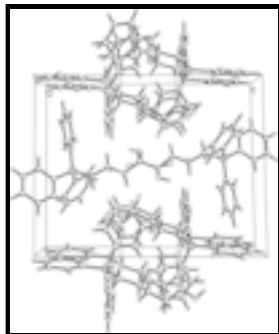
Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

(4-Carboxybutyl)triphenylphosphonium bromide

Crystal data

$C_{23}H_{24}O_2P^+ \cdot Br^-$	$Z = 4$
$M_r = 443.29$	$F_{000} = 912$
Triclinic, $P\bar{1}$	$D_x = 1.379$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.2690$ (19) Å	Cell parameters from 25 reflections
$b = 13.674$ (3) Å	$\theta = 9–13°$
$c = 17.075$ (3) Å	$\mu = 2.02$ mm^{-1}
$\alpha = 88.91$ (3)°	$T = 294$ (2) K
$\beta = 87.15$ (3)°	Block, colourless
$\gamma = 81.07$ (3)°	$0.30 \times 0.20 \times 0.10$ mm
$V = 2135.1$ (8) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.055$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0°$
Monochromator: graphite	$\theta_{min} = 1.2°$
$T = 294(2)$ K	$h = -11 \rightarrow 11$
$\omega/2\theta$ scans	$k = -16 \rightarrow 16$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 21$
$T_{min} = 0.583$, $T_{max} = 0.824$	3 standard reflections
8913 measured reflections	every 120 min
8367 independent reflections	intensity decay: none
3700 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.084$

$wR(F^2) = 0.196$

$S = 0.99$

8367 reflections

475 parameters

121 restraints

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 8P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.72$ e Å^{-3}

$\Delta\rho_{min} = -0.60$ e Å^{-3}

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 > 2sigma(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}
Br1	0.75038 (14)	0.91104 (8)	0.16509 (6)	0.0872 (4)
P1	1.2962 (2)	1.04654 (14)	0.31701 (12)	0.0425 (5)
O1	1.0624 (8)	1.0797 (4)	0.6324 (4)	0.080 (2)
O2	1.1257 (8)	1.2101 (4)	0.6878 (4)	0.086 (2)
H2B	1.1520	1.1693	0.7221	0.129*
C1	1.3356 (12)	1.0358 (8)	0.0821 (6)	0.091 (3)
H1A	1.4095	1.0416	0.0442	0.110*
C2	1.3601 (11)	1.0448 (7)	0.1601 (6)	0.077 (3)
H2A	1.4507	1.0583	0.1740	0.093*
C3	1.2581 (9)	1.0349 (6)	0.2169 (5)	0.048 (2)
C4	1.1218 (9)	1.0171 (6)	0.1935 (5)	0.055 (2)
H4A	1.0466	1.0127	0.2309	0.066*
C5	1.0996 (11)	1.0062 (7)	0.1173 (6)	0.077 (3)
H5A	1.0105	0.9899	0.1036	0.092*
C6	1.2000 (12)	1.0179 (7)	0.0602 (6)	0.080 (3)
H6A	1.1795	1.0142	0.0076	0.096*
C7	1.3757 (12)	0.7501 (7)	0.3593 (6)	0.084 (3)
H7A	1.3615	0.6925	0.3346	0.101*
C8	1.3386 (10)	0.8424 (7)	0.3196 (6)	0.071
H8A	1.3086	0.8453	0.2683	0.085*
C9	1.3489 (8)	0.9279 (5)	0.3602 (5)	0.0442 (19)
C10	1.4013 (10)	0.9180 (6)	0.4333 (5)	0.063 (2)
H10A	1.4105	0.9749	0.4605	0.075*

C11	1.4414 (10)	0.8272 (7)	0.4686 (5)	0.067 (2)
H11A	1.4764	0.8237	0.5189	0.080*
C12	1.4300 (10)	0.7428 (7)	0.4303 (6)	0.069 (3)
H12A	1.4595	0.6812	0.4532	0.083*
C13	1.6691 (9)	1.2263 (6)	0.3333 (5)	0.053 (2)
H13A	1.7452	1.2627	0.3374	0.063*
C14	1.6956 (9)	1.1236 (6)	0.3317 (5)	0.055 (2)
H14A	1.7912	1.0909	0.3332	0.066*
C15	1.5840 (9)	1.0697 (6)	0.3279 (5)	0.048 (2)
H15A	1.6044	1.0009	0.3277	0.057*
C16	1.4439 (8)	1.1154 (5)	0.3244 (4)	0.0404 (19)
C17	1.4149 (8)	1.2191 (5)	0.3242 (5)	0.048 (2)
H17A	1.3194	1.2515	0.3209	0.058*
C18	1.5273 (9)	1.2726 (6)	0.3287 (5)	0.052 (2)
H18A	1.5072	1.3414	0.3287	0.062*
C19	1.1305 (8)	1.1083 (5)	0.3668 (5)	0.046 (2)
H19A	1.0938	1.1670	0.3367	0.055*
H19B	1.0574	1.0646	0.3672	0.055*
C20	1.1476 (8)	1.1391 (6)	0.4518 (5)	0.053 (2)
H20A	1.1855	1.0813	0.4826	0.064*
H20B	1.2169	1.1854	0.4522	0.064*
C21	1.0021 (9)	1.1868 (6)	0.4880 (5)	0.061 (2)
H21A	0.9393	1.1367	0.4969	0.073*
H21B	0.9553	1.2358	0.4518	0.073*
C22	1.0196 (10)	1.2365 (6)	0.5659 (5)	0.061 (2)
H22A	1.0889	1.2825	0.5573	0.073*
H22B	0.9263	1.2746	0.5826	0.073*
C23	1.0709 (9)	1.1661 (6)	0.6308 (5)	0.052 (2)
Br2	0.05878 (11)	0.37271 (7)	0.29062 (6)	0.0675 (3)
P2	−0.3584 (2)	0.53719 (15)	0.15327 (12)	0.0433 (5)
O3	−0.3181 (9)	0.4922 (6)	0.5941 (4)	0.106 (3)
O4	−0.1554 (8)	0.5727 (5)	0.5404 (4)	0.090 (2)
H4B	−0.1504	0.5891	0.5860	0.135*
C24	−0.8304 (12)	0.6946 (9)	0.1678 (7)	0.083 (3)
H24A	−0.9253	0.7288	0.1720	0.100*
C25	−0.7298 (13)	0.7270 (8)	0.1165 (6)	0.087 (3)
H25A	−0.7584	0.7824	0.0852	0.105*
C26	−0.5902 (11)	0.6808 (7)	0.1098 (5)	0.070 (3)
H26A	−0.5242	0.7037	0.0737	0.083*
C27	−0.5446 (9)	0.5969 (6)	0.1581 (5)	0.046 (2)
C28	−0.6490 (10)	0.5632 (6)	0.2094 (5)	0.057 (2)
H28A	−0.6225	0.5078	0.2410	0.068*
C29	−0.7921 (11)	0.6120 (8)	0.2133 (6)	0.070 (3)
H29A	−0.8617	0.5887	0.2467	0.084*
C30	−0.4604 (11)	0.2666 (7)	0.1041 (6)	0.076 (3)
H30A	−0.5396	0.2407	0.0862	0.091*
C31	−0.4702 (10)	0.3675 (6)	0.1175 (5)	0.062 (2)
H31A	−0.5568	0.4095	0.1081	0.074*
C32	−0.3532 (8)	0.4058 (5)	0.1444 (4)	0.0377 (18)

C33	−0.2246 (9)	0.3432 (6)	0.1582 (5)	0.051 (2)
H33A	−0.1451	0.3684	0.1765	0.061*
C34	−0.2146 (10)	0.2429 (6)	0.1447 (6)	0.065 (3)
H34A	−0.1283	0.2007	0.1540	0.078*
C35	−0.3312 (11)	0.2059 (6)	0.1178 (5)	0.065 (3)
H35A	−0.3231	0.1384	0.1086	0.078*
C36	−0.1545 (11)	0.7136 (7)	0.0082 (6)	0.073 (3)
H36A	−0.1283	0.7764	0.0099	0.088*
C37	−0.2211 (10)	0.6756 (6)	0.0734 (5)	0.061 (2)
H37A	−0.2398	0.7129	0.1188	0.074*
C38	−0.2603 (8)	0.5817 (5)	0.0716 (4)	0.044 (2)
C39	−0.2274 (8)	0.5262 (6)	0.0050 (5)	0.052 (2)
H39A	−0.2516	0.4628	0.0032	0.062*
C40	−0.1573 (9)	0.5654 (7)	−0.0604 (5)	0.061 (2)
H40A	−0.1317	0.5271	−0.1048	0.074*
C41	−0.1266 (10)	0.6601 (8)	−0.0586 (6)	0.070 (3)
H41A	−0.0865	0.6879	−0.1031	0.084*
C42	−0.2689 (9)	0.5638 (6)	0.2411 (4)	0.047 (2)
H42A	−0.1644	0.5434	0.2323	0.056*
H42B	−0.2855	0.6349	0.2485	0.056*
C43	−0.3169 (8)	0.5153 (5)	0.3154 (4)	0.038
H43A	−0.2911	0.4441	0.3106	0.045*
H43B	−0.4224	0.5304	0.3222	0.045*
C44	−0.2487 (9)	0.5481 (5)	0.3879 (4)	0.046 (2)
H44A	−0.1439	0.5438	0.3783	0.055*
H44B	−0.2887	0.6165	0.3992	0.055*
C45	−0.2796 (10)	0.4834 (6)	0.4574 (4)	0.054 (2)
H45A	−0.3809	0.4736	0.4563	0.065*
H45B	−0.2199	0.4192	0.4498	0.065*
C46	−0.2555 (11)	0.5170 (7)	0.5363 (5)	0.062 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1248 (10)	0.0621 (7)	0.0723 (7)	−0.0051 (6)	−0.0086 (7)	−0.0098 (5)
P1	0.0439 (13)	0.0341 (11)	0.0534 (13)	−0.0164 (9)	−0.0077 (10)	−0.0008 (10)
O1	0.118 (6)	0.041 (4)	0.091 (5)	−0.040 (4)	−0.014 (4)	0.011 (3)
O2	0.144 (7)	0.047 (4)	0.072 (5)	−0.026 (4)	−0.016 (4)	−0.005 (3)
C1	0.105 (6)	0.107 (7)	0.066 (5)	−0.033 (6)	0.006 (5)	0.000 (5)
C2	0.073 (5)	0.096 (6)	0.069 (5)	−0.032 (5)	0.000 (4)	−0.017 (5)
C3	0.051 (4)	0.047 (4)	0.051 (4)	−0.022 (4)	−0.002 (3)	0.000 (4)
C4	0.047 (4)	0.061 (5)	0.057 (4)	−0.007 (4)	−0.003 (4)	−0.011 (4)
C5	0.078 (6)	0.081 (6)	0.073 (5)	−0.010 (5)	−0.021 (4)	−0.017 (5)
C6	0.108 (6)	0.076 (6)	0.058 (5)	−0.017 (5)	−0.014 (4)	−0.005 (5)
C7	0.116 (7)	0.050 (4)	0.089 (6)	−0.013 (5)	−0.019 (5)	−0.013 (4)
C8	0.071	0.071	0.071	−0.011	−0.003	−0.001
C9	0.045 (4)	0.038 (4)	0.052 (4)	−0.009 (3)	−0.009 (4)	−0.003 (3)
C10	0.083 (6)	0.049 (4)	0.058 (5)	−0.010 (4)	−0.018 (4)	−0.001 (4)

C11	0.084 (6)	0.063 (5)	0.052 (5)	−0.008 (5)	−0.005 (4)	0.006 (4)
C12	0.080 (6)	0.050 (4)	0.079 (5)	−0.013 (4)	−0.004 (5)	0.010 (4)
C13	0.042 (5)	0.058 (6)	0.064 (6)	−0.024 (4)	−0.012 (4)	0.010 (4)
C14	0.040 (5)	0.047 (5)	0.080 (6)	−0.010 (4)	−0.011 (4)	−0.005 (5)
C15	0.047 (5)	0.032 (4)	0.068 (6)	−0.014 (4)	−0.003 (4)	−0.004 (4)
C16	0.043 (5)	0.035 (4)	0.050 (5)	−0.024 (4)	−0.007 (4)	−0.001 (4)
C17	0.033 (5)	0.037 (5)	0.077 (6)	−0.014 (4)	−0.003 (4)	0.000 (4)
C18	0.059 (6)	0.044 (5)	0.060 (6)	−0.031 (4)	−0.010 (4)	0.005 (4)
C19	0.049 (5)	0.026 (4)	0.068 (6)	−0.017 (4)	−0.014 (4)	0.000 (4)
C20	0.042 (5)	0.054 (5)	0.066 (6)	−0.018 (4)	0.010 (4)	−0.003 (4)
C21	0.057 (6)	0.059 (6)	0.064 (6)	−0.004 (5)	0.002 (5)	0.000 (5)
C22	0.073 (7)	0.051 (6)	0.060 (6)	−0.010 (5)	−0.004 (5)	0.005 (5)
C23	0.063 (6)	0.045 (5)	0.048 (5)	−0.009 (4)	0.002 (4)	−0.007 (4)
Br2	0.0661 (7)	0.0614 (6)	0.0733 (7)	0.0044 (5)	−0.0272 (5)	−0.0093 (5)
P2	0.0507 (14)	0.0355 (12)	0.0465 (13)	−0.0130 (10)	−0.0120 (10)	0.0004 (9)
O3	0.145 (6)	0.117 (6)	0.068 (4)	−0.063 (5)	0.000 (4)	−0.001 (4)
O4	0.109 (5)	0.115 (5)	0.059 (4)	−0.059 (4)	−0.008 (4)	−0.004 (4)
C24	0.074 (8)	0.087 (9)	0.087 (8)	−0.001 (7)	−0.018 (7)	−0.025 (7)
C25	0.083 (9)	0.075 (8)	0.098 (9)	0.003 (7)	0.000 (7)	0.010 (7)
C26	0.071 (7)	0.064 (6)	0.069 (7)	−0.003 (5)	0.009 (5)	0.018 (5)
C27	0.058 (5)	0.040 (5)	0.045 (5)	−0.019 (4)	−0.011 (4)	−0.001 (4)
C28	0.059 (6)	0.055 (6)	0.061 (6)	−0.017 (5)	−0.021 (5)	0.000 (5)
C29	0.055 (7)	0.074 (7)	0.085 (8)	−0.023 (5)	0.006 (5)	−0.019 (6)
C30	0.063 (7)	0.057 (6)	0.114 (9)	−0.024 (5)	−0.019 (6)	−0.013 (6)
C31	0.058 (6)	0.045 (5)	0.084 (7)	−0.007 (4)	−0.022 (5)	−0.002 (5)
C32	0.043 (5)	0.028 (4)	0.042 (4)	−0.008 (3)	−0.007 (4)	0.004 (3)
C33	0.041 (5)	0.041 (5)	0.073 (6)	−0.013 (4)	−0.008 (4)	−0.005 (4)
C34	0.057 (6)	0.041 (5)	0.097 (8)	−0.005 (4)	−0.003 (5)	−0.002 (5)
C35	0.084 (7)	0.038 (5)	0.076 (7)	−0.017 (5)	−0.001 (6)	0.002 (5)
C36	0.089 (8)	0.049 (6)	0.083 (8)	−0.022 (5)	0.005 (6)	0.009 (6)
C37	0.093 (7)	0.045 (5)	0.049 (5)	−0.018 (5)	−0.011 (5)	−0.001 (4)
C38	0.057 (5)	0.033 (4)	0.043 (5)	−0.006 (4)	−0.012 (4)	0.002 (4)
C39	0.045 (5)	0.056 (6)	0.056 (6)	−0.014 (4)	−0.004 (4)	0.004 (5)
C40	0.054 (6)	0.080 (7)	0.048 (5)	−0.007 (5)	0.004 (4)	−0.014 (5)
C41	0.067 (7)	0.072 (7)	0.076 (7)	−0.031 (6)	−0.002 (5)	0.019 (6)
C42	0.052 (5)	0.039 (5)	0.052 (5)	−0.011 (4)	−0.007 (4)	0.001 (4)
C43	0.038	0.038	0.038	−0.006	−0.002	0.000
C44	0.052 (5)	0.033 (4)	0.053 (5)	−0.005 (4)	−0.010 (4)	−0.011 (4)
C45	0.070 (6)	0.047 (5)	0.046 (5)	−0.011 (4)	−0.007 (4)	−0.005 (4)
C46	0.076 (6)	0.066 (5)	0.048 (5)	−0.022 (4)	−0.001 (4)	0.002 (4)

Geometric parameters (Å, °)

P1—C9	1.777 (8)	P2—C38	1.776 (8)
P1—C3	1.778 (8)	P2—C27	1.789 (8)
P1—C16	1.788 (7)	P2—C32	1.798 (7)
P1—C19	1.814 (8)	P2—C42	1.818 (7)
O1—C23	1.196 (9)	O3—C46	1.191 (10)
O2—C23	1.317 (9)	O4—C46	1.294 (10)

O2—H2B	0.8200	O4—H4B	0.8200
C1—C2	1.373 (13)	C24—C25	1.366 (14)
C1—C6	1.388 (11)	C24—C29	1.368 (13)
C1—H1A	0.9300	C24—H24A	0.9300
C2—C3	1.340 (11)	C25—C26	1.349 (13)
C2—H2A	0.9300	C25—H25A	0.9300
C3—C4	1.401 (10)	C26—C27	1.423 (11)
C4—C5	1.342 (11)	C26—H26A	0.9300
C4—H4A	0.9300	C27—C28	1.398 (11)
C5—C6	1.340 (13)	C28—C29	1.388 (12)
C5—H5A	0.9300	C28—H28A	0.9300
C6—H6A	0.9300	C29—H29A	0.9300
C7—C12	1.333 (12)	C30—C35	1.374 (12)
C7—C8	1.423 (12)	C30—C31	1.391 (11)
C7—H7A	0.9300	C30—H30A	0.9300
C8—C9	1.390 (11)	C31—C32	1.378 (10)
C8—H8A	0.9300	C31—H31A	0.9300
C9—C10	1.358 (10)	C32—C33	1.384 (10)
C10—C11	1.376 (11)	C33—C34	1.382 (10)
C10—H10A	0.9300	C33—H33A	0.9300
C11—C12	1.358 (12)	C34—C35	1.366 (12)
C11—H11A	0.9300	C34—H34A	0.9300
C12—H12A	0.9300	C35—H35A	0.9300
C13—C18	1.373 (10)	C36—C41	1.358 (13)
C13—C14	1.388 (10)	C36—C37	1.378 (12)
C13—H13A	0.9300	C36—H36A	0.9300
C14—C15	1.365 (10)	C37—C38	1.389 (10)
C14—H14A	0.9300	C37—H37A	0.9300
C15—C16	1.355 (10)	C38—C39	1.375 (10)
C15—H15A	0.9300	C39—C40	1.403 (11)
C16—C17	1.402 (10)	C39—H39A	0.9300
C17—C18	1.369 (10)	C40—C41	1.370 (12)
C17—H17A	0.9300	C40—H40A	0.9300
C18—H18A	0.9300	C41—H41A	0.9300
C19—C20	1.542 (10)	C42—C43	1.501 (10)
C19—H19A	0.9700	C42—H42A	0.9700
C19—H19B	0.9700	C42—H42B	0.9700
C20—C21	1.512 (10)	C43—C44	1.524 (9)
C20—H20A	0.9700	C43—H43A	0.9700
C20—H20B	0.9700	C43—H43B	0.9700
C21—C22	1.533 (11)	C44—C45	1.508 (10)
C21—H21A	0.9700	C44—H44A	0.9700
C21—H21B	0.9700	C44—H44B	0.9700
C22—C23	1.499 (11)	C45—C46	1.470 (11)
C22—H22A	0.9700	C45—H45A	0.9700
C22—H22B	0.9700	C45—H45B	0.9700
C9—P1—C3	110.4 (4)	C38—P2—C27	110.9 (4)
C9—P1—C16	107.9 (4)	C38—P2—C32	109.2 (4)
C3—P1—C16	110.0 (4)	C27—P2—C32	109.4 (3)

C9—P1—C19	109.8 (4)	C38—P2—C42	107.4 (4)
C3—P1—C19	107.5 (4)	C27—P2—C42	109.9 (4)
C16—P1—C19	111.3 (3)	C32—P2—C42	110.1 (3)
C23—O2—H2B	109.5	C46—O4—H4B	109.5
C2—C1—C6	119.6 (10)	C25—C24—C29	120.3 (10)
C2—C1—H1A	120.2	C25—C24—H24A	119.9
C6—C1—H1A	120.2	C29—C24—H24A	119.9
C3—C2—C1	122.3 (10)	C26—C25—C24	121.8 (11)
C3—C2—H2A	118.8	C26—C25—H25A	119.1
C1—C2—H2A	118.8	C24—C25—H25A	119.1
C2—C3—C4	117.2 (8)	C25—C26—C27	119.6 (9)
C2—C3—P1	120.4 (7)	C25—C26—H26A	120.2
C4—C3—P1	122.4 (6)	C27—C26—H26A	120.2
C5—C4—C3	120.1 (9)	C28—C27—C26	118.1 (8)
C5—C4—H4A	119.9	C28—C27—P2	121.3 (6)
C3—C4—H4A	119.9	C26—C27—P2	120.6 (7)
C6—C5—C4	122.9 (10)	C29—C28—C27	120.3 (9)
C6—C5—H5A	118.5	C29—C28—H28A	119.9
C4—C5—H5A	118.5	C27—C28—H28A	119.9
C5—C6—C1	117.7 (10)	C24—C29—C28	119.9 (10)
C5—C6—H6A	121.2	C24—C29—H29A	120.1
C1—C6—H6A	121.2	C28—C29—H29A	120.1
C12—C7—C8	122.6 (9)	C35—C30—C31	118.7 (8)
C12—C7—H7A	118.7	C35—C30—H30A	120.7
C8—C7—H7A	118.7	C31—C30—H30A	120.7
C9—C8—C7	117.6 (9)	C32—C31—C30	120.7 (8)
C9—C8—H8A	121.2	C32—C31—H31A	119.6
C7—C8—H8A	121.2	C30—C31—H31A	119.6
C10—C9—C8	118.1 (8)	C31—C32—C33	119.5 (7)
C10—C9—P1	121.3 (6)	C31—C32—P2	121.2 (6)
C8—C9—P1	120.6 (6)	C33—C32—P2	119.1 (6)
C9—C10—C11	122.6 (8)	C34—C33—C32	119.8 (7)
C9—C10—H10A	118.7	C34—C33—H33A	120.1
C11—C10—H10A	118.7	C32—C33—H33A	120.1
C12—C11—C10	120.2 (9)	C35—C34—C33	120.1 (8)
C12—C11—H11A	119.9	C35—C34—H34A	119.9
C10—C11—H11A	119.9	C33—C34—H34A	119.9
C7—C12—C11	118.7 (9)	C34—C35—C30	121.1 (8)
C7—C12—H12A	120.7	C34—C35—H35A	119.4
C11—C12—H12A	120.7	C30—C35—H35A	119.4
C18—C13—C14	118.1 (7)	C41—C36—C37	120.7 (9)
C18—C13—H13A	121.0	C41—C36—H36A	119.7
C14—C13—H13A	121.0	C37—C36—H36A	119.7
C15—C14—C13	121.2 (8)	C36—C37—C38	120.2 (8)
C15—C14—H14A	119.4	C36—C37—H37A	119.9
C13—C14—H14A	119.4	C38—C37—H37A	119.9
C16—C15—C14	120.6 (7)	C39—C38—C37	119.1 (8)
C16—C15—H15A	119.7	C39—C38—P2	121.0 (6)
C14—C15—H15A	119.7	C37—C38—P2	119.8 (6)

C15—C16—C17	119.1 (7)	C38—C39—C40	119.9 (8)
C15—C16—P1	121.5 (6)	C38—C39—H39A	120.1
C17—C16—P1	119.4 (6)	C40—C39—H39A	120.1
C18—C17—C16	119.9 (7)	C41—C40—C39	119.9 (8)
C18—C17—H17A	120.0	C41—C40—H40A	120.1
C16—C17—H17A	120.0	C39—C40—H40A	120.1
C17—C18—C13	121.0 (8)	C36—C41—C40	120.1 (9)
C17—C18—H18A	119.5	C36—C41—H41A	120.0
C13—C18—H18A	119.5	C40—C41—H41A	120.0
C20—C19—P1	115.2 (5)	C43—C42—P2	115.9 (5)
C20—C19—H19A	108.5	C43—C42—H42A	108.3
P1—C19—H19A	108.5	P2—C42—H42A	108.3
C20—C19—H19B	108.5	C43—C42—H42B	108.3
P1—C19—H19B	108.5	P2—C42—H42B	108.3
H19A—C19—H19B	107.5	H42A—C42—H42B	107.4
C21—C20—C19	110.5 (7)	C42—C43—C44	113.3 (6)
C21—C20—H20A	109.5	C42—C43—H43A	108.9
C19—C20—H20A	109.5	C44—C43—H43A	108.9
C21—C20—H20B	109.5	C42—C43—H43B	108.9
C19—C20—H20B	109.5	C44—C43—H43B	108.9
H20A—C20—H20B	108.1	H43A—C43—H43B	107.7
C20—C21—C22	111.8 (7)	C45—C44—C43	110.2 (6)
C20—C21—H21A	109.3	C45—C44—H44A	109.6
C22—C21—H21A	109.3	C43—C44—H44A	109.6
C20—C21—H21B	109.3	C45—C44—H44B	109.6
C22—C21—H21B	109.3	C43—C44—H44B	109.6
H21A—C21—H21B	107.9	H44A—C44—H44B	108.1
C23—C22—C21	114.4 (7)	C46—C45—C44	118.5 (7)
C23—C22—H22A	108.7	C46—C45—H45A	107.7
C21—C22—H22A	108.7	C44—C45—H45A	107.7
C23—C22—H22B	108.7	C46—C45—H45B	107.7
C21—C22—H22B	108.7	C44—C45—H45B	107.7
H22A—C22—H22B	107.6	H45A—C45—H45B	107.1
O1—C23—O2	122.6 (8)	O3—C46—O4	120.7 (9)
O1—C23—C22	124.9 (8)	O3—C46—C45	123.4 (9)
O2—C23—C22	112.5 (8)	O4—C46—C45	115.8 (8)
C6—C1—C2—C3	1.5 (17)	C29—C24—C25—C26	−1.4 (12)
C1—C2—C3—C4	−1.6 (15)	C24—C25—C26—C27	−1.0 (12)
C1—C2—C3—P1	179.6 (8)	C25—C26—C27—C28	2.2 (12)
C9—P1—C3—C2	−97.1 (8)	C25—C26—C27—P2	−177.5 (6)
C16—P1—C3—C2	21.8 (9)	C38—P2—C27—C28	169.5 (6)
C19—P1—C3—C2	143.2 (7)	C32—P2—C27—C28	49.0 (7)
C9—P1—C3—C4	84.1 (8)	C42—P2—C27—C28	−72.0 (7)
C16—P1—C3—C4	−156.9 (7)	C38—P2—C27—C26	−10.8 (8)
C19—P1—C3—C4	−35.6 (8)	C32—P2—C27—C26	−131.3 (6)
C2—C3—C4—C5	2.9 (13)	C42—P2—C27—C26	107.8 (7)
P1—C3—C4—C5	−178.3 (7)	C26—C27—C28—C29	−1.1 (12)
C3—C4—C5—C6	−4.4 (15)	P2—C27—C28—C29	178.6 (6)
C4—C5—C6—C1	4.2 (16)	C25—C24—C29—C28	2.5 (13)

C2—C1—C6—C5	−2.6 (16)	C27—C28—C29—C24	−1.2 (13)
C12—C7—C8—C9	−5.4 (16)	C35—C30—C31—C32	−0.4 (15)
C7—C8—C9—C10	3.7 (13)	C30—C31—C32—C33	0.0 (13)
C7—C8—C9—P1	−176.9 (7)	C30—C31—C32—P2	174.1 (7)
C3—P1—C9—C10	171.5 (7)	C38—P2—C32—C31	−100.5 (7)
C16—P1—C9—C10	51.3 (8)	C27—P2—C32—C31	21.0 (8)
C19—P1—C9—C10	−70.2 (8)	C42—P2—C32—C31	141.8 (7)
C3—P1—C9—C8	−7.9 (8)	C38—P2—C32—C33	73.6 (7)
C16—P1—C9—C8	−128.1 (7)	C27—P2—C32—C33	−164.9 (6)
C19—P1—C9—C8	110.4 (7)	C42—P2—C32—C33	−44.1 (7)
C8—C9—C10—C11	−1.3 (14)	C31—C32—C33—C34	0.2 (13)
P1—C9—C10—C11	179.2 (7)	P2—C32—C33—C34	−174.1 (7)
C9—C10—C11—C12	0.3 (15)	C32—C33—C34—C35	0.0 (14)
C8—C7—C12—C11	4.5 (16)	C33—C34—C35—C30	−0.4 (15)
C10—C11—C12—C7	−1.9 (15)	C31—C30—C35—C34	0.6 (15)
C18—C13—C14—C15	−1.8 (13)	C41—C36—C37—C38	0.1 (15)
C13—C14—C15—C16	1.0 (13)	C36—C37—C38—C39	2.1 (13)
C14—C15—C16—C17	0.5 (12)	C36—C37—C38—P2	−175.2 (7)
C14—C15—C16—P1	178.4 (6)	C27—P2—C38—C39	−103.7 (7)
C9—P1—C16—C15	26.9 (8)	C32—P2—C38—C39	16.9 (7)
C3—P1—C16—C15	−93.5 (7)	C42—P2—C38—C39	136.3 (6)
C19—P1—C16—C15	147.4 (7)	C27—P2—C38—C37	73.6 (7)
C9—P1—C16—C17	−155.1 (6)	C32—P2—C38—C37	−165.8 (6)
C3—P1—C16—C17	84.4 (7)	C42—P2—C38—C37	−46.4 (7)
C19—P1—C16—C17	−34.6 (7)	C37—C38—C39—C40	−1.0 (12)
C15—C16—C17—C18	−1.1 (12)	P2—C38—C39—C40	176.3 (6)
P1—C16—C17—C18	−179.1 (6)	C38—C39—C40—C41	−2.4 (12)
C16—C17—C18—C13	0.2 (13)	C37—C36—C41—C40	−3.6 (15)
C14—C13—C18—C17	1.2 (13)	C39—C40—C41—C36	4.7 (14)
C9—P1—C19—C20	68.5 (6)	C38—P2—C42—C43	−168.8 (5)
C3—P1—C19—C20	−171.4 (5)	C27—P2—C42—C43	70.5 (7)
C16—P1—C19—C20	−50.9 (7)	C32—P2—C42—C43	−50.0 (7)
P1—C19—C20—C21	−178.2 (6)	P2—C42—C43—C44	−174.5 (5)
C19—C20—C21—C22	−168.7 (7)	C42—C43—C44—C45	−169.7 (6)
C20—C21—C22—C23	−67.3 (10)	C43—C44—C45—C46	−165.2 (7)
C21—C22—C23—O1	−19.9 (13)	C44—C45—C46—O3	154.3 (10)
C21—C22—C23—O2	161.1 (8)	C44—C45—C46—O4	−28.9 (12)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···Br1[i]	0.82	2.35	3.148 (7)	165
O4—H4B···Br2[ii]	0.82	2.41	3.199 (7)	163
C14—H14A···O1[iii]	0.93	2.58	3.357 (10)	141
C17—H17A···Br2[iv]	0.93	2.77	3.691 (7)	169
C19—H19A···Br2[iv]	0.97	2.88	3.790 (7)	157
C19—H19B···O1[i]	0.97	2.41	3.351 (10)	163
C33—H33A···Br2	0.93	2.79	3.623 (9)	150

C37—H37A···Br1[v]	0.93	2.82	3.574 (8)	139

Symmetry codes: (i) $-x+2$, $-y+2$, $-z+1$; (ii) $-x$, $-y+1$, $-z+1$; (iii) $-x+3$, $-y+2$, $-z+1$; (iv) $x+1$, $y+1$, z; (v) $x-1$, y, z.
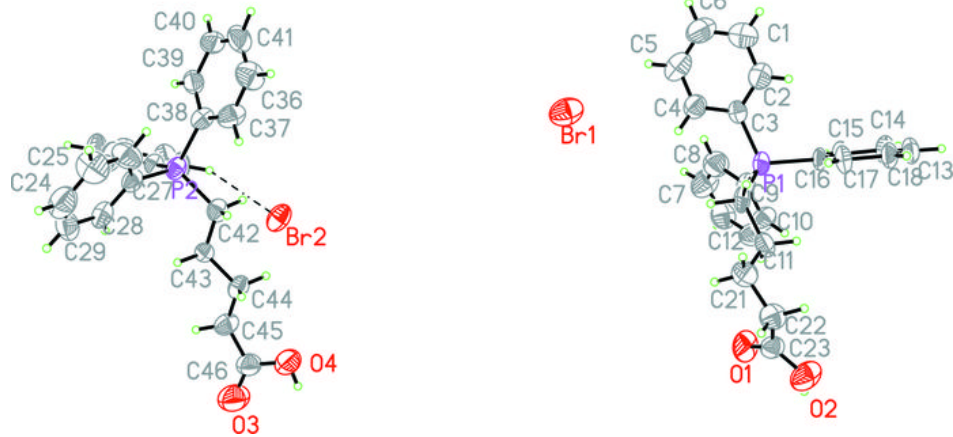
Fig. 1

Fig. 2