Data collection

Hilger Y290 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.14$, $T_{\max} = 0.23$
2259 measured reflections
2259 independent reflections
1755 observed reflections [$I > 3\sigma(I)$]
$\theta_{\max} = 25°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 9$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 100 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.039$
$wR = 0.045$
1755 reflections
167 parameters
All H-atom parameters refined
Weights: four-parameter Chebyshev polynomial
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.62$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
I(1)	0.58009 (5)	0.08604 (5)	0.69855 (4)	0.0586
As(1)	0.48010 (5)	0.33026 (8)	0.77258 (5)	0.0488
C(1)	0.5679 (5)	0.3360 (7)	0.9120 (5)	0.0449
C(2)	0.6551 (6)	0.4398 (9)	0.9395 (6)	0.0522
C(3)	0.7107 (7)	0.438 (1)	1.0436 (6)	0.0633
C(4)	0.6767 (8)	0.334 (1)	1.1183 (6)	0.0659
C(5)	0.5892 (9)	0.231 (1)	1.0928 (6)	0.0662
C(6)	0.5331 (8)	0.233 (1)	0.9904 (6)	0.0622
C(7)	0.5708 (5)	0.4767 (7)	0.6875 (4)	0.0441
C(8)	0.5007 (6)	0.5866 (8)	0.6349 (5)	0.0545
C(9)	0.5559 (8)	0.6942 (8)	0.5709 (5)	0.0601
C(10)	0.6795 (9)	0.6902 (9)	0.5564 (6)	0.0649
C(11)	0.7496 (8)	0.5800 (9)	0.6073 (6)	0.0650
C(12)	0.6946 (6)	0.4731 (8)	0.6724 (5)	0.0515

Table 2. *Selected geometric parameters* (Å, °)

I(1)—As(1)	2.600 (1)	As(1)—C(7)	1.953 (6)
As(1)—C(1)	1.942 (6)		
C(1)—As(1)—I(1)	97.0 (2)	C(6)—C(1)—As(1)	116.9 (6)
C(7)—As(1)—I(1)	98.1 (2)	C(8)—C(7)—As(1)	115.3 (5)
C(7)—As(1)—C(1)	101.7 (2)	C(12)—C(7)—As(1)	125.1 (5)
C(2)—C(1)—As(1)	124.5 (5)		

The structure was solved using *MULTAN* (Main *et al.*, 1980) and refined using the *CRYSTALS* program package (Watkin, Carruthers & Betteridge, 1985).

This work was supported by the British Council through the ROMLISS program.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, and a packing diagram of the title compound have been deposited with the IUCr (Reference: MU1163). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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L-(+)-α-Bromobenzeneacetic Acid (−)-Menthyl Ester

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Abstract

The crystal structure of L-(+)-α-bromobenzeneacetic acid (−)-menthyl ester ($C_{18}H_{25}BrO_2$) is reported. The Br atom and the O(carbonyl) atom are in *trans* positions. The 1-methyl-4-isopropylcyclohexane ring has a chair conformation. The dihedral angle between the least-squares plane through the phenyl ring and the least-squares plane of the C—CO$_2$ group is 45.0 (2)°. The bond distances and angles of the molecule are in the normal ranges.

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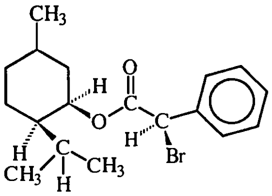
Comment

The structure determination of the title compound, (I), was undertaken in order to obtain structural information about optically active derivatives of α-bromobenzeneacetic acid; compounds of this class have not been investigated previously. The data of these compounds are important since optically active α-bromobenzeneacetic acid is related to the mandelic acids. The latter are basic natural products and play a significant role in stereochemical assignment.



(I)

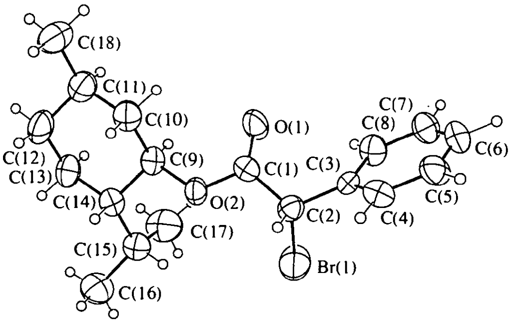


Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the title molecule showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

Experimental

The title compound was synthesized by the reaction of (±)-α-bromophenylacetyl chloride with (−)-menthol. The (±)-α-bromophenylacetyl chloride was prepared by analogy with the method of the Bristol-Banyu Research Institute (1965), by a 4 h gentle reflux of a 1:1:1.3 mixture of phenylacetyl chloride, thionyl chloride and bromine. To the crude product, obtained after removal *in vacuo* of the excess of the bromine and the thionyl chloride, (−)-menthol was added dropwise at room temperature while stirring. The mixture of the (−)-menthyl ester of L-(+)-α-bromobenzeneacetic acid and the (−)-menthyl ester of D-(−)-α-bromobenzeneacetic acid was submitted to separation by fractional recrystallization from ethanol.

The melting point, in the range 351–352 K, and specific optical rotation, $[\alpha]_D^{18} = +10.1^\circ$ (ethanol), of the isolated pure (−)-menthyl ester of L-(+)-α-bromobenzeneacetic acid are in agreement with data given by McKenzie & Smith (1923). The IR spectrum shows a band at 1732 cm^{-1} in the carbonyl stretching region while the corresponding Raman band is at 1733 cm^{-1}. There is no spectral evidence for the existence of a hydrogen bond between the C═O group and any of the H atoms.

Crystal data

$C_{18}H_{25}BrO_2$
$M_r = 353.30$
Monoclinic
$P2_1$
$a = 5.522\,(5)$ Å
$b = 19.389\,(17)$ Å
$c = 8.208\,(6)$ Å
$\beta = 92.68\,(7)^\circ$
$V = 878\,(1)$ Å^3
$Z = 2$
$D_x = 1.336$ Mg m^{-3}

Mo *Kα* radiation
$\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 7.71$–15.16°
$\mu = 2.32$ mm^{-1}
$T = 293\,(1)$ K
Approximately quadratic column
0.52 × 0.19 × 0.16 mm
Colourless

Data collection

Nicolet *R3m/V* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2606 measured reflections
1272 independent reflections
1001 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int} = 0.0452$
$\theta_{\rm max} = 22.5^\circ$
$h = -6 \rightarrow 6$
$k = -1 \rightarrow 21$
$l = -9 \rightarrow 9$
6 standard reflections
frequency: 150 min
intensity decay: none

Refinement

Refinement on *F*
$R = 0.0292$
$wR = 0.0299$
$S = 0.68$
1001 reflections
190 parameters
Unit weights applied
$(\Delta/\sigma)_{\rm max} = 0.003$

$\Delta\rho_{\rm max} = 0.3$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.3$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Br(1)	0.1989 (2)	0.0000	0.07376 (9)	0.0885
O(1)	−0.2872 (9)	0.0765 (3)	−0.1618 (6)	0.0691
O(2)	−0.0389 (8)	0.1447 (2)	−0.0115 (5)	0.0406
C(1)	−0.091 (1)	0.0898 (4)	−0.1059 (7)	0.0412
C(2)	0.135 (1)	0.0485 (4)	−0.1331 (7)	0.0413
C(3)	0.1197 (9)	−0.0011 (5)	−0.2731 (6)	0.0369
C(4)	0.297 (1)	−0.0010 (6)	−0.3847 (6)	0.0459
C(5)	0.292 (1)	−0.0452 (4)	−0.5143 (8)	0.0534
C(6)	0.109 (1)	−0.0916 (4)	−0.5333 (8)	0.0543
C(7)	−0.071 (1)	−0.0934 (4)	−0.4250 (9)	0.0558
C(8)	−0.067 (1)	−0.0485 (4)	−0.2942 (8)	0.0481
C(9)	−0.247 (1)	0.1834 (4)	0.0428 (8)	0.0391
C(10)	−0.323 (1)	0.2369 (4)	−0.0838 (9)	0.0482
C(11)	−0.533 (1)	0.2788 (4)	−0.028 (1)	0.0551
C(12)	−0.470 (1)	0.3109 (4)	0.1370 (9)	0.0602
C(13)	−0.387 (1)	0.2574 (4)	0.2619 (9)	0.0540
C(14)	−0.171 (1)	0.2149 (4)	0.2056 (8)	0.0406
C(15)	−0.077 (1)	0.1620 (4)	0.3332 (8)	0.0475
C(16)	0.022 (2)	0.1957 (4)	0.4895 (9)	0.0668
C(17)	−0.258 (2)	0.1060 (4)	0.371 (1)	0.0805
C(18)	−0.619 (2)	0.3313 (5)	−0.156 (1)	0.0740

Table 2. *Selected geometric parameters* (Å, °)

Br(1)—C(2)	1.959 (6)	C(7)—C(8)	1.38 (1)
O(1)—C(1)	1.184 (8)	C(9)—C(10)	1.51 (1)
O(2)—C(1)	1.339 (8)	C(9)—C(14)	1.511 (9)
O(2)—C(9)	1.461 (8)	C(10)—C(11)	1.51 (1)
C(1)—C(2)	1.507 (9)	C(11)—C(12)	1.52 (1)

C(2)—C(3)	1.499 (9)	C(11)—C(18)	1.52 (1)
C(3)—C(4)	1.370 (8)	C(12)—C(13)	1.51 (1)
C(3)—C(8)	1.38 (1)	C(13)—C(14)	1.54 (1)
C(4)—C(5)	1.37 (1)	C(14)—C(15)	1.539 (9)
C(5)—C(6)	1.36 (1)	C(15)—C(16)	1.52 (1)
C(6)—C(7)	1.37 (1)	C(15)—C(17)	1.52 (1)
C(1)—O(2)—C(9)	115.6 (5)	O(2)—C(9)—C(14)	106.6 (6)
O(1)—C(1)—O(2)	124.5 (7)	O(2)—C(9)—C(10)	109.9 (6)
O(2)—C(1)—C(2)	110.5 (6)	C(10)—C(9)—C(14)	112.8 (6)
O(1)—C(1)—C(2)	125.0 (6)	C(9)—C(10)—C(11)	110.8 (6)
Br(1)—C(2)—C(1)	104.1 (4)	C(10)—C(11)—C(18)	111.8 (7)
C(1)—C(2)—C(3)	116.0 (5)	C(10)—C(11)—C(12)	110.1 (7)
Br(1)—C(2)—C(3)	110.9 (5)	C(12)—C(11)—C(18)	113.1 (7)
C(2)—C(3)—C(8)	122.4 (6)	C(11)—C(12)—C(13)	112.0 (6)
C(2)—C(3)—C(4)	119.8 (6)	C(12)—C(13)—C(14)	112.3 (6)
C(4)—C(3)—C(8)	117.9 (6)	C(9)—C(14)—C(13)	107.1 (6)
C(3)—C(4)—C(5)	122.2 (6)	C(13)—C(14)—C(15)	113.3 (6)
C(4)—C(5)—C(6)	119.5 (7)	C(9)—C(14)—C(15)	113.8 (6)
C(5)—C(6)—C(7)	120.2 (7)	C(14)—C(15)—C(17)	114.2 (6)
C(6)—C(7)—C(8)	120.3 (7)	C(14)—C(15)—C(16)	112.6 (6)
C(3)—C(8)—C(7)	120.0 (7)	C(16)—C(15)—C(17)	110.5 (6)
O(1)—C(1)—C(2)—C(3)	16.4 (10)		
C(10)—C(9)—C(14)—C(13)	57.9 (8)		
C(14)—C(9)—C(10)—C(11)	−59.6 (8)		
C(9)—C(10)—C(11)—C(12)	55.4 (8)		
C(10)—C(11)—C(12)—C(13)	−54.3 (9)		
C(11)—C(12)—C(13)—C(14)	55.8 (9)		
C(12)—C(13)—C(14)—C(9)	−55.7 (8)		

Refinement was based on full-matrix least-squares methods with anisotropic displacement parameters for non-H atoms and one common isotropic displacement parameter [0.060 (4) Å^2] for the H atoms, which were placed in calculated positions. Unit weights led to featureless analysis of variance in terms of $\sin\theta$ and $(F/F_{\max})^{1/2}$. An η refinement [the data set contained the pairs $(h\ -1\ l)$, $(h\ 1\ l)$] (Rogers, 1981) gave η = 0.97 (10) and confirms the proposed chirality.

Data collection: Nicolet *R3m/V* diffractometer software. Cell refinement: Nicolet *R3m/V* diffractometer software. Data reduction: Nicolet *R3m/V* diffractometer software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus* and *SHELX*76 (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983), *PLATON* (Spek, 1982) and *MISSYM* (Le Page, 1987).

One of us (TK) thanks the Alexander von Humboldt Stiftung for a research fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1107). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(2,4-di-*tert*-butylphenyl) Pentaerythritol Diphosphite

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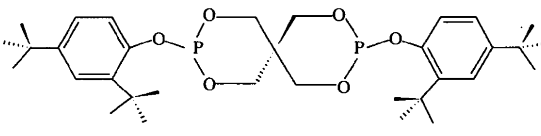
Abstract

The title compound, 3,9-bis(2,4-di-*tert*-butylphenoxy)-2,4,8,10-tetraoxa-3,9-diphosphaspiro[5.5]undecane, (1), $C_{33}H_{50}O_6P_2$, has three different conformers in the unit cell. The structure of each is very similar to the others, differing principally in the rotational conformations of the aryl *tert*-butyl groups. The remaining portion of the structure comprises two six-membered rings, each in a chair conformation and sharing a common spiro atom. Bond angles and bond lengths in the spiro rings are very similar to those of the related molecule 3,9-dimethoxy-2,4,8,10-tetraoxa-3,9-diphosphaspiro[5.5]undecane [Carpenter, Jacobson & Verkade (1979). *Phosphorus Sulfur*, **6**, 475–479].

Comment

The solution of the structure of the title compound, (I), indicated the presence of three conformers in the unit cell, differing principally in the rotational conformations of the aryl *tert*-butyl groups. Fig. 1 shows the molecule with the numbering scheme employed.



(I)

On comparison with the structure of 3,9-dimethoxy-2,4,8,10-tetraoxa-3,9-diphosphaspiro[5.5]undecane (Carpenter, Jacobson & Verkade, 1979), (II), minor differences are observed in the P—O—Me and P—O—aryl bond angles. The six P—O—aryl angles in (I) [123.02 (14), 123.2 (2), 122.3 (2), 122.2 (2), 122.2 (2) and 123.8 (2)°] are larger than the P—O—Me an-

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