

Data collection

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

Refinement

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

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$)

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{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 ($\text{\AA}$, $^\circ$)

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.

References

- Boardman, A., Small, R. W. H. & Worrall, I. J. (1986). *Inorg. Chim. Acta*, **121**, L35–L36.
- Emsley, J. (1991). In *The Elements*, 2nd ed. Oxford Univ. Press.
- Enjalbert, R. & Galy, J. (1980). *Acta Cryst.* **B36**, 914–916.
- Henrick, K., Raston, C. L., White, A. H. & Wild, S. B. (1977). *Aust. J. Chem.* **30**, 2417–2423.
- Kniep, R. & Reski, H. D. (1982). *Inorg. Chim. Acta*, **64**, L83–L84.
- McAuliffe, C. A., Beagley, B., Gott, G. A., Mackie, A. G., MacRory, P. P. & Pritchard, R. G. (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 264–265.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Millington, P. L. & Sowerby, D. B. (1994). *J. Organomet. Chem.* **480**, 227–234.
- Mundt, G., Becker, G., Stadelmann, H. & Thurn, H. (1992). *Z. Anorg. Allg. Chem.* **617**, 59–71.
- Pope, W. J. & Turner, E. E. (1920). *J. Chem. Soc.* pp. 1447–1452.
- Schmidbaur, H., Bublak, W., Huber, B. & Müller, G. (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 234–235.
- Steinkopf, W. & Schwen, G. (1921). *Ber.* **54**, 1437–1466.
- Trotter, J. (1962a). *Can. J. Chem.* **40**, 1590–1593.
- Trotter, J. (1962b). *J. Chem. Soc.* pp. 2567–2572.
- Trotter, J. (1963). *Can. J. Chem.* **41**, 191.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
- Watkin, D. J., Carruthers, J. R. & Betteridge, D. W. (1985). *CRYSTALS Users Guide*. Chemical Crystallography Laboratory, Univ. of Oxford, England.

Acta Cryst. (1995). **C51**, 1634–1636

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

TSONKO KOLEV, HANS PREUT, LOTHAR KONICZEK AND
PAUL BLECKMANN

*Fachbereich Chemie, Universität Dortmund,
D-44227 Dortmund, Germany*

IVAN JUCHNOVSKI AND MARGARITA MLADENOVA

*Institute for Organic Chemistry, Bulgarian Academy of
Sciences, Sofia 1113, Bulgaria*

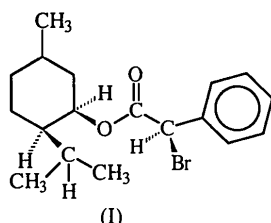
(Received 8 November 1994; accepted 30 January 1995)

Abstract

The crystal structure of L-(+)- α -bromobenzeneacetic acid (-)-menthyl ester (C₁₈H₂₅BrO₂) 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—CO₂ group is 45.0 (2)°. The bond distances and angles of the molecule are in the normal ranges.

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.

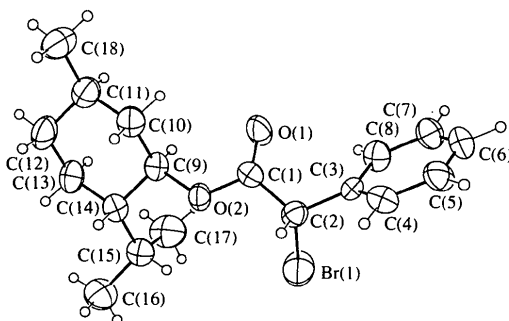


Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the title molecule showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

Experimental

The title compound was synthesized by the reaction of ($\pm$)- α -bromophenylacetyl chloride with (–)-menthol. The ($\pm$)- 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

$\text{C}_{18}\text{H}_{25}\text{BrO}_2$
 $M_r = 353.30$
 Monoclinic
 $P2_1$
 $a = 5.522(5)\text{ \AA}$
 $b = 19.389(17)\text{ \AA}$
 $c = 8.208(6)\text{ \AA}$
 $\beta = 92.68(7)^\circ$
 $V = 878(1)\text{ \AA}^3$
 $Z = 2$
 $D_x = 1.336\text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71073\text{ \AA}$
 Cell parameters from 30 reflections
 $\theta = 7.71\text{--}15.16^\circ$
 $\mu = 2.32\text{ mm}^{-1}$
 $T = 293(1)\text{ K}$
 Approximately quadratic column
 $0.52 \times 0.19 \times 0.16\text{ mm}$
 Colourless

Data collection

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

$R_{\text{int}} = 0.0452$
 $\theta_{\text{max}} = 22.5^\circ$
 $h = -6 \rightarrow 6$
 $k = -1 \rightarrow 21$
 $l = -9 \rightarrow 9$
 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
 $(\Delta/\sigma)_{\text{max}} = 0.003$

$\Delta\rho_{\text{max}} = 0.3\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.3\text{ e \AA}^{-3}$
 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 ($\text{\AA}^2$)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{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)	–0.0228 (11)	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 ($\text{\AA}$, $^\circ$)

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) Å²] 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 $\eta = 0.97$ (10) and confirms the proposed chirality.

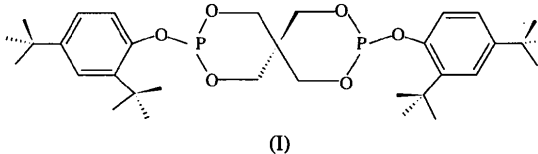
Data collection: Nicolet R3m/V diffractometer software. Cell refinement: Nicolet R3m/V diffractometer software. Data reduction: Nicolet R3m/V diffractometer software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus* and *SHELX76* (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.

References

- Bristol-Banyu Research Institute (1965). *Res. Inst. Neth.* P6404841; *Chem. Abstr.* **62**, P16271a.
- Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.
- McKenzie, A. & Smith, I. (1923). *J. Chem. Soc.* **125**, 1282–1293.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Rogers, D. (1981). *Acta Cryst.* **A37**, 734–741.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet R3m/V crystallographic systems. Nicolet Instrument Corporation, Madison, Wisconsin, USA.
- Spek, A. L. (1982). *The EUCLID Package. Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.
- Acta Cryst.* (1995). **C51**, 1636–1639
- ## Bis(2,4-di-*tert*-butylphenyl) Pentaerythritol Diphosphite
- JAMES P. BARREN, GAROLD L. BRYANT† AND MARY F. GARBAUSKAS
- General Electric Research and Development, Schenectady, NY 12301, USA*
- JAMES A. MAHOOD
- General Electric Plastics, Parkersburg, WV 12301, USA*
- (Received 11 October 1993; accepted 18 January 1995)
- ## Abstract
- The title compound, 3,9-bis(2,4-di-*tert*-butylphenoxy)-2,4,8,10-tetraoxa-3,9-diphosphaspiro[5.5]undecane, (I), C₃₃H₅₀O₆P₂, 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.



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-

† Present address: The Upjohn Company, Physical and Analytical Chemistry Research, Kalamazoo, MI 49001, USA.