Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,3,4,5,6-Pentabromophenyl phenyl ether

Lars Eriksson[a]* and Jiwei Hu[b]

[a]Division of Structural Chemistry, Arrhenius Laboratory, Stockholm University, S-106 91 Stockholm, Sweden, and [b]Department of Chemistry, University of Jyväskylä, FIN-40 351 Jyväskylä, Finland

Correspondence e-mail: lerik@struc.su.se

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.036
wR factor = 0.066
Data-to-parameter ratio = 17.4

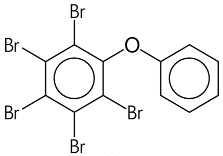
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{12}H_5Br_5O$, belongs to a group of flame retardants known as polybrominated diphenyl ethers (PBDE). Salient features of the packing are stabilization due to intermolecular Br···Br contacts in the *ab* plane and aromatic intermolecular contacts along the *c* direction.

Received 5 June 2002
Accepted 18 June 2002
Online 29 June 2002

Comment

An important group of flame retardants are the polybrominated diphenyl ethers (PBDE). There are a total of 209 different PBDE's. The modelling of the reactivity of different PBDE's is a task that requires accurate geometries of the molecular species. Geometric data from crystallographic measurements on brominated diphenyl ethers without hetero substituents other than bromine are rather limited (Örn *et al.*, 1996; Eriksson *et al.*, 1999; Mrse *et al.*, 2000; Eriksson & Hu, 2001, 2002; Eriksson *et al.*, 2002*a*,*b*). In addition, a partial structure of bis(4-bromophenyl) ether (Toussaint, 1946) has been published. In a search of the Cambridge Structural Database (Allen & Kennard, 1993), including hetero substituents other than bromine, a larger set of structures for use as model compounds was found, but still only in the order of 10–15 different structures.



(I)

The bromine-substituted ring is planar with an r.m.s. deviation of 0.012 Å for the C atoms (C1–C6) of the ring. The O atom deviates most from this plane, by 0.163 (8) Å, while atoms Br1, Br2 and Br3 lie within 2σ of the plane; atoms Br4 and Br5 deviate by 0.037 (8) and 0.035 (8) Å, respectively. The second, unsubstituted, aromatic ring (C7–C12) is more strictly planar, with an r.m.s. deviation of 0.004 Å for the C atoms. The O atom is coplanar with this ring. The angle between the two ring planes is 89.7 (2)°.

A view of the arrangement of the molecules of the title compound, (I), is shown in Fig. 2, where the two shortest intermolecular Br···Br contacts are marked. The closest intermolecular Br···Br contacts are: Br3···Br4^{i} = 3.567 (1) Å and Br2···Br2^{ii} = 3.698 (1) Å [symmetry codes: (i) $-x$, $-1/2+y$, $1/2-z$; (ii) $1-x$, $1/2+y$, $1/2-z$]. The Br2 atoms of different molecules form a connected zigzag chain of close contacts along the *b* axis. Similarly, atoms Br3 and Br4 form a set of intermolecular contacts along the *b* axis and also

DOI: 10.1107/S1600536802010942

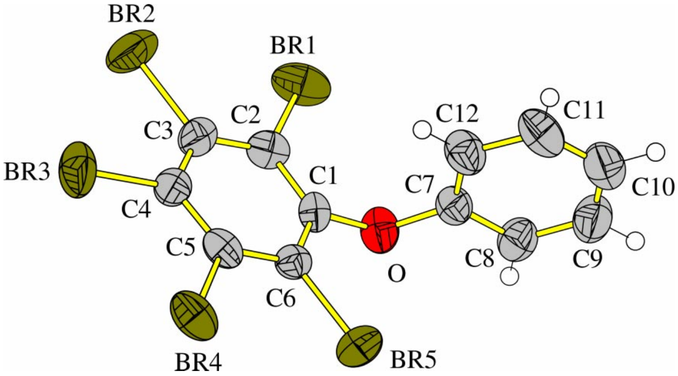


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

contribute to the bonding in the *a*-axis direction. The short intermolecular Br···Br contacts stabilize the structure in the *ab* plane, as shown in Fig. 3. Along the *c*-axis direction, the structure is essentially stabilized by interaction between the unsubstituted aromatic ring system and symmetry-related equivalents, also interactions between both Br1 and Br5 with the unsubstituted aromatic ring.

A related packing pattern was found for 2,4-dibromophenyl 4-bromophenyl ether (Eriksson & Hu, 2002), where the bonding in two directions of the unit cell can be described as partly due to Br···Br interactions. The packing in the third direction is governed mainly by interactions between the aromatic ring systems. The molecules form sheets, with the less substituted ring pointing outwards in every second molecule (*cf*. Fig. 3) of the title compound, while for the aforementioned 2,4-dibromophenyl 4-bromophenyl ether (Eriksson & Hu, 2002), there are larger cavities where two equivalents of the less substituted ring may fit.

Experimental

The synthesis of the PBDE was carried out by coupling of diphenyliodonium bromide with sodium pentabromophenylate (Beringer *et al.*, 1959; Ziegler & Marr, 1962; Hu, 1996, 1999). The title compound was recrystallized from methanol.

Crystal data

$C_{12}H_5Br_5O$
$M_r = 564.71$
Monoclinic, $P2_1/c$
$a = 11.579$ (4) Å
$b = 5.3284$ (11) Å
$c = 24.133$ (6) Å
$\beta = 102.66$ (3)°
$V = 1452.7$ (6) Å^3
$Z = 4$
$D_x = 2.582$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1573 reflections
$\theta = 1.7$–$26.0°$
$\mu = 13.82$ mm^{-1}
$T = 293$ (2) K
Slab, colourless
0.30 × 0.09 × 0.08 mm

Data collection

Stoe IPDS area-detector diffractometer
φ scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1997)
$T_{\min} = 0.081$, $T_{\max} = 0.328$
18710 measured reflections
2843 independent reflections
1503 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.114$
$\theta_{\max} = 26.0°$
$h = -14 \rightarrow 14$
$k = -6 \rightarrow 6$
$l = -29 \rightarrow 29$

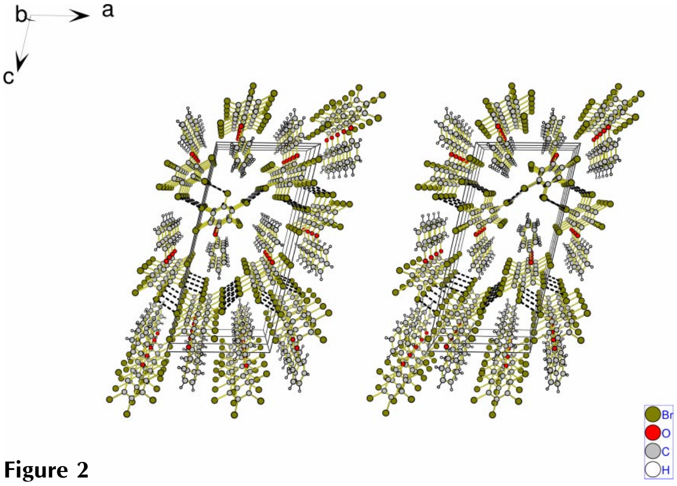


Figure 2
Stereoview of the packing, viewed along the *b* direction, with the two shortest Br···Br contacts indicated by thick dashed bonds.

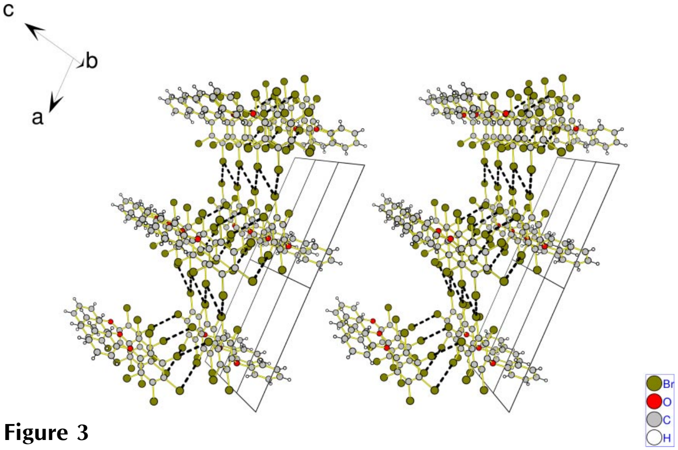


Figure 3
Stereoview of the packing, viewed along the *c* direction, with the two shortest Br···Br contacts indicated by thick dashed bonds. The base plane (2*a* × 3*b*) is marked by the six rectangles.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.066$
$S = 1.03$
2843 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.005P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.55$ e Å^{-3}
$\Delta\rho_{\min} = -0.68$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.866 (6)	Br5—C6	1.876 (6)
Br2—C3	1.900 (6)	O—C1	1.371 (7)
Br3—C4	1.880 (6)	O—C7	1.403 (8)
Br4—C5	1.882 (6)		
C1—O—C7	118.9 (4)	C12—C7—C8	120.8 (6)
O—C1—C2	119.1 (5)	C12—C7—O	123.8 (6)
O—C1—C6	119.0 (6)	C8—C7—O	115.5 (6)
C2—C1—C6	121.6 (6)		
C7—O—C1—C2	100.4 (7)	C1—O—C7—C12	−12.9 (9)
C7—O—C1—C6	−86.2 (7)	C1—O—C7—C8	166.8 (5)

Two data sets were recorded with the Stoe IPDS system, using different settings of the crystal. The data sets were brought to a common scale by use of batch scale factors (BASF) determined with *SHELXL*97, and were merged. The rather high internal R value of 0.114 decreases to 0.073 when only reflections with $I \geq 2\sigma(I)$ are used.

Data collection: *EXPOSE* in *IPDS* (Stoe & Cie, 1997); cell refinement: *CELL* in *IPDS*; data reduction: *INTEGRATE* in *IPDS* and *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996).

This work was supported by a grant from the Swedish Natural Science Research Council.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News,* **8**, 1, 31–37.

Bergerhoff, G. (1996). *DIAMOND*. Gerhard-Domagk-Straße 1, 53121 Bonn, Germany.

Beringer, F. M., Falk, R. A., Karniol, M., Lillien, G., Masullo, M., Mausner, M. & Sommer, E. (1959). *J. Am. Chem. Soc.* **81**, 342–351.

Eriksson, J., Eriksson, L. & Hu, J. (2002*a*). *Acta Cryst.* E**58**, o263–o265.

Eriksson, J., Eriksson, L. & Hu, J. (2002*b*). *Acta Cryst.* E**58**, o347–o349.

Eriksson, J., Eriksson, L. & Jakobsson, E. (1999). *Acta Cryst.* C**55**, 2169–2171.

Eriksson, L. & Hu, J. (2001). *Acta Cryst.* E**57**, o930–o932.

Eriksson, L. & Hu, J. (2002). *Acta Cryst.* E**58**, o696–o698.

Hu, J. (1996). Licenciate Thesis, Department of Environmental Chemistry, Stockholm University, Sweden.

Hu, J. (1999). Doctorate Thesis, Department of Chemistry, Research Report No. 73, University of Jyväskylä, Jyväskylä, Finland.

Mrse, A. A., Watkins, S. F. & Fronczek, F. R. (2000). *Acta Cryst.* C**56**, e576–e577.

Örn, U., Eriksson, L., Jakobsson, E. & Bergman, Å. (1996). *Acta Chem. Scand.* **50**, 802–807.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Stoe & Cie (1997). *IPDS* (Version 2.87) and *X-RED* (Version 1.09). Stoe & Cie GmbH, Hilpertstraße 10, D-64295 Darmstadt, Germany.

Toussaint, J. (1946). *Bull. Soc. R. Sci. Liège,* **15**, 86–93.

Ziegler, H. & Marr, C. (1962). *J. Org. Chem.* **27**, 3335–3336.