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4-Bromophenyl 2,6-dibromophenyl ether

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Key indicators

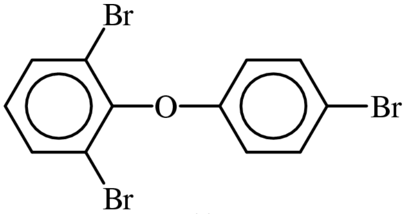
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.039
wR factor = 0.060
Data-to-parameter ratio = 17.2

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

The title compound, $C_{12}H_7Br_3O$, is an example of a compound related to some important flame retardants, *e.g.* decabromodiphenyl ether. Salient intermolecular interactions are the short contacts between the bromine substituents and the C atoms of the aromatic ring system. The molecules make up chains through these short Br$\cdots$C contacts; these chains pack into sheets parallel to the *ab* plane, and copies of these sheets pack on to each other along the *c* axis in a tweed-like pattern.

Comment

The title compound, (I), belongs to a class of compounds known as brominated diphenyl ethers that are used as flame retardants. Commercially available mixtures mainly consist of highly brominated congeners, such as decabromodiphenyl ether (Eriksson *et al.*, 1999). The occurrence in the environment of less brominated congeners is dependent on both primary sources and decomposition from higher brominated diphenyl ethers (Eriksson *et al.*, 2001). The crystal structures and packing patterns of these compounds are of fundamental importance in order to model the reaction mechanisms in the solid state, *e.g.* flame retardants adsorbed on soot particles that decompose by various reactions.



(I)

Both aromatic rings of the title compound (Fig. 1) are planar within less than 0.01 Å. The O atom deviates by 0.042 (7) Å from the ring plane of the first ring (C1–C6), while the Br3 is within the plane. The substituents of the second ring (C7–C12) deviate more from the ring plane: O [0.071 (7) Å], Br1 [0.093 (8) Å] and Br2 [0.010 (7) Å]. The angle between the two ring planes is 85.5 (2)°. The deviations from planarity could possibly be accounted for by the observation of the short intermolecular contact distances Br1$\cdots$C5($-x$, $-y$, $-z$) 3.476 (5) Å and Br2$\cdots$C3($-x-1$, $-y-1$, $-z$) 3.433 (7) Å. These intermolecular Br$\cdots$C contact distances (Fig. 2) can be considered very short compared to previously known structures (Allen & Kennard, 1993). A histogram of known aromatic C$\cdots$Br distances is shown in Fig. 3. The interactions between the molecules in the present structure, mediated by the two above-mentioned contacts, give rise to chains along

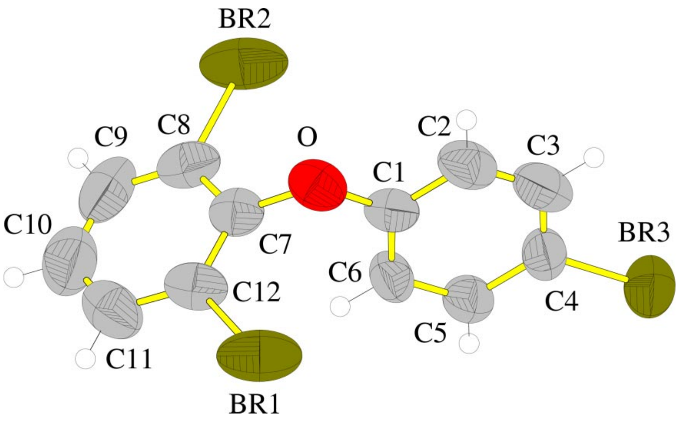


Figure 1
One molecule of (I) with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

[110] at $z \simeq 0$ and along [$\bar{1}$10] at $z \simeq 0.5$. Each of these chains packs with symmetry-related ones into sheets parallel to the *ab* plane, at $z \simeq 0, 0.5, 1.0$, *etc*. The sheets make up a tweed-like pattern seen along the *c* axis, and bind to each other partly through interactions between symmetry-equivalent Br3 atoms. The intermolecular contact distances Br3···Br3($-0.5-x$, $0.5+y$, $-0.5-z$) and Br3···Br3($-0.5-x$, $-0.5+y$, $-0.5-z$) are both 3.929 (1) Å, a fairly long distance. Thus, one might suspect that the solid material would easily be cleaved between these planes. Further intermolecular interactions are the usual 3.7 Å distance between the different aromatic rings in a zigzag pattern.

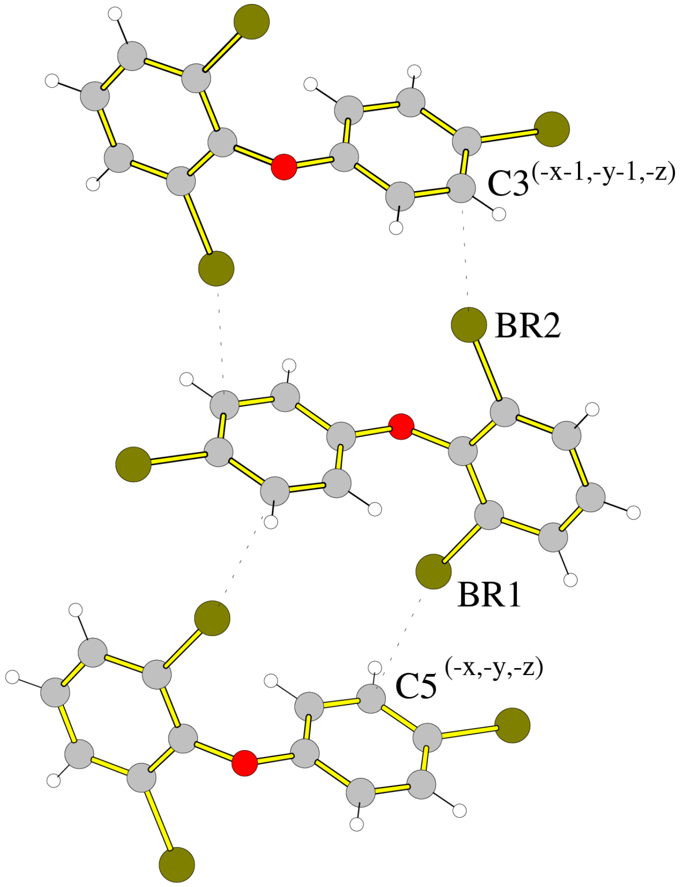


Figure 2
Intermolecular C···Br interactions along the [110] direction.

Experimental

The synthesis of PBDE was carried out by coupling the diphenyl iodonium salt with a bromophenylate (Beringer *et al.*, 1959; Ziegler & Marr, 1962; Hu, 1996, 1999). The title compound was recrystallized from methanol.

Crystal data

$C_{12}H_7Br_3O$
$M_r = 406.91$
Monoclinic, $P2_1/n$
$a = 11.935$ (3) Å
$b = 4.9785$ (10) Å
$c = 22.151$ (9) Å
$\beta = 99.43$ (4)°
$V = 1298.5$ (7) Å^3
$Z = 4$

$D_x = 2.081$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1153 reflections
$\theta = 1.7$–26.1°
$\mu = 9.30$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.12 × 0.10 × 0.08 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1997)
$T_{\min} = 0.323$, $T_{\max} = 0.487$
18021 measured reflections
2491 independent reflections

1100 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.081$
$\theta_{\max} = 26.0°$
$h = -14 \rightarrow 14$
$k = -6 \rightarrow 5$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.060$
$S = 1.06$
2491 reflections
145 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.64$ e Å^{-3}
$\Delta\rho_{\min} = -0.54$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C12	1.890 (5)	O—C7	1.384 (6)
Br2—C8	1.897 (5)	O—C1	1.388 (6)
Br3—C4	1.907 (5)		
C7—O—C1	118.1 (4)	O—C7—C8	122.4 (5)
C6—C1—O	123.4 (5)	O—C7—C12	120.8 (5)
C2—C1—O	115.6 (5)		
C12—C7—O—C1	96.4 (6)	C6—C1—O—C7	−16.5 (7)
C8—C7—O—C1	−87.4 (6)	C2—C1—O—C7	164.9 (4)

Two data sets were collected with the Stoe IPDS system (Stoe, 1997), merged and scaled together. The rather high internal *R* value is, to a large extent, dependent on the contribution from the large number of non-significant reflections. The internal *R* value using the reflections with $I \geq 2\sigma(I)$ is 0.040.

Data collection: *EXPOSE* (Stoe & Cie, 1997); cell refinement: *CELL* (Stoe & Cie, 1997); data reduction: *INTEGRATE* (Stoe & Cie, 1997) 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).

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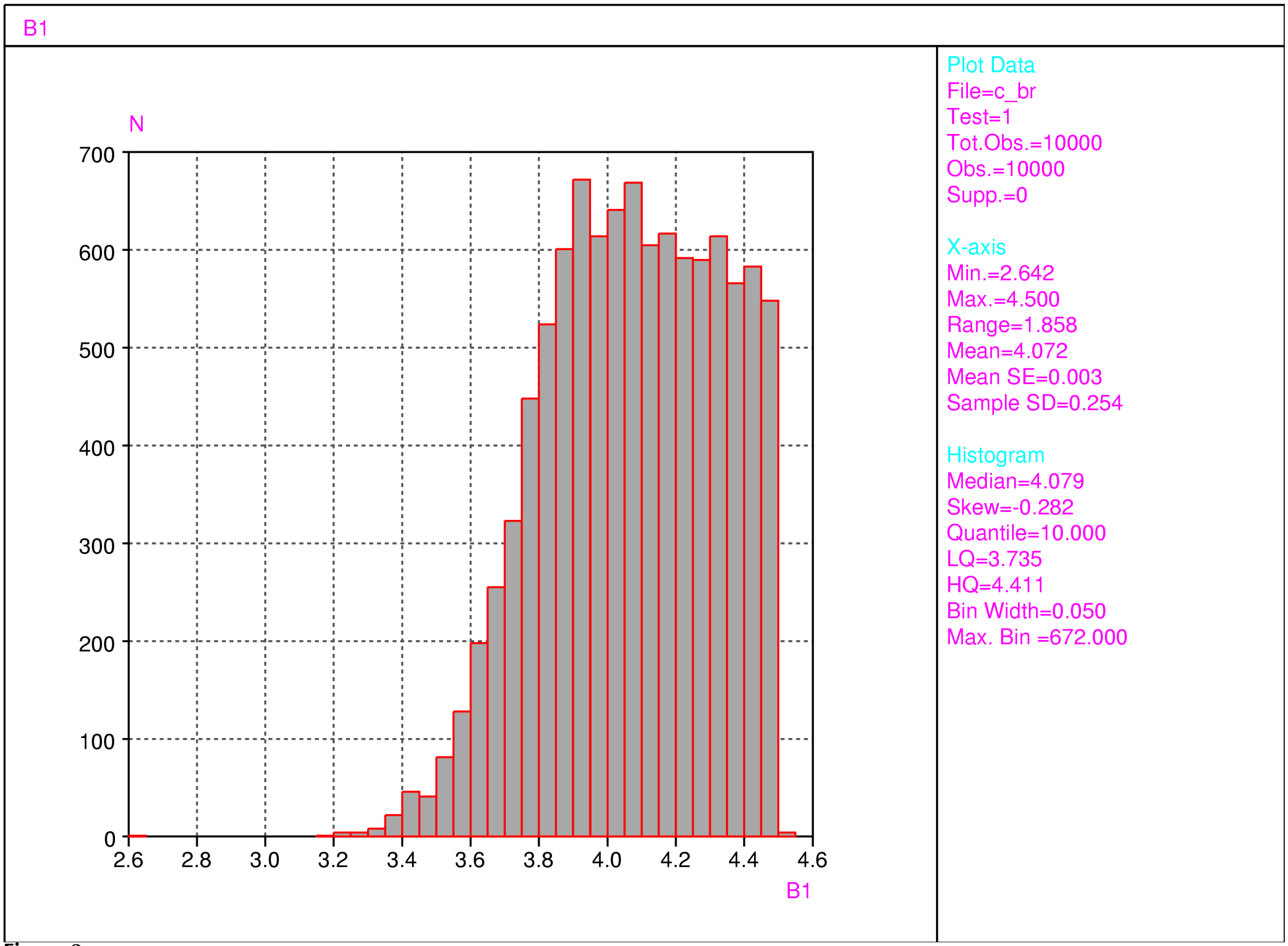

Figure 3

Histogram of intermolecular aromatic C—Br distances where the Br is a substituent on the aromatic ring. Data from the CSD autumn 2001 release (Allen & Kennard, 1993).

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

Eriksson, J., Jakobsson, E, Marsh, G. & Bergman, Å. (2001). *Proceedings of the Second International Workshop on Brominated Flame Retardants*, May 14–16, 2001, Stockholm University.

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

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

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

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

Stoe. (1997). *IPDS* (Version 2.87) and *X-RED* (Version 1.09). Stoe and Cie GmbH, Darmstadt, Germany.

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

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