Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Phenyl 2,4,6-tribromophenyl 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) = 0.006 Å
R factor = 0.033
wR factor = 0.065
Data-to-parameter ratio = 16.7

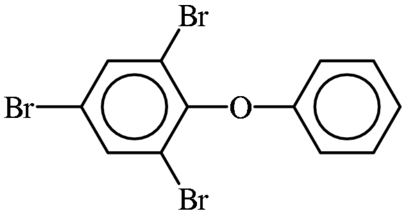
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 August 2001
Accepted 7 September 2001
Online 20 September 2001

The title compound, $C_{12}H_7Br_3O$, is the third well characterized of a total of 209 different brominated diphenyl ethers. The bromine-substituted rings pack in the crystal in a common plane with the same ring from symmetry-related neighbouring molecules. The short Br···Br contact distance [3.519 (2) Å], together with a pair of considerably longer Br···Br contact distances [3.966 (2) Å], may be part of a model describing this packing.

Comment

One of the most important groups of flame retardants is that of the polybrominated diphenyl ethers (PBDE). There are 209 possible brominated congeners, but most of the commercially available mixtures consist of highly brominated congeners such as decabromodiphenyl ether (Eriksson *et al.*, 1999). Brominated diphenyl ethers are additive flame retardants, which means that they are only mixed together with plastic material and therefore they migrate more easily to the environment than if they had been covalently bonded with the polymer material (Kuryla & Papa, 1979). The number of known PBDEs without any heterosubstituent, such as hydroxyls *etc.*, is rather limited. In the autumn 2001 release of the Cambridge Structural Database (CSD; Allen & Kennard, 1993), only three PBDE's are listed and one of these without coordinates. Including hetero substituents other than bromine gives a larger set of structures for use as model compounds, but still only of the order of 10–15 structures. One salient feature of the PBDEs is that they are often not found in the environment in proportion to their use. A possible reason for this is that they are decomposed by sunlight, radicals or some other reactions in the environment (Örn *et al.*, 1996). It is a long-term goal to try to model the reactivity of different PBDEs that are often found deposited on solid soot particles *etc*. Thus an accurate model of the crystal structure is important.



(I)

The packing of the title compound, (I) (Fig. 1), shows some interesting features. The brominated ring (C1–C6) is planar within 0.005 Å, with Br1 deviating by 0.034 (6) Å, Br2 in the

DOI: 10.1107/S1600536801014702

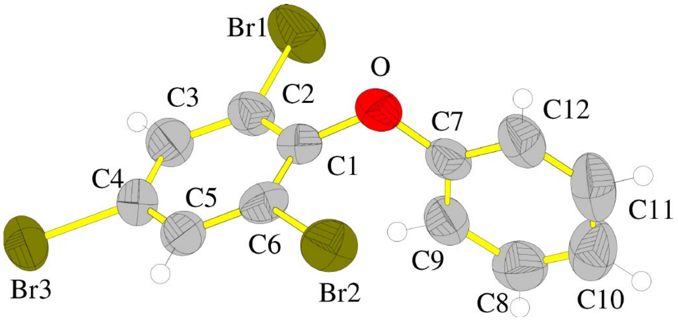


Figure 1
The molecule of the title compound with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

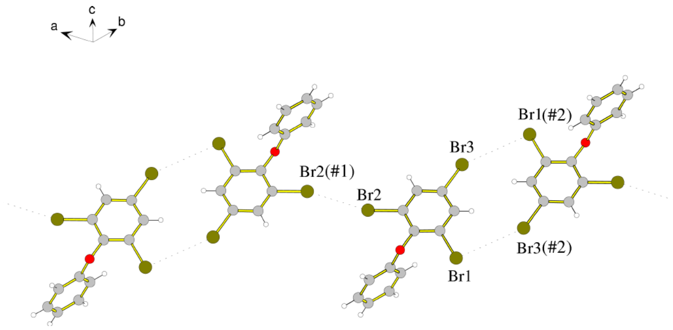


Figure 2
Packing diagram of the title compound [symmetry codes: (#1) $-x + 2$, $-y + 1$, $-z+ + 1$; (#2) $-x$, $-y+2$, $-z + 1$].

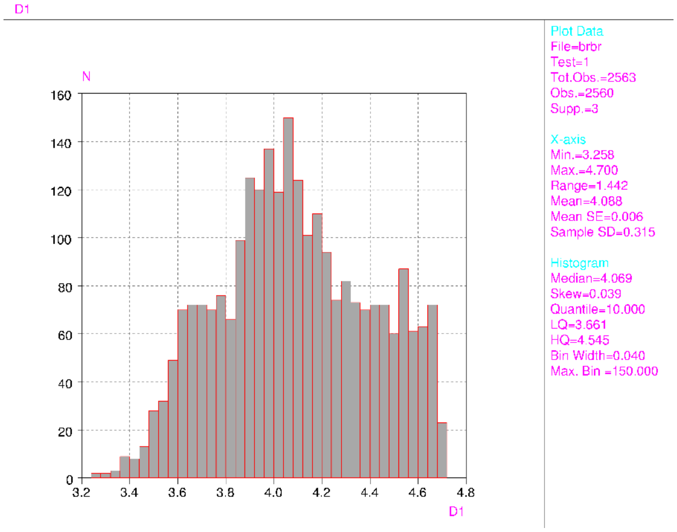


Figure 3
Histogram of intermolecular Br···Br distances where Br is an aromatic ring substituent. Data are from the CSD autumn 2001 release (Allen & Kennard, 1993).

ring plane, Br3 deviating by 0.036 (5) Å and the O atom deviating by 0.084 (5) Å from the ring plane; the other benzene ring is planar within <0.01 Å, also with the O atom in the plane. The angle between the ring planes is 89.8 (1)°. The molecules pack in such a way that bromine-substituted rings from different molecules all occupy the same plane (Fig. 2).

The C atoms of the constituent Br-substituted rings deviate by <0.01 Å from the ring plane. At least one of the Br contacts, Br2···Br2($-x + 2$, $-y + 1$, $-z + 1$), is rather short at 3.519 (2) Å, while the next longer contact, Br3···Br1($-x$, $-y + 2$, $-z + 1$), is 3.966 (2) Å. The first of these Br···Br contacts is considered short compared with similar distances from a search of all intermolecular Br···Br distances in Br-substituted aromatic compounds in the CSD (Allen & Kennard, 1993), shown in Fig. 3. Whether the packing of the molecules in the crystal is an effect of the interhalogen bonding or if the short halogen contacts are consequences of the packing in the crystal is an open question.

Experimental

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

Crystal data

$C_{12}H_7Br_3O$
$M_r = 406.91$
Triclinic, $P\bar{1}$
$a = 5.994$ (2) Å
$b = 10.182$ (5) Å
$c = 11.479$ (5) Å
$\alpha = 109.43$ (5)°
$\beta = 99.76$ (4)°
$\gamma = 97.37$ (5)°
$V = 638.1$ (3) Å^3
$Z = 2$
$D_x = 2.118$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1255 reflections
$\theta = 1.7$–25.0°
$\mu = 9.46$ mm^{-1}
$T = 293$ (2) K
Irregular, colourless
0.24 × 0.15 × 0.13 mm

Data collection

Stoe IPDS diffractometer
Area-detector scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1997)
$T_{\min} = 0.104$, $T_{\max} = 0.303$
9866 measured reflections
2425 independent reflections
1701 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.055$
$\theta_{\max} = 25.9°$
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.066$
$S = 1.23$
2425 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.59$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.884 (4)	O—C1	1.381 (4)
Br2—C6	1.894 (4)	O—C7	1.394 (4)
Br3—C4	1.907 (4)		
C1—O—C7	117.3 (3)	C9—C7—O	123.2 (3)
O—C1—C6	121.7 (4)	C12—C7—O	115.5 (4)
O—C1—C2	119.7 (3)		
C7—O—C1—C6	78.7 (4)	C1—O—C7—C9	26.2 (5)
C7—O—C1—C2	−107.1 (4)	C1—O—C7—C12	−155.5 (3)

Data collection: *EXPOSE* in *IPDS Software* (Stoe, 1997); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; 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.

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