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Rerefinement of decabromodiphenyl ether from new area-detector data

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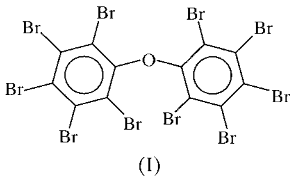
Refinement of the title compound, bis(pentabromophenyl) ether, $C_{12}Br_{10}O$, from 100 K CCD data yields physically reasonable anisotropic displacement parameters for the C atoms. The C—Br distances are in the range 1.867 (4)–1.891 (4) Å and the dihedral angle formed by the phenyl rings is 78.3 (2)°. All endocyclic C—C—C angles are within 0.8 (4)° of the ideal value of 120°.

Comment

Ericksson *et al.* (1999) recently reported the structure of the title flame-retardant compound, (I), at 100 K. They were unable to refine the C atoms with anisotropic displacement parameters, as some became non-positive definite. They observed an asymmetry in the O—C—C angles to both rings, as well as a spread of 117.3 (5)–122.0 (4)° among the endocyclic angles within phenyl ring C1–C6, but no such asymmetry in phenyl ring C7–C12. The asymmetry was attributed to intermolecular Br···Br contacts.



(I)

With serial diffractometer (CAD-4) data at both 100 and 150 K, we likewise experienced problems treating the C atoms of the title compound as anisotropic and observed that angles O1—C6—C5 and O1—C7—C12 were 5–10° larger than O1—C6—C1 and O1—C7—C8, but did not observe the asymmetric endocyclic bond angles. We have now refined the structure *versus* CCD data at 100 K, which yielded physically reasonable ellipsoids for the C atoms. Further, all endocyclic C—C—C angles are within 0.8° (2σ) of the ideal value of 120°. We do find the asymmetry in the O—C—C angles, however, it is also present in 1-bromo-4-(4-bromophenoxy)benzene (Mrse *et al.*, 1998), and is thus unrelated to the perbromination of the title compound.

It is noteworthy that the smallest endocyclic C—C—C angle found by Ericksson *et al.* (1999) is at the C atom (C2) with the shortest C—Br distance, and likewise the largest C—C—C angle at a brominated site is at C3, which has the longest C—Br distance. This implies that the observed spread in C—C—C angles is likely a result of slightly misplaced C atoms, or of underestimation of the uncertainties in the C positions.

The cell dimensions of the title compound at 150 K are a = 8.3194 (8), b = 8.9602 (7), c = 14.3638 (11) Å, α = 80.368 (7), β = 84.494 (8), γ = 63.140 (8)°; V = 941.5 (2) $Å^3$; those at 297 K are a = 8.4295 (13), b = 9.0108 (6), c = 14.4141 (15) Å, α = 80.742 (11), β = 85.009 (14), γ = 62.848 (10)°; V = 961.4 (3) $Å^3$.

Experimental

The title compound was kindly provided by the Albemarle Corporation. Single crystals were obtained by slow evaporation of a solution in dichlorobenzene.

Crystal data

$C_{12}Br_{10}O$
M_r = 959.22
Triclinic, $P\overline{1}$
a = 8.3070 (5) Å
b = 8.9554 (4) Å
c = 14.3472 (8) Å
α = 80.407 (3)°
β = 84.441 (2)°
γ = 63.194 (2)°
V = 938.97 (9) $Å^3$

Z = 2
D_x = 3.393 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 388 reflections
θ = 2.5–40.2°
μ = 21.34 mm^{-1}
T = 100 K
Prism, colourless
0.10 × 0.07 × 0.05 mm

Data collection

KappaCCD diffractometer (with Oxford Cryosystems Cryostream)
ω scans with κ offsets
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.140, T_{max} = 0.344
12 388 measured reflections

10 939 independent reflections
4884 reflections with $I > 2\sigma(I)$
R_{int} = 0.095
θ_{max} = 40.2°
$h = 0 \rightarrow 15$
$k = -14 \rightarrow 15$
$l = -25 \rightarrow 26$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.085
S = 0.892
10939 reflections
209 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0227P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.01 e $Å^{-3}$
$\Delta\rho_{min}$ = −1.81 e $Å^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00156 (12)

The coordinates of Eriksson *et al.* (1999) were used as a starting model, and were translated by $x + \frac{1}{2}$ to avoid most negative values. The largest final difference peak is located 0.78 Å from Br7, and the deepest hole is located 0.55 Å from Br10.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: coordinates of Eriksson *et al.* (1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick,

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1997); software used to prepare material for publication: *SHELXL*97.

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References

Eriksson, J., Eriksson, L. & Jakobsson, E. (1999). *Acta Cryst.* C**55**, 2169–2171.
Mrse, A. A., Lee, Y., Bryant, P. L., Fronczek, F. R., Simeral, L. S. & Butler, L. G. (1998). *Chem. Mater.* **10**, 1291–1300.
Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
Sheldrick, G. (1997). *SHELXL*97. University of Göttingen, Germany.