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2,2′,6,6′-Tetrabromobiphenyl

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.013 Å
R factor = 0.048
wR factor = 0.127
Data-to-parameter ratio = 15.8

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

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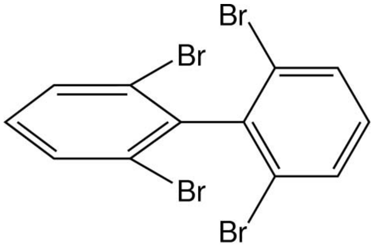
The title compound, $C_{12}H_6Br_4$, is isostructural with the corresponding Cl analogue. The molecule lies on a twofold rotation axis which runs perpendicular to the central C—C bond and the dihedral angle between the benzene rings is 85.2 (2)°.

Comment

We are currently interested in the structural analysis of 1,8-aryl-substituted biphenylenes, which are of potential interest for the study of π–π interactions (Cozzi & Siegel, 1995). The synthesis of such compounds requires suitable *o*,*o*′-tetrahalobiphenyl derivatives. The title compound, (I), is a common precursor used in this area of chemistry (Rajca *et al.*, 1996), for which we now report the crystal structure.



(I)

Compound (I) is isostructural with the corresponding Cl-containing compound previously characterized by Field *et al.* (1985) and presents the expected geometry (Table 1). The asymmetric unit contains one half-molecule, with a twofold rotation axis running perpendicular to the central C—C bond (Fig. 1). The C1—C1^{i} [symmetry code: (i) $1 - x, y, \frac{1}{2} - z$] bond length of 1.509 (17) Å found in (I) is very close to that found in the Cl analogue (1.494 Å). Thus, the nature of the halogen substituent at positions 2, 6, 2′ and 6′ in the biphenyl system does not modify the geometry of this central core. In contrast, the dihedral angle between the benzene rings clearly depends on the halogen, being 81.7° in the case of the Cl compound *versus* 85.2 (2)° in (I). This difference of *ca* 3.5° is obviously related to the steric demands of the Br atoms compared with that of the Cl atoms. This effect is confirmed by comparing the same dihedral angle for F– and I-containing biphenyl compounds. For instance, an acute angle of 57.4° is observed in the case of 2,2′,4,4′,6,6′-hexafluorobiphenyl (Leroux *et al.*, 2004), while the benzene rings are virtually perpendicular (dihedral angle 90.3°) in the structure of an iodo-derivative, 2,2′,6,6′-tetraiodo-3,3′,4,4′-tetramethoxybiphenyl (Waldvogel *et al.*, 2002).

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Molecules of (I) are stacked in the solid state without significant π–π interactions. The shortest separation between the centroids of two neighbouring rings in the cell (symmetry code: $\frac{3}{2} - x, \frac{1}{2} + y, z$) is 5.0309 (9) Å.

Experimental

Compound (I) was prepared following the published procedure of Rajca *et al.* (1996), and single crystals were obtained by recrystallization from CH_2Cl_2 at 298 K.

Crystal data

$C_{12}H_6Br_4$
$M_r = 469.81$
Orthorhombic, *Pbcn*
$a = 10.615$ (3) Å
$b = 9.8137$ (18) Å
$c = 12.768$ (3) Å
$V = 1330.2$ (5) Å^3
$Z = 4$
$D_x = 2.346$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 64 reflections
$\theta = 4.8$–11.8°
$\mu = 12.07$ mm^{-1}
$T = 298$ (2) K
Irregular fragment, colourless
0.4 × 0.4 × 0.3 mm

Data collection

Bruker *P*4 diffractometer
$\omega/2\theta$ scans
Absorption correction: part of the refinement model (ΔF) [*DIFABS* in *WinGX* (Walker & Stuart, 1983; Farrugia, 1999)]
$T_{\min} = 0.004$, $T_{\max} = 0.027$
2756 measured reflections
1168 independent reflections

729 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.053$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 12$
$k = -11 \rightarrow 8$
$l = -15 \rightarrow 15$
3 standard reflections every 97 reflections intensity decay: 7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.127$
$S = 1.05$
1168 reflections
74 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 5.0627P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.65$ e Å^{-3}
$\Delta\rho_{\min} = -0.45$ e Å^{-3}
Extinction correction: *SHELXTL-Plus* (Sheldrick, 1998)
Extinction coefficient: 0.0024 (5)

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.882 (9)	C1—C1$^{\rm i}$	1.509 (17)
Br2—C6	1.883 (9)		
C2—C1—C6	115.7 (8)	C3—C2—Br1	117.2 (7)
C2—C1—C1$^{\rm i}$	121.1 (8)	C5—C6—C1	122.5 (9)
C6—C1—C1$^{\rm i}$	123.2 (8)	C5—C6—Br2	118.2 (7)
C1—C2—C3	122.7 (8)	C1—C6—Br2	119.3 (7)
C1—C2—Br1	120.1 (7)		

Symmetry code: (i) $-x + 1, y, -z + \frac{1}{2}$.

Although containing a high proportion of Br, the title compound proved to be a poorly diffracting sample [mean value of $I/\sigma(I) = 10.8$ for the data set collected]. Moreover, the sample decomposes in the X-ray beam, affecting the quality of the data (7% decay over a period of 33 h). Data for (I) were thus collected using a rather large sample, yielding a μ × size value larger than 4. Unfortunately, single crystals do not present well shaped faces, preventing an accurate absorption correction based on a numerical absorption procedure. Two strategies were probed. Firstly, using a set of ψ-scans, an isotropic model including Br, C and H atoms converged to $R_1 = 0.175$ for data with $I > 2\sigma(I)$. Secondly, an empirical correction based on ΔF values [*DIFABS* (Walker & Stuart, 1983)] was probed, giving a better refinement ($R_1 = 0.135$). The structure was then further refined with anisotropic displacement parameters for non-H atoms, following routine procedures. H atoms were placed in idealized positions and refined using a riding model, with C—H distances constrained to 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(carrier C atom).

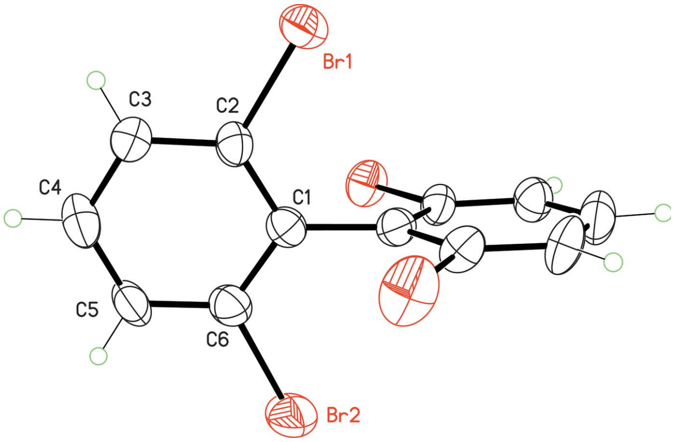

Figure 1
The structure of (I), with displacement ellipsoids at the 30% probability level and the numbering scheme for the asymmetric unit. Unlabelled atoms are related to labelled atoms by the symmetry operator (i) given in Table 1.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL-Plus*; molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXTL-Plus*.

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