

Redetermination of 4,4'-dibromobiphenyl

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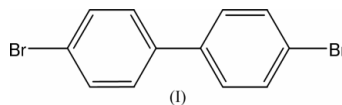
Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å
 R factor = 0.036
 wR factor = 0.077
Data-to-parameter ratio = 15.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{12}\text{H}_8\text{Br}_2$, previously reported by Kronebusch, Gleason & Britton [*Cryst. Struct. Commun.* (1976), **5**, 839–842] has been re-refined against new intensity data. Geometric parameters agree quite well. However, the positions of the hydroxyl H atoms could be determined employing our new data. Furthermore, the results of the present structure determination are of significantly higher precision. There are two almost identical molecules in the asymmetric unit, which show close intermolecular $\text{Br}\cdots\text{Br}$ contacts less than 3.48 Å.

Comment

A perspective view of the title compound, (I), is shown in Fig. 1. The original structure was reported by Kronebusch *et al.* (1976) in space group $P2_1/c$. We have retained the atom numbering of these authors, but we have decided to describe the structure in $P2_1/n$, since the β angle then decreases from 116.6 (2) to 97.32 (3)°. The geometric parameters of both determinations agree quite well (Table 1). However, the present work is of significantly improved precision and we were able to determine the positions of the H atoms. There are two almost identical molecules in the asymmetric unit, which show rather close intermolecular $\text{Br}\cdots\text{Br}$ contacts: $\text{Br1}\cdots\text{Br2}^i = 3.4316$ (10) Å and $\text{Br1}'\cdots\text{Br2}'^i = 3.4782$ (10) Å [symmetry code: (i) $x, 1 + y, z$]. Furthermore, it is interesting to note that the structure of (I) is isostructural with 4,4'-dichlorobiphenyl (Brock *et al.*, 1978), but not with 4,4'-difluorobiphenyl (Halstead *et al.*, 1976).



Experimental

Our aim was to synthesize a long-chain metasiloxane containing an Si—O—Si framework by mixing CoCl_2 , 4,4'-dibromobiphenyl and tetrasodium bis(1,1,3,3,5,5,7,7-octaphenyltetrasiloxanediolate) in pyridine solution. From the product mixture, we isolated suitable single crystals; unfortunately, these proved to be the starting material.

Crystal data

$\text{C}_{12}\text{H}_8\text{Br}_2$
 $M_r = 312.00$
Monoclinic, $P2_1/n$
 $a = 9.7007$ (19) Å
 $b = 14.114$ (3) Å
 $c = 15.610$ (3) Å
 $\beta = 97.32$ (3)°
 $V = 2119.8$ (7) Å³
 $Z = 8$

$D_x = 1.955$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 9816 reflections
 $\theta = 3.4$ – 25.2 °
 $\mu = 7.60$ mm⁻¹
 $T = 173$ (2) K
Block, colourless
 $0.18 \times 0.16 \times 0.11$ mm

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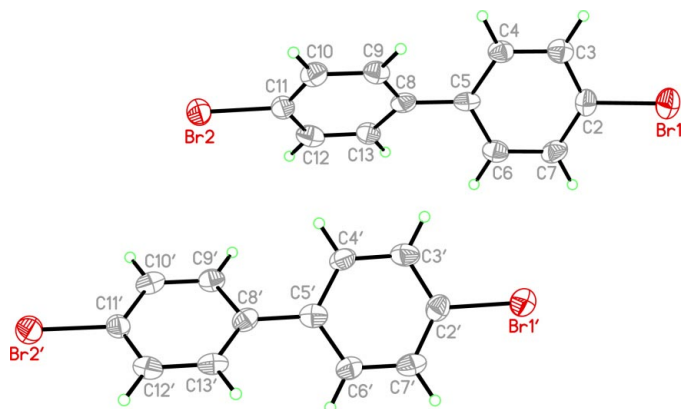


Figure 1
Perspective view of the asymmetric unit of the title compound, with the atom numbering. Displacement ellipsoids are shown at the 50% probability level.

Data collection

Stoe IPDS-II two-circle
diffractometer
 ω scans
Absorption correction: multi-scan
(*MULABS*; Spek, 1990; Blessing,
1995)
 $T_{\min} = 0.241$, $T_{\max} = 0.433$
17434 measured reflections

3820 independent reflections
2578 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.078$
 $\theta_{\text{max}} = 25.3^\circ$
 $h = -11 \rightarrow 11$
 $k = -16 \rightarrow 16$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.077$
 $S = 0.87$
3820 reflections
253 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0364P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.59 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.86 \text{ e } \text{\AA}^{-3}$

Table 1

Comparison of the geometric parameters ($^\circ$, \AA) of the present structure, (I), with those of Kronebusch *et al.* (1976), (II).

	(I)	(II)
$C2-C7 \cdots C8-C13$	38.5 (2)	38.7
$C2'-C7' \cdots C8'-C13'$	42.3 (1)	41.4
$C2-Br1$	1.909 (5)	1.893
$C11-Br2$	1.910 (5)	1.916
$C2'-Br1'$	1.918 (5)	1.886
$C11'-Br2'$	1.912 (5)	1.899
$C5-C8$	1.472 (7)	1.496
$C5'-C8'$	1.491 (7)	1.453

All H atoms could be located unequivocally by difference Fourier synthesis. They were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$], using a riding model with $C-H = 0.99 \text{ \AA}$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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