C7*B*	0.9663 (3)	0.5911 (13)	0.3524 (3)	0.093 (2)
C8*B*	0.8973 (3)	0.4304 (10)	0.3505 (2)	0.079 (2)
C9*B*	0.7566 (3)	0.2915 (7)	0.3819 (2)	0.050 (1)
C11*B*	0.8425 (3)	0.6412 (9)	0.4215 (2)	0.058 (1)
C12*B*	0.8338 (2)	0.4529 (8)	0.3851 (2)	0.053 (1)
C13*B*	0.7056 (2)	0.3088 (8)	0.4290 (2)	0.050 (1)
C14*B*	0.7210 (3)	0.5055 (8)	0.4631 (2)	0.056 (1)
C15*B*	0.7019 (2)	0.3757 (7)	0.3392 (2)	0.047 (1)
H*B*	0.647 (4)	0.671 (12)	0.307 (2)	0.15 (2)

Table 2. *Selected geometric parameters (Å, °)*

Molecule *A*		Molecule *B*	
C9*A*—C12*A*	1.504 (5)	C9*B*—C12*B*	1.497 (5)
C9*A*—C13*A*	1.512 (5)	C9*B*—C13*B*	1.514 (5)
C11*A*—C12*A*	1.377 (5)	C11*B*—C12*B*	1.381 (6)
C13*A*—C14*A*	1.367 (5)	C13*B*—C14*B*	1.384 (5)
C11*A*—O10*A*	1.385 (5)	C11*B*—O10*B*	1.377 (6)
C14*A*—O10*A*	1.379 (5)	C14*B*—O10*B*	1.368 (5)
C9*A*—C15*A*	1.524 (5)	C9*B*—C15*B*	1.509 (5)
C15*A*—O1*A*	1.280 (4)	C15*B*—O1*B*	1.283 (4)
C15*A*—O2*A*	1.214 (4)	C15*B*—O2*B*	1.220 (4)
O1*A*—H*A*	1.09 (8)	O1*B*—H*B*	1.09 (6)
C12*A*—C9*A*—C13*A*	111.2 (3)	C12*B*—C9*B*—C13*B*	111.8 (3)
O10*A*—C11*A*—C12*A*	122.3 (4)	O10*B*—C11*B*—C12*B*	122.9 (4)
C9*A*—C12*A*—C11*A*	121.0 (4)	C9*B*—C12*B*—C11*B*	120.7 (4)
C9*A*—C13*A*—C14*A*	120.8 (4)	C9*B*—C13*B*—C14*B*	120.0 (4)
O10*A*—C14*A*—C13*A*	122.9 (4)	O10*B*—C14*B*—C13*B*	123.2 (4)
C11*A*—O10*A*—C14*A*	118.3 (3)	C11*B*—O10*B*—C14*B*	118.7 (3)
C12*A*—C9*A*—C15*A*	112.3 (3)	C12*B*—C9*B*—C15*B*	111.9 (3)
C13*A*—C9*A*—C15*A*	107.1 (3)	C13*B*—C9*B*—C15*B*	107.5 (3)
C9*A*—C15*A*—O1*A*	115.3 (3)	C9*B*—C15*B*—O1*B*	115.5 (4)
C9*A*—C15*A*—O2*A*	120.7 (3)	C9*B*—C15*B*—O2*B*	121.7 (3)
O1*A*—C15*A*—O2*A*	123.9 (4)	O1*B*—C15*B*—O2*B*	122.8 (4)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H*A*···O2*B*	1.09 (8)	1.61 (8)	2.650 (4)	158 (6)
O1*B*—H*B*···O2*A*	1.09 (6)	1.60 (6)	2.647 (4)	160 (5)

The Laue symmetry and the observed reflection-limiting conditions indicated the space group to be either *Pnma* (centrosymmetric) or $Pna2_1$. The centrosymmetric option was explored without success, but the direct-methods program *SHELXS*86 (Sheldrick, 1985) yielded acceptable initial positions of the C and O atoms in space group $Pna2_1$ (No. 33), which was adopted. Refinement proceeded well and the space group was retained.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data, together with a stereoview of the unit cell, have been deposited with the IUCr (Reference: FG1134). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Accurate Redetermination of Diiodobis(phenylsulfonyl)methane, $(PhSO_2)_2CI_2$

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Abstract

The structure of the title compound, $C_{13}H_{10}I_2O_4S_2$, has been redetermined, with proper treatment of both anomalous dispersion and absorption. The unusual structural features reported in an earlier determination [Stergioudis, Bozopoulos, Kavounis & Rentzeperis (1989). *Z. Kristallogr.* **189**, 57–63] are not reproduced by the present analysis; the structure and conformation of the title compound are now found to be very similar to those of $(PhSO_2)_2CBr_2$; the molecules are joined into chains along the *a* direction by very short I···O interactions [I···O 2.927 (7) Å].

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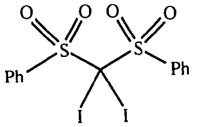
Comment

The structures of $(PhSO_2)_2CH_2$ (I) and $(PhSO_2)_2CBr_2$ (II) have recently been reported by Glidewell, Lightfoot & Patterson (1995). Both (I) and (II) showed significant differences from the reported structure of $(PhSO_2)_2CI_2$ (III) (Stergioudis, Bozopoulos, Kavounis & Rentzeperis, 1989). Although no crystallographic symmetry is imposed upon the molecules, the two independent C(sp^3)—S distances are essentially identical in each of (I) and (II); by contrast, the corresponding C—S distances in (III) are reported to be very different, 1.72 (2) and 1.83 (2) Å. Similarly, the reported values of the two independent O—S—O angles in (III) are 110.0 (8) and 112.4 (7)°, much smaller than the values found in both (I) and (II), and in all other neutral sulfones (Cotton & Stokeley, 1970; Berthou, Jéminet & Laurent, 1972; De Lucchi, Pasquato, Modena & Valle, 1985; Foss, Kvammen & Maröy, 1985), where these angles lie in the range 118.5 (3)–121.0 (3)°. These differences between the structures of (I) and (II) on the one hand and that of (III) on the other would, if confirmed, be of considerable theoretical interest: we have therefore redetermined the structure of (III), and we conclude that the structural details previously reported are incorrect.



(III)

Compound (III) crystallizes in the orthorhombic system, in space group $P2_12_12_1$; the unit-cell dimensions and the space group are identical with those reported earlier (Stergioudis *et al.*, 1989) and confirm that the present work and the previous report refer to the same polymorph of (III). In contrast to the previous report, we find the following (Table 2): the two independent C(sp^3)—S distances are identical within experimental uncertainly, with values very similar to those found in (II); the two independent O—S—O angles lie within the normal range for neutral sulfones; and the non-bonding I···S distances span a much smaller range, 3.155 (3)–3.287 (3) Å, than previously reported, 3.09 (2)–3.42 (2) Å. The overall conformation of (III) (Fig. 1 and Table 2) is remarkably similar to that of (II), despite the fact that these two compounds crystallize in quite different space groups.

None of the anomalous structural features previously reported for (III) by Stergioudis *et al.* (1989) are reproduced in the present analysis. Factors which may have contributed to the erroneous structural details reported earlier include the apparent averaging of Friedel reflections and the absence of any absorption correction, despite the large value of the absorption coefficient. The final $\Delta\rho$ map for (III) for the present structure was devoid of the additional peaks found adjacent to the phenyl rings in the previous account of the structure.

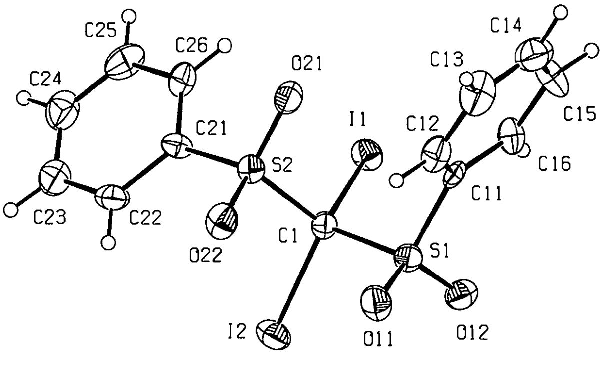


Fig. 1. A view of (III) with our numbering scheme [which has been chosen to be the same as used in $(PhSO_2)_2CBr_2$]. Displacement ellipsoids are drawn at the 30% probability level.

A feature of interest in the crystal structure of (III), not remarked upon by Stergioudis *et al.* (1989), is the very short I1···O11($x - 1, y, z$) distance, 2.927 (7) Å: this is significantly shorter than the sum of the van der Waals radii, 3.45 Å (Bondi, 1964). This I···O distance is even shorter, relative to the van der Waals radii, than the very short I···N distance of 3.127 (4) Å in 4-iodobenzonitrile (Desiraju & Harlow, 1989). Such very short distances involving the heavier halogens have been ascribed to halogen polarizability (Desiraju & Harlow, 1989), and have the effect in (III) of generating chains parallel to the *a* direction.

Experimental

Compound (III) was prepared according to the published method (Hadjiarapoglou & Varvoglis, 1988), and crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in dichloromethane/hexane.

Crystal data

$C_{13}H_{10}I_2O_4S_2$
$M_r = 548.13$
Orthorhombic
$P2_12_12_1$
$a = 6.9936\ (6)$ Å
$b = 13.1255\ (12)$ Å
$c = 17.988\ (6)$ Å
$V = 1651.2\ (5)$ Å^3
$Z = 4$
$D_x = 2.205$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 12.60$–$19.60°$
$\mu = 4.03$ mm^{-1}
$T = 294\ (1)$ K
Needle
0.43 × 0.11 × 0.07 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer

$R_{\text{int}} = 0.038$
$\theta_{\max} = 26.88°$

$\theta/2\theta$ scans
Absorption correction: numerical by Gaussian integration from crystal shape
$T_{\min} = 0.6205$, $T_{\max} = 0.7731$
4000 measured reflections
3577 independent reflections
2172 observed reflections $[I > 2\sigma(I)]$

$h = -8 \rightarrow 8$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 22$
3 standard reflections
frequency: 120 min
intensity decay: no decay, variation 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0481$
$wR(F^2) = 0.1115$
$S = 0.961$
3577 reflections
190 parameters
H atoms riding [*SHELXL*93 (Sheldrick, 1993) defaults, C—H = 0.93 Å]
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.780$ e Å^{-3}
$\Delta\rho_{\min} = -0.548$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = 0.02 (5)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
I1	−0.03715 (10)	0.45206 (6)	0.69784 (4)	0.0473 (2)
I2	0.36708 (12)	0.56682 (6)	0.78267 (4)	0.0540 (2)
S1	0.3804 (4)	0.3456 (2)	0.7135 (2)	0.0399 (6)
S2	0.3417 (4)	0.5246 (2)	0.60340 (15)	0.0383 (6)
O11	0.5807 (10)	0.3623 (6)	0.7088 (5)	0.058 (2)
O12	0.3009 (11)	0.3112 (5)	0.7812 (4)	0.050 (2)
O21	0.2668 (11)	0.4584 (5)	0.5479 (4)	0.050 (2)
O22	0.5423 (11)	0.5410 (6)	0.6072 (4)	0.054 (2)
C1	0.2678 (13)	0.4710 (7)	0.6940 (5)	0.036 (2)
C11	0.3181 (15)	0.2594 (7)	0.6432 (6)	0.037 (3)
C12	0.4463 (17)	0.2432 (9)	0.5857 (7)	0.051 (3)
C13	0.408 (2)	0.1680 (11)	0.5339 (8)	0.071 (4)
C14	0.244 (2)	0.1121 (11)	0.5393 (7)	0.072 (4)
C15	0.1216 (18)	0.1279 (9)	0.5964 (7)	0.061 (4)
C16	0.1569 (17)	0.2025 (8)	0.6484 (6)	0.049 (3)
C21	0.2253 (15)	0.6418 (8)	0.5965 (6)	0.039 (3)
C22	0.3205 (18)	0.7311 (8)	0.6210 (6)	0.053 (3)
C23	0.222 (3)	0.8232 (10)	0.6158 (7)	0.068 (4)
C24	0.038 (3)	0.8246 (10)	0.5880 (7)	0.069 (4)
C25	−0.051 (2)	0.7383 (11)	0.5650 (7)	0.066 (4)
C26	0.0441 (18)	0.6467 (9)	0.5687 (6)	0.049 (3)

Table 2. *Selected geometric parameters (Å, °)*

I1—C1	2.148 (9)	S1—C1	1.857 (9)
I2—C1	2.146 (10)	S2—O21	1.424 (7)
S1—O11	1.420 (7)	S2—O22	1.421 (8)
S1—O12	1.412 (8)	S2—C21	1.745 (11)
S1—C11	1.753 (11)	S2—C1	1.850 (10)
O11—S1—O12	119.3 (5)	O22—S2—C1	106.9 (4)
O11—S1—C1	105.7 (4)	O22—S2—C21	109.3 (5)
O11—S1—C11	107.5 (5)	C1—S2—C21	105.5 (5)
O12—S1—C1	106.2 (5)	I1—C1—I2	111.4 (4)
O12—S1—C11	108.5 (5)	I1—C1—S1	108.2 (4)
C1—S1—C11	109.3 (4)	I1—C1—S2	110.5 (5)
O21—S2—O22	119.3 (5)	I2—C1—S1	104.0 (4)
O21—S2—C1	106.4 (4)	I2—C1—S2	110.0 (4)
O21—S2—C21	108.5 (5)	S1—C1—S2	112.7 (5)
O22—S2—C1—S1	−62.6 (6)	O12—S1—C1—S2	−175.9 (5)
O21—S2—C1—S1	65.9 (6)	O11—S1—C1—S2	56.4 (6)
C21—S2—C1—S1	−179.0 (5)	C11—S1—C1—S2	−59.0 (6)

As the molecule contains two I atoms and the space group is chiral, two adjacent octants of data were collected to allow an unequivocal determination of the chirality of the crystal studied. Examination of the structure with *PLATON* (Spek, 1995*a*) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *DIFRAC* (Gabe & White, 1993). Cell refinement: *DIFRAC*. Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: Patterson heavy-atom method and *NRCVAX*94. Program(s) used to refine structure: *NRCVAX*94 and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*94, *PLATON* (Spek, 1995*a*), *PLUTON* (Spek 1995*b*), and *ORTEP*II (Johnson, 1976) as implemented in *PLATON*. Software used to prepare material for publication: *NRCVAX*94, *SHELXL*93 and *WordPerfect* macro *PREPCIF*.

GF thanks NSERC (Canada) for Research Grants.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1343). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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