

2,3,5,6-Tetrafluoro-1,4-diiodobenzene–dimethyl sulfoxide (1/1)

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Key indicators

Single-crystal X-ray study

T = 297 K

Mean $\sigma(\text{C}-\text{C}) = 0.013 \text{ \AA}$

R factor = 0.045

wR factor = 0.125

Data-to-parameter ratio = 14.1

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

Tetrafluorodiiodobenzene and dimethyl sulfoxide (DMSO) crystallize in a 1/1 complex, $\text{C}_6\text{F}_4\text{I}_2 \cdot \text{C}_2\text{H}_6\text{OS}$, in space group *Pbca*. Infinite $\cdots\text{C}_6\text{F}_4\text{I}_2 \cdots \text{O} \cdots \text{IC}_6\text{F}_4 \cdots$ chains are held together by strong $\text{I} \cdots \text{O}$ contacts. The DMSO molecules, which lie on mirror planes, are disordered, giving rise to two $\text{I} \cdots \text{O}$ contacts of 2.898 (9) and 2.803 (10) Å, both with approximately linear $\text{C}-\text{I} \cdots \text{O}$ arrangements. The $\text{C}_6\text{F}_4\text{I}_2$ molecules lie on inversion centers.

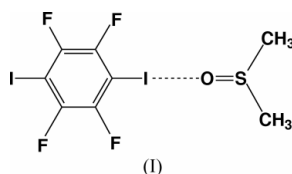
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Comment

In the course of preparing the complex between dicyanodurene and tetrafluorodiiodobenzene (Britton & Gleason, 2002), a similar complex, (I), of DMSO and tetrafluorodiiodobenzene was discovered. Its structure is reported here.



The labeling and the anisotropic displacement parameters are shown in Fig. 1. The bond lengths and angles in the diiodide are normal; within experimental error they are the same as those in the dicyanodurene complex or in the high-temperature polymorph of tetrafluorodiiodobenzene itself (Chaplot *et al.*, 1981).

The packing is shown in Fig. 2. There are zigzag $\text{I}-\text{C}_6\text{F}_4-\text{I} \cdots \text{O} \cdots \text{I}-\text{C}_6\text{F}_4-\text{I} \cdots \text{O} \cdots$ chains parallel to the *b* axis. The geometry of the interaction is consistent with the $\text{I} \cdots \text{O}$ interaction having a Lewis acid–base character [$\text{C}-\text{I} \cdots \text{O} = 173.0$ (8) and 174.9 (8)°, $\text{I} \cdots \text{O} = 2.898$ (9) and 2.803 (9) Å, and $\text{I} \cdots \text{O}-\text{S} = 131.0$ (7) and 122.6 (7)°; the two values given in

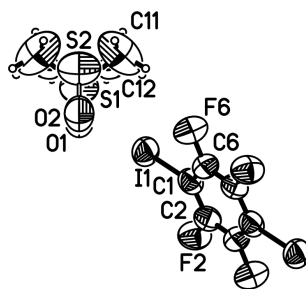


Figure 1

$\text{C}_6\text{F}_4\text{I}_2/\text{DMSO}$. Displacement ellipsoids are shown at the 50% probability level. The unlabeled atoms are related to the labeled atoms by a center of symmetry in the $\text{C}_6\text{F}_4\text{I}_2$ molecule and by a mirror plane in the disordered DMSO molecule.

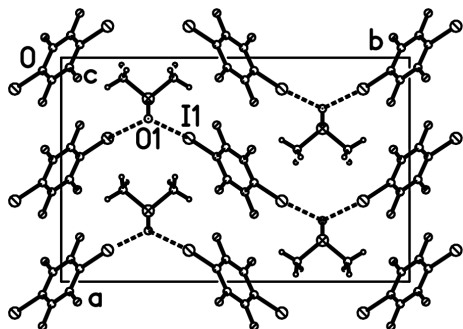


Figure 2

A view of the structure of (I) along the *c* axis. I...O interactions are shown as dotted lines. Only the more abundant of the disordered DMSO are shown.

each case are for the major and minor components of the disorder, respectively]. The only other I...O interaction involving C₆F₄I₂, in *p*-tetrafluorodiodobenzene/4,4'-dipyridyl-*N,N'*-dioxide (Messina *et al.*, 2001), has an I...O distance of 2.754 (2) Å; this is the shortest intramolecular I...O distance yet reported. It is possible that the longer distance found in the DMSO complex is a consequence of the dibasic character of the O atom in DMSO compared with the monobasic character of the O atom in the dipyridyldioxide.

Experimental

The diiodide was obtained from Aldrich Chemical Co. Inc. In an attempt to recrystallize it from DMSO, crystals of the title complex were obtained instead.

Crystal data

C₆F₄I₂·C₂H₆OS
M_r = 479.99
 Orthorhombic, *Pnma*
a = 11.819 (3) Å
b = 18.418 (5) Å
c = 6.0750 (15) Å
V = 1322.4 (6) Å³
Z = 4
D_x = 2.411 Mg m⁻³

Mo *Kα* radiation
 Cell parameters from 24 reflections
 θ = 16.5–19.7°
 μ = 4.94 mm⁻¹
T = 297 (2) K
 Needle, colorless
 0.50 × 0.15 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: ψ scans (North *et al.*, 1968)
T_{min} = 0.42, *T_{max}* = 0.48
 2820 measured reflections
 1340 independent reflections
 673 reflections with *I* > 2 σ (*I*)

R_{int} = 0.065
 θ_{max} = 26.0°
h = 0 → 14
k = 0 → 22
l = -7 → 7
 3 standard reflections
 frequency: 70 min
 intensity decay: less than 1%

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.045
wR(*F*²) = 0.125
S = 0.92
 1340 reflections
 95 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.051P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.78 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.59 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXTL*
 Extinction coefficient: 0.0029 (4)

The DMSO complex is disordered. Two orientations, inverted with respect to one another, were refined with variable occupancies. The bond distances and angles were constrained to have normal values. The S1 orientation (see Fig. 1) had an occupancy of 0.64 (2). The anisotropic displacement ellipsoids are large. The four highest peaks in the final difference map are in the DMSO region rather than near the I atoms where the peaks might be expected. Together, these observations suggest that the disorder is not completely modeled, but no better model could be found. Presumably, the O-atom position is reasonably well defined by the I...O interactions, but the rest of the molecule is only loosely constrained. H atoms were treated as riding, with C–H = 0.96 Å and *U_{iso}*(H) = 1.5*U_{eq}*(C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1983); cell refinement: *CAD-4 Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structure: *TEXSAN*, *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1994); molecular graphics: *TEXSAN* and *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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