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(2-Methoxy-5-methylphenyl)(4-methoxyphenyl)iodonium trifluoroacetate

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The asymmetric unit of the title compound $C_{15}H_{16}IO_2{}^+\cdot CF_3CO_2{}^-$, comprises an iodonium cation and a trifluoroacetate anion. The benzene rings are inclined at an angle of 87.76 (5)° to one another. There is evidence for strong electrostatic interactions between the I atom and two adjacent trifluoroacetate counter-ions.

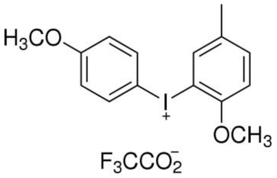
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.035
wR factor = 0.079
Data-to-parameter ratio = 14.6

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

Comment

Diaryliodonium salts are useful in organic synthesis for arylation of organic and inorganic bases (Shah *et al.* 1997; Shah *et al.*, 1998). The title compound, (I) (Fig. 1), is an important representative of such reagents. The iodine atom lies almost in the plane of both benzene rings with deviations of 0.022 (3) and 0.015 (3) Å from the C1–C6 and C9–C14 mean planes, respectively. These rings are nearly orthogonal, with a dihedral angle between them of 87.76 (5)°.



(I)

Each I atom interacts with two O atoms from one adjacent trifluoroacetate anion and one O atom from another, giving the I atoms a distorted trigonal–bipyramidal geometry. Inversion-related pairs of iodonium cations and trifloroacetate anions form cyclic units (Fig. 2). Similar interactions have been observed previously in iodonium salts (Hinkle & McDonald, 2002). The distances I1—O3, I1—O3^i and I1—O4^i [symmetry code: (i) 2 − *x*, 1 − *y*, 1 − *z*] are 2.860 (3), 3.318 (4) and 2.961 (4) Å, respectively. Weak intermolecular C—H···O interactions further stabilize the structure (Table 1).

Experimental

Sodium perborate tetrahydrate (35.39 g, 230 mmol) was added in batches to a stirred mixture of 2-iodo-4-methylanisole (5.71 g, 23 mmol) in acetic acid (100 ml) and acetic anhydride (50 ml) at 318 K. The suspension was stirred for 4.5 h at 318 K, diluted with 600 ml of water and extracted three times with dichloromethane. The organic extracts were dried with sodium sulfate, the solvent removed in vacuum and the residue crystallized from diethyl ether to obtain 2-methoxy-5-methylbis(acetoxy)iodobenzene.

Trifluoromethanesulfonic acid (0.63 ml, 8.2 mmol) was added dropwise to a stirred suspension of this product (1.5 g, 4.1 mmol) in dichloromethane (50 ml) at 263 K under nitrogen. The mixture was

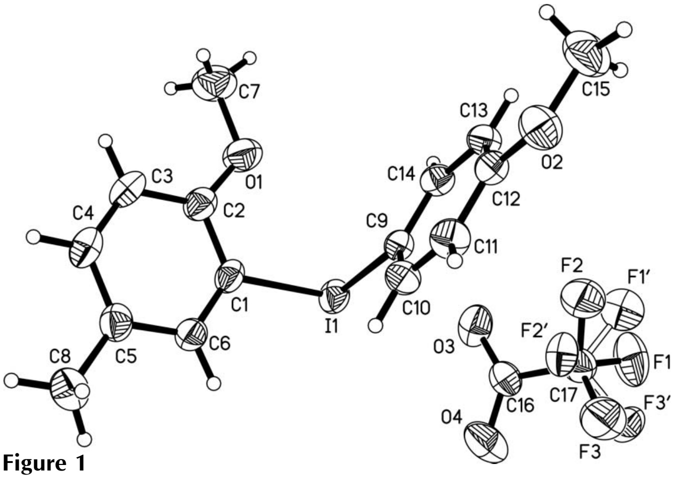


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Open bonds connect atoms of the minor disorder component of the CF_3 group.

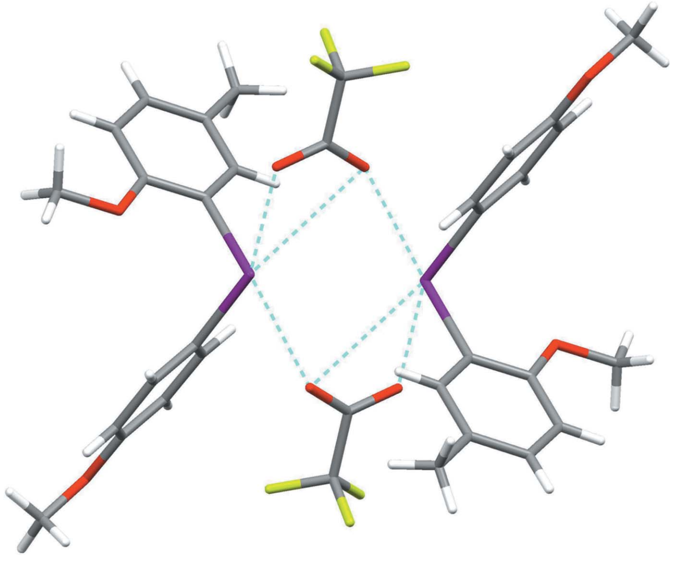


Figure 2
A view of the cyclic units formed by I···O interactions in (I), drawn as dashed lines.

stirred for 30 min at 263 K, then at room temperature for a further 1.5 h, cooled to 263 K and anisole (0.45 ml, 4.1 mmol) added dropwise *via* a syringe. The mixture was stirred at 263 K for 1 h and then at room temperature overnight, solvent was removed in vacuum and the residue crystallized from diethyl ether. Crystals suitable for X-ray analysis (m.p. 436–438 K) were obtained by slow evaporation of a dichloromethane/dimethyl ether mixture (10:1 *v*:*v*).

Crystal data

$C_{15}H_{16}IO_2^+\cdot C_2F_3O_2^-$
$M_r = 468.20$
Monoclinic, $P2_1/n$
$a = 12.805$ (3) Å
$b = 10.620$ (2) Å
$c = 14.334$ (3) Å
$\beta = 109.803$ (3)°
$V = 1833.9$ (6) Å^3
$Z = 4$
$D_x = 1.696$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.79$ mm^{-1}
$T = 294$ (2) K
Block, colorless
0.26 × 0.24 × 0.20 mm

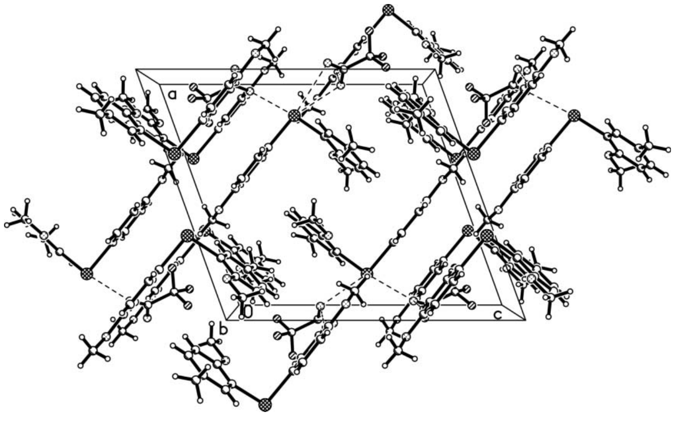


Figure 3
The crystal packing for (I). Dashed lines represent intermolecular I···O interactions.

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{min} = 0.644$, $T_{max} = 0.716$
10107 measured reflections
3744 independent reflections
2099 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 26.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.079$
$S = 0.99$
3744 reflections
257 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 0.6825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O3^{i}	0.93	2.48	3.297 (6)	147
C13—H13···O4^{ii}	0.93	2.49	3.368 (6)	158

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

F atoms in the CF_3 group were found to be disordered over two positions. Their occupancy factors refined to 0.881 (7) and 0.119 (7). The C—F distance was restrained to 1.37 (1) Å.

All H atoms bound to carbon were positioned geometrically and refined using a riding model with C—H = 0.93 Å, $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic and C—H = 0.96 Å, $U_{iso}(H) = 1.5U_{eq}(C)$ for CH_3 atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

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