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Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.046
 wR factor = 0.133
Data-to-parameter ratio = 15.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

1,1'-(1,4-Butanediyl)bis(tetrahydrofuranium) trifluoromethanesulfonate

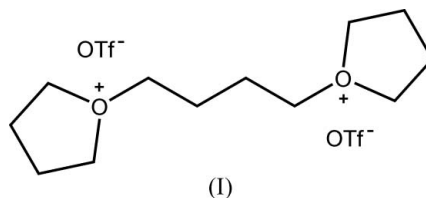
The title compound, $\text{C}_{12}\text{H}_{24}\text{O}_2^{2+} \cdot 2\text{CF}_3\text{O}_3\text{S}^-$, is the first bisoxonium salt for which a crystal structure is reported. The cation is located on an inversion centre and it features a nonplanar C_3O^+ oxonium unit where the O atom is displaced by 0.375 (2) Å from the plane of its substituents.

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Comment

It is well known that organosilyl trifluoromethanesulfonates (OTfs), such as Me_3SiOTf , are potent initiators for the cationic polymerization of tetrahydrofuran (THF) (Hrkach & Matyjaszewski, 1995). The title compound, (I), obtained accidentally from a THF solution of *t*-BuH₂SiOTf (Uhlig & Tzschach, 1989) as colourless hygroscopic crystals, can be regarded as an oligomeric intermediate of the THF polymerization. The same bisoxonium salt has been reported previously from the reaction of triflic acid anhydride with THF but was not characterized by X-ray crystallography at that time (Smith *et al.*, 1977).



The molecular structure of (I) is shown in Fig. 1. The cation is centrosymmetric and features a nonplanar C_3O^+ oxonium unit, where the O atom is displaced by 0.375 (2) Å from the plane of its substituents. The shortest oxonium-to-trifluoromethanesulfonate $\text{O} \cdots \text{O}$ distance of 3.200 (4) Å is slightly longer than the sum of the van der Waals radii (3.04 Å; Bondi, 1964). The cations and anions are arranged into homoionic columns running along the crystallographic *c* axis.

Experimental

Crystals of (I) suitable for structure analysis were obtained directly from a THF solution of *t*-BuH₂SiOTf prepared according to the literature procedure (Uhlig & Tzschach, 1989).

Crystal data

$\text{C}_{12}\text{H}_{24}\text{O}_2^{2+} \cdot 2\text{CF}_3\text{O}_3\text{S}^-$
 $M_r = 498.45$
Monoclinic, $P2_1/c$
 $a = 6.3708$ (14) Å
 $b = 12.542$ (3) Å
 $c = 13.334$ (3) Å
 $\beta = 102.765$ (5)°
 $V = 1039.1$ (4) Å³

$Z = 2$
 $D_x = 1.593$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.35$ mm⁻¹
 $T = 173$ (2) K
Plate, colourless
 $0.50 \times 0.15 \times 0.06$ mm

Data collection

Bruker SMART CCD area-detector
 diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2000)
 $T_{\min} = 0.789$, $T_{\max} = 0.979$

8382 measured reflections
 2249 independent reflections
 1708 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.077$
 $\theta_{\text{max}} = 27.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.133$
 $S = 1.11$
 2249 reflections
 146 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.1747P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.58 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|--------------------------|-------------|-------------|-------------|
| C1—O1 | 1.492 (3) | C5—O1 | 1.494 (2) |
| C4—O1 | 1.492 (3) | | |
| C4—O1—C1 | 108.62 (15) | C1—O1—C5 | 115.23 (16) |
| C4—O1—C5 | 117.84 (16) | | |
| O1—C5—C6—C6 ⁱ | -59.6 (3) | C6—C5—O1—C4 | -65.5 (2) |
| C3—C4—O1—C5 | -167.5 (3) | C6—C5—O1—C1 | 164.06 (18) |
| C2—C1—O1—C5 | 154.3 (2) | | |

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

One of the C atoms of the tetrahydrofuranium ring is disordered over two positions, C3 and C3', with occupancy factors of 0.73 (2) and 0.27 (2), respectively. All H atoms were introduced at calculated positions and constrained to ride on their parent atoms with C—H distances of 0.99 \AA and $U_{\text{eq}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Bergerhoff *et al.*, 1996); software used to prepare material for publication: SHELXL97.

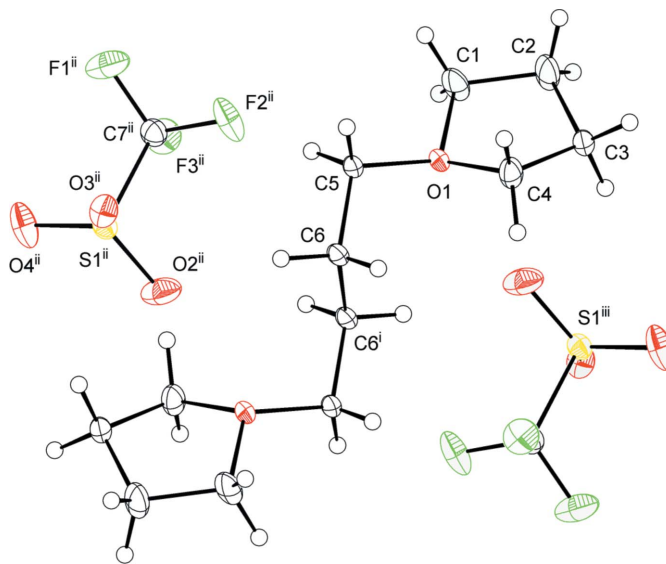


Figure 1

View of (I), showing the labelling of the atoms. The minor position of the disordered atom C3 is not shown. Displacement ellipsoids are shown at the 30% probability level. [Symmetry codes: (i) $1 - x, 2 - y, 1 - z$; (ii) $-x, 1 - y, 1 - z$; (iii) $1 + x, 1 + y, z$.

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