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### 1,3,5,7-Tetrakis(trifluoromethyl)-2,6-dioxabicyclo[3.3.0]octane-3,7-diol

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## Abstract

There are two molecules in the asymmetric unit of  $C_{10}H_6F_{12}O_4$  that differ primarily in their packing and hydrogen bonding. The molecules are linked in groups of four by the hydrogen bonding between the hydroxyls about an inversion center. In one of the independent molecules, one hydroxyl is a hydrogen donor and the other is an acceptor, while in the second molecule the hydroxyls act as both donor and acceptor.

## Comment

A number of crystal structures of substituted 2,6-dioxabicyclo[3.3.0]octanes have been reported, e.g. the methyl glycoside of 2-C-benzyl-3-keto-L-lyxo-hexulo-

sonic acid lactone (Hvoslef & Nordenson, 1976), 3a,6a-dimethyl-2,3,3a,5,6,6a-hexahydrofuro[3,2-b]-furan-2,5-dione (Caira, Watson, Kagan & Singh, 1984), 1,4:3,6-dihydro-D-glucitol (van Koningsveld, Peters & Jansen, 1984), methyl 3,6-anhydro- $\beta$ -D-glucofuranoside (Kopf & Koll, 1984) and  $\beta$ , $\beta$ -D-thero-3,4-hexodiulose (Angyal, Craig & Kusmann, 1989). Comparison of the nonplanar fused rings shows a range of conformations. In the title compound the torsion angles O2—C1—C5—O6 and C4—C5—C1—C8 about the bridge are 94.1 and  $-138.2^\circ$ , respectively, for one molecule, and 94.1 and  $-138.5^\circ$ , respectively, for the other molecule in the asymmetric unit. In the references listed above these torsion angles range from 82.3 to 110.1° and from  $-141.4$  to  $-123.4^\circ$ , respectively.

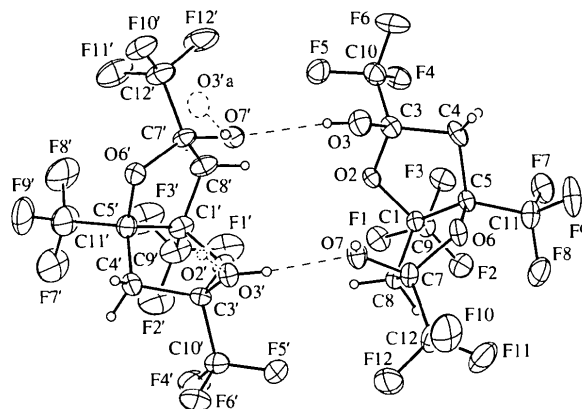
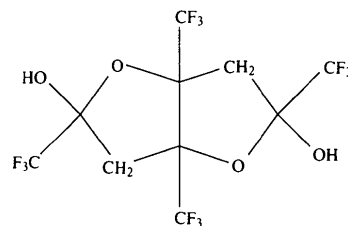


Fig. 1. A displacement ellipsoid plot of the title compound with ellipsoids drawn at the 20% probability level. Dotted lines represent hydrogen bonds and O3'a is a symmetry-related atom.

## Experimental

Strontium hexafluoroacetylacetonate  $[Sr(HFA)_2]$ , from a reaction between excess H(HFA) and strontium, was purified by washing with excess H(HFA) (Purdy, Berry, Holm, Fatemi & Gaskill, 1989). After recovery of the H(HFA) from the filtrate on the vacuum line, the orange residue was dissolved in ether, transferred to a stoppered vial and set aside in the 'dri-lab'. Three years later, the ether had evaporated and small colorless crystals formed.

## Crystal data

C<sub>10</sub>H<sub>6</sub>F<sub>12</sub>O<sub>4</sub>*M<sub>r</sub>* = 418.13

Monoclinic

C2/c

*a* = 23.462 (5) Å*b* = 10.600 (2) Å*c* = 23.907 (6) Å

β = 102.08 (2)°

*V* = 5814 (2) Å<sup>3</sup>*Z* = 16*D<sub>x</sub>* = 1.911 Mg m<sup>-3</sup>

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 10–13°

μ = 0.24 mm<sup>-1</sup>*T* = 295 K

Irregular

0.40 × 0.40 × 0.25 mm

Clear colorless

## Data collection

Siemens R3m/V diffractometer

2θ–θ scans

Absorption correction:

empirical

*T<sub>min</sub>* = 0.43, *T<sub>max</sub>* = 0.67

4192 measured reflections

3822 independent reflections

2081 observed reflections

[*I* > 3σ(*F*)]*R<sub>int</sub>* = 0.019θ<sub>max</sub> = 22.5°*h* = 0 → 25*k* = 0 → 11*l* = -25 → 24

3 standard reflections

monitored every 97

reflections

intensity decay: 0.028%,

random

F10	0.7270 (2)	0.6249 (4)	0.6556 (2)	0.109 (2)
F11	0.6457 (2)	0.6473 (4)	0.6811 (2)	0.114 (3)
F12	0.6483 (2)	0.6459 (4)	0.5923 (2)	0.100 (2)
C1'	0.5830 (3)	0.1624 (7)	0.4401 (3)	0.062 (3)
O2'	0.5932 (2)	0.2793 (4)	0.4708 (2)	0.054 (2)
C3'	0.6297 (3)	0.3626 (6)	0.4476 (3)	0.048 (3)
O3'	0.6836 (2)	0.3713 (4)	0.4844 (2)	0.051 (2)
C4'	0.6362 (3)	0.3045 (6)	0.3910 (3)	0.067 (3)
C5'	0.6243 (3)	0.1652 (7)	0.3974 (3)	0.059 (3)
O6'	0.6752 (2)	0.1008 (4)	0.4265 (2)	0.058 (2)
C7'	0.6679 (3)	0.0405 (6)	0.4775 (3)	0.053 (3)
O7'	0.7022 (2)	0.0981 (5)	0.5247 (2)	0.053 (2)
C8'	0.6042 (3)	0.0534 (6)	0.4799 (3)	0.067 (3)
C9'	0.5168 (4)	0.1579 (11)	0.4187 (5)	0.098 (5)
C10'	0.6002 (4)	0.4896 (8)	0.4416 (4)	0.073 (4)
C11'	0.6060 (5)	0.1029 (12)	0.3388 (5)	0.110 (6)
C12'	0.6885 (4)	-0.0951 (8)	0.4748 (4)	0.080 (4)
F1'	0.4904 (2)	0.1632 (5)	0.4632 (3)	0.132 (3)
F2'	0.4966 (2)	0.2502 (6)	0.3833 (3)	0.130 (3)
F3'	0.5004 (2)	0.0486 (6)	0.3923 (3)	0.159 (3)
F4'	0.5474 (2)	0.4867 (4)	0.4078 (2)	0.114 (2)
F5'	0.5936 (2)	0.5353 (4)	0.4914 (2)	0.092 (2)
F6'	0.6309 (2)	0.5715 (4)	0.4190 (2)	0.112 (3)
F7'	0.5606 (3)	0.1583 (6)	0.3065 (2)	0.158 (3)
F8'	0.5924 (3)	-0.0181 (6)	0.3434 (2)	0.142 (3)
F9'	0.6506 (3)	0.1092 (6)	0.3129 (2)	0.142 (3)
F10'	0.7442 (2)	-0.1009 (4)	0.4722 (2)	0.091 (2)
F11'	0.6592 (2)	-0.1555 (4)	0.4286 (3)	0.128 (3)
F12'	0.6816 (2)	-0.1602 (4)	0.5192 (2)	0.127 (3)

## Refinement

Refinement on *F**R* = 0.058*wR* = 0.048*S* = 1.25

2081 reflections

482 parameters

Only coordinates of H atoms refined

*w* = 1/[σ<sup>2</sup>(*F*) + 0.00023*F*<sup>2</sup>](Δ/σ)<sub>max</sub> = 0.12Δρ<sub>max</sub> = 0.27 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.28 e Å<sup>-3</sup>

Extinction correction:

empirical, isotropic

Extinction coefficient:

χ = 0.0005 (1)

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
C1	0.5981 (3)	0.2931 (7)	0.6625 (3)	0.043 (3)
O2	0.6157 (2)	0.1845 (4)	0.6351 (2)	0.047 (2)
C3	0.6667 (3)	0.1272 (7)	0.6679 (3)	0.052 (3)
O3	0.7138 (2)	0.1457 (5)	0.6411 (2)	0.063 (2)
C4	0.6783 (3)	0.1885 (7)	0.7264 (3)	0.058 (3)
C5	0.6482 (3)	0.3151 (7)	0.7150 (3)	0.046 (3)
O6	0.6859 (2)	0.4050 (4)	0.6958 (2)	0.056 (2)
C7	0.6624 (3)	0.4513 (7)	0.6403 (3)	0.050 (3)
O7	0.6931 (2)	0.4034 (6)	0.6013 (2)	0.058 (2)
C8	0.5993 (3)	0.4099 (6)	0.6260 (3)	0.051 (3)
C9	0.5374 (3)	0.2638 (8)	0.6715 (3)	0.059 (3)
C10	0.6550 (4)	-0.0147 (8)	0.6699 (3)	0.065 (4)
C11	0.6358 (4)	0.3705 (9)	0.7699 (3)	0.070 (4)
C12	0.6708 (4)	0.5949 (8)	0.6422 (4)	0.078 (4)
F1	0.5021 (2)	0.2402 (4)	0.6206 (2)	0.076 (2)
F2	0.5143 (2)	0.3617 (4)	0.6940 (2)	0.076 (2)
F3	0.5357 (2)	0.1635 (4)	0.7044 (2)	0.075 (2)
F4	0.6087 (2)	-0.0395 (4)	0.6916 (2)	0.076 (2)
F5	0.6438 (2)	-0.0640 (4)	0.6172 (2)	0.088 (2)
F6	0.6996 (2)	-0.0747 (4)	0.7010 (2)	0.101 (2)
F7	0.6023 (2)	0.2944 (4)	0.7938 (2)	0.084 (2)
F8	0.6081 (2)	0.4810 (5)	0.7609 (2)	0.091 (2)
F9	0.6844 (2)	0.3896 (5)	0.8080 (2)	0.102 (2)

Table 2. Geometric parameters (Å, °)

C1—O2	1.427 (8)	C1'—O2'	1.435 (9)
C1—C5	1.547 (8)	C1'—C5'	1.549 (11)
C1—C8	1.519 (9)	C1'—C8'	1.513 (10)
C1—C9	1.517 (10)	C1'—C9'	1.531 (11)
O2—C3	1.424 (7)	O2'—C3'	1.422 (8)
C3—O3	1.403 (9)	C3'—O3'	1.384 (7)
C3—C4	1.513 (9)	C3'—C4'	1.524 (10)
C3—C10	1.531 (11)	C3'—C10'	1.507 (11)
C4—C5	1.515 (10)	C4'—C5'	1.516 (10)
C5—O6	1.442 (8)	C5'—O6'	1.422 (8)
C5—C11	1.520 (11)	C5'—C11'	1.528 (13)
O6—C7	1.413 (7)	O6'—C7'	1.419 (9)
C7—O7	1.391 (9)	C7'—O7'	1.384 (8)
C7—C8	1.512 (9)	C7'—C8'	1.514 (10)
C7—C12	1.534 (11)	C7'—C12'	1.522 (11)
C9—F1	1.344 (8)	C9'—F1'	1.340 (14)
C9—F2	1.335 (10)	C9'—F2'	1.315 (12)
C9—F3	1.327 (10)	C9'—F3'	1.336 (13)
C10—F4	1.325 (10)	C10'—F4'	1.330 (10)
C10—F5	1.338 (9)	C10'—F5'	1.324 (11)
C10—F6	1.313 (9)	C10'—F6'	1.315 (11)
C11—F7	1.335 (10)	C11'—F7'	1.316 (13)
C11—F8	1.334 (10)	C11'—F8'	1.331 (14)
C11—F9	1.318 (9)	C11'—F9'	1.325 (15)
C12—F10	1.329 (11)	C12'—F10'	1.323 (11)
C12—F11	1.322 (11)	C12'—F11'	1.336 (11)
C12—F12	1.315 (10)	C12'—F12'	1.302 (12)
O2—C1—C5	104.6 (5)	O2'—C1'—C5'	105.1 (6)
O2—C1—C8	110.6 (5)	O2'—C1'—C8'	109.9 (5)
C5—C1—C8	103.9 (5)	C5'—C1'—C8'	105.0 (6)
O2—C1—C9	105.7 (5)	O2'—C1'—C9'	104.3 (6)
C5—C1—C9	119.4 (6)	C5'—C1'—C9'	120.8 (7)
C8—C1—C9	112.3 (5)	C8'—C1'—C9'	111.4 (7)
C1—O2—C3	112.4 (4)	C1'—O2'—C3'	112.8 (5)
O2—C3—O3	109.9 (5)	O2'—C3'—O3'	109.8 (5)
O2—C3—C4	107.1 (5)	O2'—C3'—C4'	106.0 (5)
O3—C3—C4	110.9 (5)	O3'—C3'—C4'	110.2 (6)
O2—C3—C10	107.5 (5)	O2'—C3'—C10'	106.9 (6)
O3—C3—C10	108.3 (6)	O3'—C3'—C10'	110.0 (6)
C4—C3—C10	113.1 (6)	C4'—C3'—C10'	113.9 (6)
C3—C4—C5	103.2 (5)	C3'—C4'—C5'	104.5 (6)
C1—C5—C4	105.0 (5)	C1'—C5'—C4'	103.8 (6)
C1—C5—O6	104.7 (5)	C1'—C5'—O6'	104.2 (5)
C4—C5—O6	110.4 (5)	C4'—C5'—O6'	111.3 (6)

C1—C5—C11	120.6 (6)	C1'—C5'—C11'	120.1 (7)
C4—C5—C11	110.8 (6)	C4'—C5'—C11'	110.5 (7)
O6—C5—C11	105.2 (6)	O6'—C5'—C11'	106.7 (7)
C5—O6—C7	112.6 (4)	C5'—O6'—C7'	113.4 (5)
O6—C7—O7	110.3 (5)	O6'—C7'—O7'	110.4 (5)
O6—C7—C8	106.5 (5)	O6'—C7'—C8'	106.8 (5)
O7—C7—C8	111.2 (5)	O7'—C7'—C8'	109.8 (6)
O6—C7—C12	107.1 (5)	O6'—C7'—C12'	107.1 (7)
O7—C7—C12	107.5 (7)	O7'—C7'—C12'	108.4 (6)
C8—C7—C12	114.1 (6)	C8'—C7'—C12'	114.3 (6)
C1—C8—C7	104.0 (5)	C1'—C8'—C7'	104.1 (6)
C1—C9—F1	109.1 (6)	C1'—C9'—F1'	109.8 (8)
C1—C9—F2	111.8 (6)	C1'—C9'—F2'	113.3 (8)
F1—C9—F2	106.5 (5)	F1'—C9'—F2'	108.3 (8)
C1—C9—F3	113.1 (6)	C1'—C9'—F3'	110.9 (8)
F1—C9—F3	107.3 (7)	F1'—C9'—F3'	106.0 (8)
F2—C9—F3	108.7 (6)	F2'—C9'—F3'	108.2 (8)
C3—C10—F4	111.9 (7)	C3'—C10'—F4'	112.9 (6)
C3—C10—F5	110.9 (6)	C3'—C10'—F5'	112.1 (7)
F4—C10—F5	105.9 (6)	F4'—C10'—F5'	106.7 (7)
C3—C10—F6	111.5 (6)	C3'—C10'—F6'	110.4 (8)
F4—C10—F6	107.7 (7)	F4'—C10'—F6'	106.7 (7)
F5—C10—F6	108.7 (7)	F5'—C10'—F6'	107.7 (7)
C5—C11—F7	111.9 (7)	C5'—C11'—F7'	112.5 (9)
C5—C11—F8	112.0 (6)	C5'—C11'—F8'	111.5 (8)
F7—C11—F8	106.8 (7)	F7'—C11'—F8'	107.5 (9)
C5—C11—F9	111.2 (7)	C5'—C11'—F9'	107.9 (9)
F7—C11—F9	107.4 (6)	F7'—C11'—F9'	109.0 (9)
F8—C11—F9	107.3 (7)	F8'—C11'—F9'	108.3 (10)
C7—C12—F10	111.1 (7)	C7'—C12'—F10'	111.7 (7)
C7—C12—F11	111.3 (7)	C7'—C12'—F11'	112.0 (7)
F10—C12—F11	107.3 (6)	F10'—C12'—F11'	106.3 (8)
C7—C12—F12	110.9 (6)	C7'—C12'—F12'	111.9 (8)
F10—C12—F12	108.1 (8)	F10'—C12'—F12'	107.8 (7)
F11—C12—F12	108.1 (7)	F11'—C12'—F12'	106.9 (7)

Data were corrected for Lorentz and polarization effects. The structure solution, by direct methods, and the full-matrix least-squares refinement used programs in *SHELXTL-Plus* (Sheldrick, 1987). Hydroxyl H-atom coordinates were refined with fixed isotropic displacement parameters.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Ebrotidine

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## Abstract

The crystal structure of a new histamine H<sub>2</sub>-receptor antagonist, 4-bromo-*N*-[2-({2-[(diaminomethylene)amino]-1,3-thiazol-4-yl} methylthio)ethylaminomethylene]benzenesulfonamide, C<sub>14</sub>H<sub>17</sub>BrN<sub>6</sub>O<sub>2</sub>S<sub>3</sub>, has been determined. The main difference between the title compound and other related derivatives is in the torsion angles defining the conformation of the side chain. In the present compound the conformation of the C(thiazole)—C—S—C—C—N side chain is given by torsion angles of 74.5 (3), -76.0 (3), -91.5 (3), 72.2 (3) and -89.0 (3)°, this being the first reported derivative having this conformation. Another difference is seen in the intermolecular hydrogen-bonding scheme.

## Comment

Ebrotidine is a new histamine H<sub>2</sub>-receptor antagonist synthesized by Grupo Ferrer Research Center (Anglada, Márquez, Sacristán & Ortiz, 1988). Structurally, the cyanoguanidine moiety found in cimetidine and the 2-nitro-1,1-ethenediamine moiety found in ranitidine are here replaced by an *N*-sulfonylformamidine group. The crystal structures of several related compounds have been determined (Hadicke, Fickel & Franke, 1978; Kojic-Prodic, Ruzic-Toros, Bresciani-Pahor & Randaccio, 1980; Kojic-Prodic, Ruzic-Toros & Toso, 1982; Yanagisawa, Hirata & Ishii, 1987; Solans, Font-Altava & Cuevas-Diarte, 1988, 1995; Golic, Djinovic & Florjanic, 1989; Ishida, In, Doi, Inoue & Yanagisawa, 1989) and a structure-activity relationship study was carried out by Ishida *et al.* (1989). In order to obtain geometrical data for the title compound, (I), the present crystal structure determination was carried out.

