Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
Rajagopalan, K. V. (1991). *Advances in Enzymology and Related Areas of Molecular Biology*, Vol. 64, edited by A. Meister, pp. 215–290. New York: John Wiley.
Rowe, D. J., Garner, C. D. & Joule, J. A. (1985). *J. Chem. Soc. Perkin Trans.* 1, pp. 1907–1910.
Russell, J. R., Garner, C. D. & Joule, J. A. (1992*a*). *J. Chem. Soc. Perkin Trans.* 1, pp. 1245–1249.
Russell, J. R., Garner, C. D. & Joule, J. A. (1992*b*). *Tetrahedron Lett.* **33**, 3371–3374.
Russell, J. R., Garner, C. D. & Joule, J. A. (1992*c*). *Synlett.* pp. 711–712.
Sheldrick, G. M. (1985). *SHELXS86*. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1993). C**49**, 1652–1654

Tetra(*n*-butyl)ammonium Trifluoromethanesulfonate

ALEXANDER J. BLAKE,* CHRISTIAN RADEK AND MARTIN SCHRÖDER

Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

(*Received* 21 *October* 1992; *accepted* 10 *February* 1993)

Abstract

The title structure comprises two ion pairs in each asymmetric unit. There are no close contacts between any of the ions. Although the structure determination was carried out at reduced temperature there are large vibration amplitudes for some F atoms and disorder in one of the *n*-butyl chains.

Comment

Trifluoromethanesulfonic acid has been reported in its anhydrous form (Bartmann & Mootz, 1990) and with increasing degrees of hydration (Lundgren & Spencer, 1973; Delaplane, Lundgren & Olovsson, 1975*a*,*b*; Lundgren, 1978*a*,*b*). One series of salts with nonaaqualanthanide(III) cations has been reported (Harrowfield, Kepert, Patrick & White, 1983; Castellano, Machado, Santos & Vicentini, 1985; Chatterjee, Maslen & Watson, 1988). Salts with the cations $[V(H_2O)_6]^{2+}$ (Holt, Larkworthy, Leigh, Povey & Smith, 1989) and $[V(H_2O)_6]^{3+}$ (Cotton, Fair, Lewis, Mott, Ross, Schultz & Williams, 1984) are also known. Although $CF_3SO_3^-$ is a common counteranion for complex metal ions, there are relatively few examples with simple inorganic cations such as oxonium (Lundgren, Olovsson & Tellgren, 1978), ammonium (Brauer & Ganswein, 1975) and seleninyl (Kapoor, Kapoor, Sawyer & Wadhawan, 1988). Salts containing simple alkylammonium cations are even rarer (*e.g.* $CH_3NH_3^+$; Alberts, Noltes, Roelofsen & Spek, 1982).

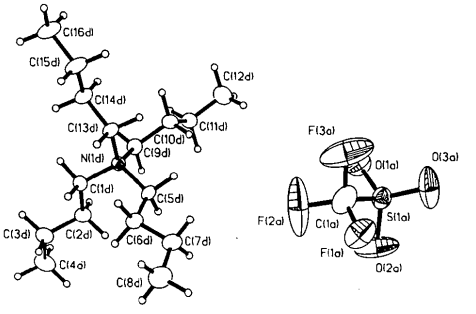


Fig. 1. View of one ion pair showing the labelling of the non-H atoms. Thermal ellipsoids are drawn at the 50% probability level except for H atoms which have artificial radii of 0.10 Å^2 for clarity.

Selected geometric parameters are shown in Table 1. C—C bond lengths are normal; bond angles are in the range 108–112° except for those listed; torsion angles are all in the range 60 ± 10° or 180 ± 10° except those listed.

Experimental

Crystal data

$C_{16}H_{36}N^+.CF_3SO_3^-$
M_r = 391.5
Monoclinic
$P2_1/a$
a = 15.531 (4) Å
b = 18.136 (6) Å
c = 16.895 (4) Å
β = 114.674 (20)°
V = 4324 Å^3
Z = 8
D_x = 1.202 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 41 reflections
θ = 15–16°
μ = 0.181 mm^{-1}
T = 150.0 K
Block
1.04 × 0.70 × 0.70 mm
Colourless
Crystal source: recrystallization from MeOH/H_2O

Data collection

Stoe Stadi-4 diffractometer
ω–2θ scans
Absorption correction: none
5901 measured reflections
5901 independent reflections
3823 observed reflections [$F>4\sigma(F)$]
θ_{max} = 22.5°
$h = -16 \rightarrow 15$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: 2.5% (isotropic decay)

Refinement

Refinement on F	$w = [\sigma^2(F) + 0.000228F^2]^{-1}$
Final R = 0.0557	$(\Delta/\sigma)_{max}$ = 0.001
wR = 0.0661	$\Delta\rho_{max}$ = 0.72 e Å^{-3}
S = 1.342	$\Delta\rho_{min}$ = −0.64 e Å^{-3}
3823 reflections	Extinction correction: none
450 parameters	Atomic scattering factors from *SHELX*76
Only H-atom U's refined	

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

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S(1*A*)	0.06121 (9)	0.16678 (7)	0.87909 (8)	0.0366 (8)
O(1*A*)	0.02705 (23)	0.20308 (19)	0.79711 (20)	0.0460 (24)
O(2*A*)	0.15412 (25)	0.1345 (3)	0.9036 (3)	0.087 (4)
O(3*A*)	0.0511 (4)	0.20292 (22)	0.94757 (25)	0.086 (4)
C(1*A*)	−0.0101 (4)	0.0853 (3)	0.8614 (3)	0.051 (4)
F(1*A*)	0.01310 (21)	0.04280 (17)	0.93037 (19)	0.0592 (23)
F(2*A*)	−0.0150 (4)	0.04515 (23)	0.7973 (3)	0.141 (5)
F(3*A*)	−0.10146 (25)	0.1062 (3)	0.8413 (3)	0.113 (4)
S(1*B*)	−0.48274 (8)	0.76989 (7)	0.61336 (7)	0.0336 (8)
O(1*B*)	−0.56833 (22)	0.73996 (20)	0.61354 (23)	0.053 (3)
O(2*B*)	−0.47703 (25)	0.76501 (21)	0.53118 (21)	0.056 (3)
O(3*B*)	−0.39776 (22)	0.75239 (20)	0.68844 (21)	0.0476 (24)
C(1*B*)	−0.4953 (3)	0.8682 (3)	0.6249 (3)	0.045 (4)
F(1*B*)	−0.57159 (21)	0.89545 (18)	0.55963 (23)	0.074 (3)
F(2*B*)	−0.42171 (19)	0.90605 (16)	0.62569 (20)	0.0573 (23)
F(3*B*)	−0.5008 (3)	0.88472 (21)	0.69887 (24)	0.092 (3)
N(1*C*)	−0.27582 (23)	0.26923 (19)	0.69270 (21)	0.0260 (23)
C(1*C*)	−0.2027 (3)	0.23471 (25)	0.6661 (3)	0.030 (3)
C(2*C*)	−0.2398 (3)	0.2085 (3)	0.5722 (3)	0.034 (3)
C(3*C*)	−0.1578 (3)	0.1762 (3)	0.5552 (3)	0.040 (3)
C(4*C*)	−0.1909 (4)	0.1521 (3)	0.4605 (3)	0.047 (4)
C(5*C*)	−0.2279 (3)	0.28412 (25)	0.79076 (25)	0.026 (3)
C(6*C*)	−0.1424 (3)	0.3346 (3)	0.8211 (3)	0.029 (3)
C(7*C*)	−0.1112 (3)	0.3519 (3)	0.9167 (3)	0.042 (4)
C(8*C*)	−0.0190 (3)	0.3952 (3)	0.9552 (3)	0.046 (4)
C(9*C*)	−0.3115 (3)	0.34009 (25)	0.6410 (3)	0.030 (3)
C(10*C*)	−0.3692 (3)	0.39034 (25)	0.6724 (3)	0.033 (3)
C(11*C*)	−0.4007 (4)	0.4582 (3)	0.6133 (3)	0.045 (4)
C(12*C*)	−0.4527 (4)	0.5137 (3)	0.6449 (3)	0.055 (4)
C(13*C*)	−0.3605 (3)	0.21913 (25)	0.6737 (3)	0.029 (3)
C(14*C*)	−0.3361 (3)	0.1415 (3)	0.7114 (3)	0.042 (4)
C(15*C*)	−0.4164 (4)	0.0898 (4)	0.6779 (5)	0.090 (6)
C(16)	−0.4933 (6)	0.1133 (5)	0.7088 (6)	0.0500
C(16′)	−0.5232 (5)	0.1085 (8)	0.6440 (9)	0.0500
N(1*D*)	0.12889 (23)	0.17088 (20)	0.19672 (21)	0.0247 (23)
C(1*D*)	0.1449 (3)	0.10861 (25)	0.1442 (3)	0.029 (3)
C(2*D*)	0.2447 (3)	0.1043 (3)	0.1470 (3)	0.035 (3)
C(3*D*)	0.2542 (3)	0.0330 (3)	0.1042 (3)	0.044 (4)
C(4*D*)	0.3481 (4)	0.0287 (3)	0.0968 (4)	0.058 (4)
C(5*D*)	0.1933 (3)	0.1640 (3)	0.29391 (25)	0.027 (3)
C(6*D*)	0.1967 (3)	0.0886 (3)	0.3334 (3)	0.034 (3)
C(7*D*)	0.2440 (4)	0.0962 (3)	0.4331 (3)	0.041 (4)
C(8*D*)	0.2622 (4)	0.0221 (3)	0.4771 (3)	0.061 (4)
C(9*D*)	0.1504 (3)	0.24393 (24)	0.1648 (3)	0.030 (3)
C(10*D*)	0.1266 (3)	0.31243 (24)	0.2034 (3)	0.033 (3)
C(11*D*)	0.1825 (3)	0.3780 (3)	0.1937 (3)	0.038 (3)
C(12*D*)	0.1581 (4)	0.4494 (3)	0.2260 (4)	0.052 (4)
C(13*D*)	0.0263 (3)	0.1670 (3)	0.1857 (3)	0.028 (3)
C(14*D*)	−0.0491 (3)	0.1793 (3)	0.0939 (3)	0.034 (3)
C(15*D*)	−0.1460 (3)	0.1693 (3)	0.0930 (3)	0.045 (4)
C(16*D*)	−0.2267 (3)	0.1808 (3)	0.0036 (3)	0.050 (4)

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

S(1*A*)—O(1*A*)	1.421 (4)	C(1*B*)—F(1*B*)	1.332 (7)
S(1*A*)—O(2*A*)	1.448 (5)	C(1*B*)—F(2*B*)	1.328 (6)
S(1*A*)—O(3*A*)	1.394 (5)	C(1*B*)—F(3*B*)	1.322 (7)
S(1*A*)—C(1*A*)	1.794 (6)	N(1*C*)—C(1*C*)	1.520 (6)
C(1*A*)—F(1*A*)	1.317 (7)	N(1*C*)—C(5*C*)	1.530 (6)
C(1*A*)—F(2*A*)	1.281 (8)	N(1*C*)—C(9*C*)	1.522 (6)
C(1*A*)—F(3*A*)	1.368 (7)	N(1*C*)—C(13*C*)	1.519 (6)
S(1*B*)—O(1*B*)	1.437 (4)	N(1*D*)—C(1*D*)	1.519 (6)
S(1*B*)—O(2*B*)	1.431 (4)	N(1*D*)—C(5*D*)	1.530 (6)
S(1*B*)—O(3*B*)	1.433 (4)	N(1*D*)—C(9*D*)	1.520 (6)
S(1*B*)—C(1*B*)	1.812 (6)	N(1*D*)—C(13*D*)	1.527 (6)
O(1*A*)—S(1*A*)—O(2*A*)	112.84 (25)	F(1*B*)—C(1*B*)—F(2*B*)	106.3 (4)
O(1*A*)—S(1*A*)—O(3*A*)	117.7 (3)	F(2*B*)—C(1*B*)—F(3*B*)	105.5 (4)
O(1*A*)—S(1*A*)—C(1*A*)	105.38 (25)	N(1*C*)—C(5*C*)—C(6*C*)	115.7 (4)
O(2*A*)—S(1*A*)—O(3*A*)	113.4 (3)	C(6*C*)—C(7*C*)—C(8*C*)	112.8 (4)
O(2*A*)—S(1*A*)—C(1*A*)	100.7 (3)	N(1*C*)—C(9*C*)—C(10*C*)	115.6 (4)
O(3*A*)—S(1*A*)—C(1*A*)	104.5 (3)	N(1*C*)—C(13*C*)—C(14*C*)	114.9 (4)
S(1*A*)—C(1*A*)—F(1*A*)	114.4 (4)	C(13*C*)—C(14*C*)—C(15*C*)	113.9 (5)
S(1*A*)—C(1*A*)—F(2*A*)	115.1 (5)	C(14*C*)—C(15*C*)—C(16′)	127.4 (7)
F(1*A*)—C(1*A*)—F(3*A*)	104.7 (5)	N(1*D*)—C(1*D*)—C(2*D*)	115.5 (4)
F(2*A*)—C(1*A*)—F(3*A*)	105.0 (5)	N(1*D*)—C(5*D*)—C(6*D*)	115.7 (4)
O(1*B*)—S(1*B*)—O(2*B*)	114.26 (22)	C(2*B*)—C(1*B*)—F(3*B*)	105.5 (4)
O(1*B*)—S(1*B*)—O(3*B*)	115.18 (22)	C(1*C*)—N(1*C*)—C(5*C*)	107.9 (3)
O(1*B*)—S(1*B*)—C(1*B*)	103.32 (24)	C(1*C*)—N(1*C*)—C(13*C*)	112.3 (3)
O(2*B*)—S(1*B*)—O(3*B*)	115.98 (22)	C(9*C*)—N(1*C*)—C(13*C*)	107.9 (3)
O(2*B*)—S(1*B*)—C(1*B*)	102.60 (24)	N(1*C*)—C(1*C*)—C(2*C*)	115.7 (4)
O(3*B*)—S(1*B*)—C(1*B*)	102.89 (23)	N(1*D*)—C(9*D*)—C(10*D*)	115.5 (4)
S(1*B*)—C(1*B*)—F(2*B*)	112.3 (4)	C(10*D*)—C(11*D*)—C(12*D*)	112.9 (4)
S(1*B*)—C(1*B*)—F(3*B*)	112.2 (4)	N(1*D*)—C(13*D*)—C(14*D*)	116.0 (4)
		C(14*D*)—C(15*D*)—C(16*D*)	113.5 (4)

C(1*C*)—N(1*C*)—C(9*C*)—C(10*C*)	−167.6 (4)
C(5*C*)—N(1*C*)—C(9*C*)—C(10*C*)	−48.8 (5)
C(13*C*)—N(1*C*)—C(9*C*)—C(10*C*)	70.6 (5)
N(1*C*)—C(13*C*)—C(14*C*)—C(15*C*)	−168.3 (4)
C(13*C*)—C(14*C*)—C(15*C*)—C(16′)	−27.8 (10)
C(1*D*)—N(1*D*)—C(5*D*)—C(6*D*)	−47.9 (5)
C(9*D*)—N(1*D*)—C(5*D*)—C(6*D*)	−168.0 (4)
C(13*D*)—N(1*D*)—C(5*D*)—C(6*D*)	71.0 (5)
N(1*D*)—C(5*D*)—C(6*D*)—C(7*D*)	−166.5 (4)
N(1*D*)—C(9*D*)—C10*D*)—C(11*D*)	159.3 (4)

The crystal was cooled in the nitrogen gas stream of an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986). The structure was solved by automatic direct methods (*SHELXS*86; Sheldrick, 1986) and refined using *SHELX*76 (Sheldrick, 1976). The illustration was prepared using *SHELXTL-PC* (Sheldrick, 1990) and molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Anisotropic thermal motion was allowed for all non-H atoms except C(16) and C(16′) which constitute a 0.61:0.39 disorder pair. The disorder was modelled by constraining the C(15*C*)—C(16) and C(15*C*)—C(16′) bonds to be 1.55 Å. A common isotropic thermal parameter for H atoms in calculated positions refined to 0.056 (2) Å^2.

We thank the SERC for support.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71091 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1038]

References

Alberts, A. H., Noltes, J. G., Roelofsen, G. & Spek, A. L. (1982). *Cryst. Struct. Commun.* **11**, 1863–1868.

Bartmann, K. & Mootz, D. (1990). *Acta Cryst.* C**46**, 319–320.

Brauer, G. & Ganswein, B. (1975). *Z. Anorg. Allg. Chem.* **415**, 125–132.

Castellano, E. E., Machado, L. C., Santos, C. O. P. & Vicentini, G. (1985). *Inorg. Chim. Acta*, **110**, 83–86.

Chatterjee, A., Maslen, E. N. & Watson, K. J. (1988). *Acta Cryst.* B**44**, 381–386.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Cotton, F. A., Fair, C. K., Lewis, G. E., Mott, G. N., Ross, F. K., Schultz, A. J. & Williams, J.M. (1984). *J. Am. Chem. Soc.* **106**, 5319–5323.
Delaplane, R. G., Lundgren, J. O. & Olovsson, I. (1975*a*). *Acta Cryst.* B**31**, 2202–2207.
Delaplane, R. G., Lundgren, J. O. & Olovsson, I. (1975*b*). *Acta Cryst.* B**31**, 2208–2213.
Gould, R. O. & Taylor, P. (1985). *CALC*. Program for crystallographic calculations. Univ. of Edinburgh, Scotland.
Harrowfield, J. McB., Kepert, D. L., Patrick, J. M. & White, A. H. (1983). *Aust. J. Chem.* **36**, 483–492.
Holt, D. G. L., Larkworthy, L. L., Leigh, G. J., Povey, D. C. & Smith, G. W. (1989). *Polyhedron*, **8**, 1823–1824.
Kapoor, P., Kapoor, R., Sawyer, J. F. & Wadhawan, P. (1988). *Can. J. Chem.* **66**, 2367–2374.
Lundgren, J. O. (1978*a*). *Acta Cryst.* B**34**, 2428–2431.
Lundgren, J. O. (1978*b*). *Acta Cryst.* B**34**, 2432–2435.
Lundgren, J. O., Olovsson, I. & Tellgren, R. (1978). *Acta Cryst.* B**34**, 2945–2947.
Lundgren, J. O. & Spencer, J. B. (1973). *Acta Cryst.* B**29**, 1923–1928.
Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1990). *SHELXTL-PC*. Version 4.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1993). C**49**, 1654–1655

Structure of (±)-1β-*tert*-Butoxy-3aα,4β,5,6,7,7a-hexahydro-7aβ-methyl-5-oxo-4α-indancarboxylic Acid Methyl Ester at 153 K

EHMKE POHL, REGINE HERBST-IRMER AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie, Universität Göttingen, Tammannstrasse 4, 3400 Göttingen, Germany

SIEGLINDE VAN HOMMELEN AND ULRICH GROTH

Institut für Organische Chemie, Universität Göttingen, Tammannstrasse 2, 3400 Göttingen, Germany

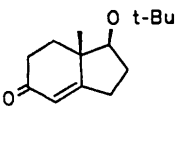
(*Received* 6 *October* 1992; *accepted* 15 *February* 1993)

Abstract

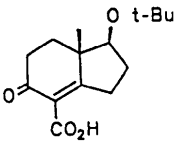
The X-ray structure of (±)-1β-*tert*-butoxy-3aα,4β,5,6,-7,7a-hexahydro-7aβ-methyl-5-oxo-4α-indancarboxylic acid methyl ester is reported, in which the six-membered ring adopts a pseudo chair conformation and the five-membered ring an envelope conformation.

Comment

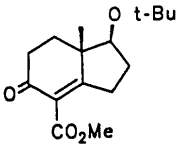
The title compound was prepared in a three-step sequence from tetrahydroindanone (I) and promises to be a suitable CD building block for the construction of 7α-substituted steroids. Direct carboxylation of (I) with magnesium methyl carbonate in dimethylformamide afforded the unsaturated β-keto acid (II) (Micheli *et al.*, 1975). Subsequent esterification with methanol in the presence of dicyclohexylcarbodiimide and dimethylaminopyridine in methylenedichloride yielded the β-keto ester (III) (Neises & Steglich, 1978). High stereospecificity was observed in the hydrogenation of (III) with $Pd/BaSO_4$ and H_2 in methanol which gave only the *trans* product (IV). The



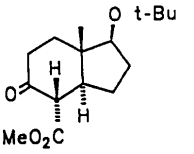
(I)



(II)



(III)



(IV)

structure determination was undertaken to investigate the stereospecificity of this step. Colourless crystals were obtained by slow evaporation from a mixture of diethyl ether-pentane at room temperature. The six-membered ring adopts a pseudo chair conformation and the five-membered ring an envelope conformation. The bond distances are comparable with the corresponding distances in other hexahydroindan derivatives (Schomer, Sheldrick & Wagner, 1978; D'Angelo *et al.*, 1983; Caine *et al.*, 1987).

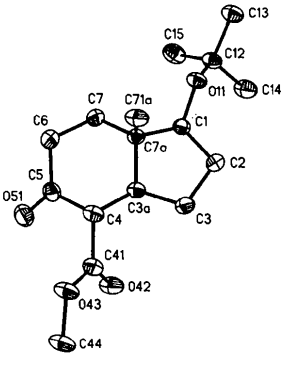


Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.