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Triethylammonium Isopropyl [(Aminosulfonyl)(difluoro)methyl]phosphonate

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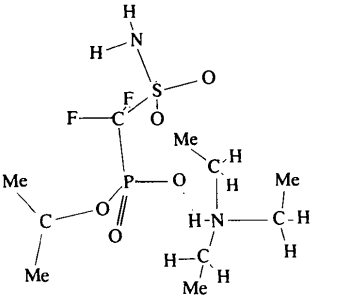
Abstract

The title salt, $[(C_2H_5)_3NH]^+.C_4H_9F_2NO_5PS^-$, was the unexpected product in an attempted synthesis of $[(CH_3)_2CHO]_2P(O)CF_2SO_2NH_2$ from $[(CH_3)_2CHO]_2P(O)CF_2Br$ and hydroxylamine-*O*-sulfonic acid. Both the S and P atoms are surrounded tetrahedrally, with the O—S—O and O—P—O bond angles of 120.7 (2) and 121.4 (1)°, respectively, showing the greatest distortion from true tetrahedral geometry. The remaining bond distances and angles have typical values, with the bond distances to C(1), the fluorinated C atom, being slightly longer.

Comment

Single crystals of the title compound, (I), were obtained while attempting the reaction of $[(CH_3)_2CHO]_2P(O)CF_2Br$ and H_2NOSO_3H to prepare $[(CH_3)_2CHO]_2P(O)CF_2SO_2NH_2$. The spectroscopic data for the product are: ^{19}F NMR −111.0 p.p.m. (*d*, $J_{P,F}$ = 78 Hz); ^{31}P NMR −4.1 p.p.m. (*d*); ^1H NMR 1.44 (*d*, $J_{H,H}$ = 6.1 Hz), 4.9 (septet), 7.4 p.p.m. (*s*) (exchangeable); ^{13}C NMR 121.1 (*td*, J_{PC} = 178, J_{CF} = 302 Hz), 75.4 (*d*, J_{POC} = 6.42 Hz), 26.0 (*d*, J_{POCC} = 3.63 Hz), 48.5 (*s*), 10.7 p.p.m. (*s*) [Et_3NH^+].



(I)

The bond distances and angles in the title salt are close to expected values for this type of molecule (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The S and P atoms are surrounded by four groups in slightly distorted tetrahedra. The O—S—O, O—P—O, and P—C(1)—S bond angles are 120.7 (2), 121.4 (1) and 117.3 (2)°, respectively, and show the largest distortions from tetrahedral geometry. The C(1)—P [1.865 (3) Å] and C(1)—S [1.832 (3) Å] bond distances are slightly longer than normal, as are the C—F distances [C(1)—F(1), C(1)—F(2) 1.370 (4) Å]. The shortened C—C distances in the cation are a likely consequence of the large thermal motion.

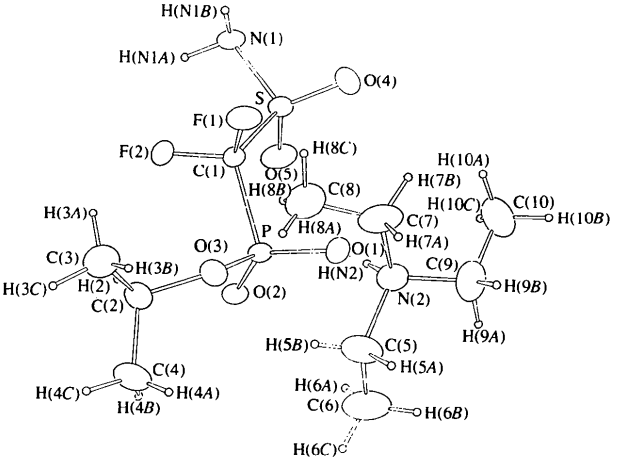


Fig. 1. A view of the hydrogen-bonded anion–cation pair in the title compound. Displacement ellipsoids are at the 25% probability level.

Experimental

Crystal data

$[(C_2H_5)_3NH]^+.C_4H_9F_2NO_5PS^-$ — Mo $K\alpha$ radiation, λ = 0.71073 Å

M_r = 354.356
Orthorhombic
$P2_12_12_1$
a = 7.479 (2) Å
b = 12.221 (5) Å
c = 19.306 (6) Å
V = 1764.5 (1.0) Å^3
Z = 4
D_x = 1.33 Mg m^{-3}

Cell parameters from 16 reflections
θ = 20–25°
μ = 0.311 mm^{-1}
T = 295 K
Needle
0.57 × 0.26 × 0.18 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
Profile data from θ–2θ scans
Absorption correction: none
13 199 measured reflections
1810 independent reflections
1510 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.024

θ_{max} = 25°
$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$
3 standard reflections monitored every 200 reflections
frequency: 60 min
intensity decay: <4.6%

Refinement

Refinement on F
R = 0.034
wR = 0.056
S = 1.17
1510 reflections
191 parameters
$w = 1/[\sigma^2(F) + (0.04F)^2]$
$(\Delta/\sigma)_{max}$ = 0.22

$\Delta\rho_{max}$ = 0.2 e Å^{-3}
$\Delta\rho_{min}$ = −0.2 e Å^{-3}
Extinction correction: Zachariasen (1963)
Extinction coefficient: 2.8×10^{-7}
Atomic scattering factors from *MolEN* (Fair, 1990)

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

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S	0.4434 (1)	0.20935 (7)	0.39365 (5)	0.0389 (3)
P	0.0815 (1)	0.07652 (7)	0.39647 (4)	0.0319 (1)
F(1)	0.4150 (3)	−0.0016 (2)	0.4048 (1)	0.0546 (6)
F(2)	0.3296 (3)	0.0919 (2)	0.4948 (1)	0.0456 (5)
O(1)	0.0867 (3)	0.0643 (2)	0.3202 (1)	0.0452 (6)
O(2)	−0.0204 (3)	0.1665 (2)	0.4299 (1)	0.0384 (6)
O(3)	0.0316 (4)	−0.0394 (2)	0.4281 (1)	0.0450 (6)
O(4)	0.5057 (4)	0.1837 (3)	0.3262 (1)	0.0637 (8)
O(5)	0.3208 (4)	0.2977 (2)	0.4045 (2)	0.0581 (8)
N(1)	0.6060 (4)	0.2255 (3)	0.4436 (2)	0.0458 (8)
N(2)	0.4797 (5)	0.1206 (3)	0.7456 (2)	0.0557 (9)
C(1)	0.3197 (4)	0.0892 (3)	0.4240 (2)	0.0322 (8)
C(2)	−0.0240 (6)	−0.0533 (3)	0.5005 (2)	0.0518 (10)
C(3)	0.0605 (9)	−0.1570 (5)	0.5253 (3)	0.086 (1)
C(4)	−0.2228 (7)	−0.0577 (5)	0.5047 (3)	0.081 (1)
C(5)	0.6568 (7)	0.1653 (5)	0.7694 (3)	0.092 (1)
C(6)	0.7940 (8)	0.0814 (6)	0.7733 (4)	0.123 (3)
C(7)	0.3348 (7)	0.2079 (5)	0.7486 (4)	0.092 (1)
C(8)	0.2908 (8)	0.2439 (5)	0.8182 (4)	0.098 (3)
C(9)	0.489 (1)	0.0762 (6)	0.6746 (3)	0.096 (1)
C(10)	0.331 (1)	0.0077 (5)	0.6561 (3)	0.104 (3)

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

S—O(4)	1.419 (3)	C(2)—O(3)	1.469 (5)
S—O(5)	1.431 (3)	C(2)—C(3)	1.494 (7)
S—N(1)	1.565 (3)	C(2)—C(4)	1.490 (7)
S—C(1)	1.832 (3)	N(2)—C(5)	1.505 (6)
P—O(1)	1.480 (2)	N(2)—C(7)	1.522 (7)
P—O(2)	1.485 (2)	N(2)—C(9)	1.476 (6)
P—O(3)	1.587 (2)	C(5)—C(6)	1.452 (9)
P—C(1)	1.865 (3)	C(7)—C(8)	1.451 (9)
C(1)—F(1)	1.370 (4)	C(9)—C(10)	1.489 (10)
C(1)—F(2)	1.370 (4)	N(2)···O(1^i)	2.725 (4)
O(4)—S—O(5)	120.7 (2)	S—C(1)—P	117.3 (2)
O(4)—S—N(1)	109.8 (2)	S—C(1)—F(1)	107.4 (2)
O(4)—S—C(1)	106.4 (2)	S—C(1)—F(2)	105.8 (2)
O(5)—S—N(1)	108.2 (2)	P—C(1)—F(1)	110.6 (2)
O(5)—S—C(1)	103.5 (2)	P—C(1)—F(2)	109.7 (2)
N(1)—S—C(1)	107.3 (2)	F(1)—C(1)—F(2)	105.2 (3)
O(1)—P—O(2)	121.4 (1)	C(3)—C(2)—C(4)	111.9 (4)
O(1)—P—O(3)	107.4 (1)	C(3)—C(2)—O(3)	106.4 (4)
O(1)—P—C(1)	105.4 (1)	C(4)—C(2)—O(3)	109.7 (4)
O(2)—P—O(3)	111.9 (1)	N(2)—C(5)—C(6)	112.4 (6)
O(2)—P—C(1)	107.8 (1)	N(2)—C(7)—C(8)	114.2 (5)
O(3)—P—C(1)	100.9 (1)	N(2)—C(9)—C(10)	113.2 (5)
P—O(3)—C(2)	122.4 (2)	N(2)—H(N2)···O(1^i)	165
O(4)—S—C(1)—P	−83.6 (2)	O(2)—P—C(1)—S	−66.7 (2)
O(4)—S—C(1)—F(1)	41.7 (2)	O(2)—P—C(1)—F(1)	169.6 (2)
O(4)—S—C(1)—F(2)	153.7 (2)	O(2)—P—C(1)—F(2)	54.0 (2)
O(5)—S—C(1)—P	44.6 (2)	O(3)—P—C(1)—S	175.9 (2)
O(5)—S—C(1)—F(1)	169.9 (2)	O(3)—P—C(1)—F(1)	52.2 (2)
O(5)—S—C(1)—F(2)	−78.1 (2)	O(3)—P—C(1)—F(2)	−63.4 (2)
N(1)—S—C(1)—P	159.0 (2)	P—O(3)—C(2)—C(3)	−141.0 (3)
N(1)—S—C(1)—F(1)	−75.8 (2)	P—O(3)—C(2)—C(4)	97.7 (4)
N(1)—S—C(1)—F(2)	36.2 (2)	C(7)—N(2)—C(5)—C(6)	174.7 (5)
O(1)—P—O(3)—C(2)	−166.3 (3)	C(9)—N(2)—C(5)—C(6)	−63.0 (6)
O(2)—P—O(3)—C(2)	−30.8 (3)	C(5)—N(2)—C(7)—C(8)	−66.4 (6)
C(1)—P—O(3)—C(2)	83.6 (3)	C(9)—N(2)—C(7)—C(8)	169.5 (5)
O(1)—P—C(1)—S	64.3 (2)	C(5)—N(2)—C(9)—C(10)	165.8 (5)
O(1)—P—C(1)—F(1)	−59.4 (2)	C(7)—N(2)—C(9)—C(10)	−70.6 (6)
O(1)—P—C(1)—F(2)	−175.0 (2)		

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

The scan range was $(0.6 + 0.35\tan\theta)°$ with a scan speed of 1.0–4.0° min^{-1}; the background was measured as 25% of the range, below and above, and counting time as reflections/background = 2/1.

The structure was determined by direct methods. All non-H atoms were refined with anisotropic displacement parameters. All H atoms were located from electron-density difference maps, but were placed at 0.95 Å from C atoms and given isotropic displacement parameters equivalent to those of their parent atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *MolEN*. Software used to prepare material for publication: *MolEN*.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1095). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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