# Crystal structure of (3*S*,1′*S*)-2,2-dimethyl-3-[1,2-cyclohexylidenedioxyethyl]-tetrahydro-1,2-oxazolium tetrafluoroborate, $(C_{13}H_{24}NO_3)[BF_4]$

W. Frey, M. Henneböhle and V. Jäger*

Institut für Organische Chemie, Universität Stuttgart, Pfaffenwaldring 55, 70569 Stuttgart, Germany

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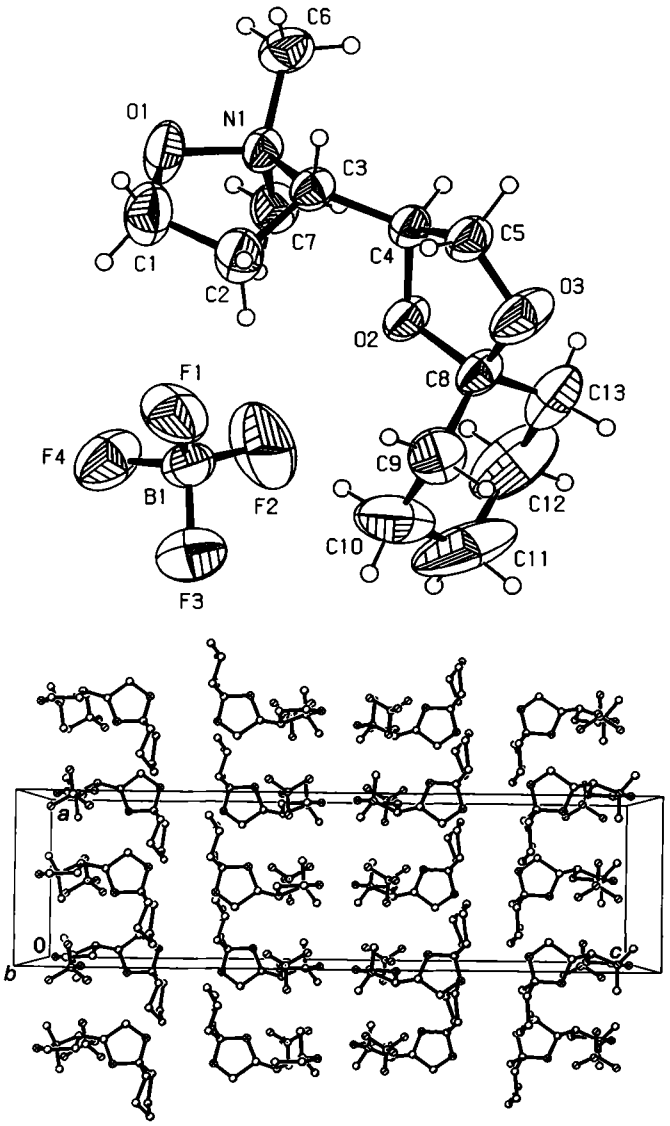


**Abstract**

$C_{13}H_{24}BF_4NO_3$, orthorhombic, $C222_1$ (no. 20), $a = 9.320(2)$ Å, $b = 10.6761(8)$ Å, $c = 33.786(2)$ Å, $V = 3361.6$ Å$^3$, $Z = 8$, $R_{gt}(F) = 0.064$, $wR_{ref}(F^2) = 0.185$, $T = 293$ K.

**Source of material**

The title compound has been obtained by 1,3-dipolar cycloaddition of ethylene and the nitrile oxide obtained *in situ* from hydroximoyl chloride, derived from 2,3-*O*-cyclohexylidene-D-glyceraldehyde [1-3]. This was followed by *N*-methylation of the intermediate isoxazoline with trimethyloxonium tetrafluoroborate and reduction on treatment with sodium borohydride in ethanol [2]. Further methylation of the corresponding major isoxazolidine isomer (separated by MPLC, ratio of diastereoisomers = 84:16) with trimethyloxonium tetrafluoroborate [3-5] and recrystallization from ethanol afforded the isoxazolidinium salt in the form of colorless crystals (m.p. 443-444 K, $\alpha_D^{20} = -29.4°$, $c = 0.81$, $CH_2Cl_2$).

**Discussion**

The isoxazolidine ring system shows an *envelope* conformation (figure, top). Three atoms (C10,C11,C12) of the cyclohexylidene moiety exhibit very large displacement parameters. A resolution of eventual disorder via split positions was not performed, the distances of the C–C bonds deviate less than 5 % from normal values. The fluorine atoms of the tetrafluoroborate anion have also large displacement parameters, which indicates considerable rotation of the anion.

The packing diagram reveals a bilayer-type stacking of the molecules with alternating polarity along the *c* axis (figure, bottom). The non-polar bilayer is built up by a face-to-face orientation of the cyclohexylidene moieties; the polar bilayer is built by the hetero atoms of the isoxazolidines and the tetrafluoroborate anions.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colorless lath, size 0.10 × 0.25 × 1.2 mm |
| Wavelength: | Cu $K_\alpha$ radiation (1.54178 Å) |
| $\mu$: | 10.24 cm$^{-1}$ |
| Diffractometer, scan mode: | Siemens P4, $\omega$ |
| $2\theta_{max}$: | 109.94° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 1613, 1501 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1174 |
| $N(param)_{refined}$: | 199 |
| Programs: | SHELXS-97 [6], SHELXL-97 [7] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1A) | 8*c* | 0.0618 | 0.5178 | 0.4513 | 0.146 |
| H(1B) | 8*c* | −0.1065 | 0.5198 | 0.4543 | 0.146 |
| H(2A) | 8*c* | 0.0584 | 0.4363 | 0.3923 | 0.120 |
| H(2B) | 8*c* | −0.1006 | 0.4873 | 0.3909 | 0.120 |
| H(3) | 8*c* | −0.1956 | 0.3044 | 0.4095 | 0.082 |
| H(4) | 8*c* | −0.0728 | 0.1290 | 0.3796 | 0.083 |
| H(5A) | 8*c* | −0.2387 | 0.1880 | 0.3358 | 0.102 |
| H(5B) | 8*c* | −0.1904 | 0.3296 | 0.3366 | 0.102 |

* Correspondence author (e-mail: jager.ioc@po.uni-stuttgart.de)

**Table 2.** Continued.

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(6A) | 8c | –0.2233 | 0.2026 | 0.4674 | 0.139 |
| H(6B) | 8c | –0.0951 | 0.1492 | 0.4923 | 0.139 |
| H(6C) | 8c | –0.1353 | 0.0880 | 0.4516 | 0.139 |
| H(7A) | 8c | 0.1786 | 0.2594 | 0.4292 | 0.126 |
| H(7B) | 8c | 0.1156 | 0.1234 | 0.4278 | 0.126 |
| H(7C) | 8c | 0.1547 | 0.1845 | 0.4685 | 0.126 |
| H(9A) | 8c | 0.0829 | 0.4215 | 0.3099 | 0.135 |
| H(9B) | 8c | 0.1214 | 0.3536 | 0.2702 | 0.135 |

**Table 2.** Continued.

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(10A) | 8c | 0.3019 | 0.3713 | 0.3357 | 0.206 |
| H(10B) | 8c | 0.3223 | 0.4437 | 0.2957 | 0.206 |
| H(11A) | 8c | 0.4682 | 0.2682 | 0.2992 | 0.278 |
| H(11B) | 8c | 0.3641 | 0.2628 | 0.2627 | 0.278 |
| H(12A) | 8c | 0.3568 | 0.0628 | 0.2933 | 0.230 |
| H(12B) | 8c | 0.3242 | 0.1229 | 0.3348 | 0.230 |
| H(13A) | 8c | 0.1327 | 0.1176 | 0.2698 | 0.157 |
| H(13B) | 8c | 0.1093 | 0.0479 | 0.3102 | 0.157 |

**Table 3.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | x | y | z | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| F(1) | 8c | 0.3766(5) | 0.3963(5) | 0.4213(2) | 0.080(3) | 0.142(4) | 0.220(6) | –0.006(3) | 0.018(3) | –0.028(4) |
| F(2) | 8c | 0.516(1) | 0.2274(4) | 0.4090(2) | 0.288(9) | 0.068(2) | 0.266(7) | –0.005(5) | –0.029(8) | –0.011(3) |
| F(3) | 8c | 0.5787(6) | 0.4134(5) | 0.3900(2) | 0.135(4) | 0.143(4) | 0.128(3) | 0.007(3) | 0.025(3) | 0.032(3) |
| F(4) | 8c | 0.5860(5) | 0.3683(6) | 0.4524(1) | 0.113(3) | 0.222(6) | 0.092(3) | 0.017(4) | –0.004(2) | –0.004(3) |
| B(1) | 8c | 0.512(1) | 0.3437(8) | 0.4183(2) | 0.092(6) | 0.094(6) | 0.086(5) | 0.000(5) | 0.019(5) | 0.004(5) |
| O(1) | 8c | –0.0208(7) | 0.3607(4) | 0.4703(1) | 0.184(5) | 0.081(3) | 0.064(2) | –0.003(4) | –0.010(3) | –0.019(2) |
| O(2) | 8c | 0.0799(4) | 0.2356(5) | 0.35652(9) | 0.068(2) | 0.122(3) | 0.052(2) | –0.001(3) | 0.003(2) | –0.012(2) |
| O(3) | 8c | –0.0721(5) | 0.2239(8) | 0.3040(1) | 0.088(3) | 0.257(8) | 0.057(2) | 0.005(5) | –0.005(2) | –0.008(4) |
| N(1) | 8c | –0.0300(5) | 0.2522(4) | 0.4455(1) | 0.084(3) | 0.072(3) | 0.050(2) | –0.004(3) | –0.003(2) | –0.004(2) |
| C(1) | 8c | –0.025(1) | 0.4689(6) | 0.4470(2) | 0.20(1) | 0.079(4) | 0.090(5) | –0.007(6) | –0.002(6) | –0.008(4) |
| C(2) | 8c | –0.035(1) | 0.4329(5) | 0.4051(2) | 0.154(7) | 0.066(3) | 0.081(4) | –0.006(5) | 0.002(5) | 0.005(3) |
| C(3) | 8c | –0.0912(6) | 0.2996(5) | 0.4065(1) | 0.072(3) | 0.079(3) | 0.053(3) | 0.002(3) | –0.001(2) | 0.003(3) |
| C(4) | 8c | –0.0614(6) | 0.2168(6) | 0.3717(1) | 0.069(3) | 0.085(4) | 0.052(3) | –0.006(3) | 0.001(3) | –0.008(3) |
| C(5) | 8c | –0.1564(7) | 0.2438(8) | 0.3361(2) | 0.077(4) | 0.123(5) | 0.055(3) | –0.009(4) | 0.000(3) | –0.007(4) |
| C(6) | 8c | –0.1297(8) | 0.1653(7) | 0.4660(2) | 0.108(5) | 0.097(5) | 0.071(4) | –0.002(4) | 0.013(4) | 0.018(3) |
| C(7) | 8c | 0.1178(7) | 0.2003(7) | 0.4425(2) | 0.077(4) | 0.098(4) | 0.077(4) | 0.001(4) | –0.012(3) | –0.003(3) |
| C(8) | 8c | 0.0728(7) | 0.2347(7) | 0.3140(1) | 0.081(4) | 0.107(5) | 0.046(3) | 0.008(4) | 0.001(3) | –0.002(3) |
| C(9) | 8c | 0.135(1) | 0.3512(8) | 0.2987(2) | 0.138(7) | 0.108(6) | 0.092(5) | –0.003(6) | –0.012(5) | 0.020(5) |
| C(10) | 8c | 0.288(1) | 0.366(2) | 0.3073(3) | 0.127(9) | 0.27(2) | 0.120(8) | –0.08(1) | –0.012(7) | 0.07(1) |
| C(11) | 8c | 0.368(1) | 0.261(3) | 0.2914(3) | 0.080(6) | 0.49(3) | 0.128(8) | 0.04(2) | 0.036(5) | 0.08(2) |
| C(12) | 8c | 0.308(2) | 0.131(2) | 0.3065(4) | 0.18(1) | 0.26(2) | 0.129(8) | 0.15(1) | 0.032(9) | 0.03(1) |
| C(13) | 8c | 0.148(1) | 0.1230(9) | 0.2981(2) | 0.18(1) | 0.135(8) | 0.074(4) | 0.037(8) | 0.019(6) | –0.012(5) |

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