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

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Fe	0.69887 (7)	0.1619 (1)	0.42116 (7)	3.36 (2)
O(1)	1.0530 (4)	0.0840 (7)	0.1362 (4)	5.5 (1)
O(2)	0.9051 (4)	−0.0748 (7)	0.0664 (4)	5.4 (1)
C(1)	0.9598 (5)	0.0061 (8)	0.1443 (5)	4.0 (1)
C(2)	0.9295 (5)	0.0248 (8)	0.2527 (5)	3.7 (1)
C(3)	0.8402 (5)	−0.0390 (7)	0.2854 (5)	3.4 (1)
C(4)	0.7546 (6)	−0.1499 (9)	0.2141 (6)	4.9 (2)
C(5)	0.8178 (5)	−0.0025 (8)	0.3956 (5)	3.5 (1)
C(6)	0.8713 (5)	0.1210 (8)	0.4696 (5)	3.9 (1)
C(7)	0.8223 (6)	0.1193 (9)	0.5631 (5)	4.4 (2)
C(8)	0.7360 (7)	0.0021 (9)	0.5490 (5)	4.9 (2)
C(9)	0.7333 (6)	−0.0765 (8)	0.4471 (5)	4.3 (2)
C(10)	0.6220 (7)	0.2569 (9)	0.2708 (6)	5.5 (2)
C(11)	0.6785 (7)	0.3762 (9)	0.3394 (6)	5.6 (2)
C(12)	0.6272 (9)	0.380 (1)	0.4314 (7)	9.0 (2)
C(13)	0.5411 (7)	0.255 (1)	0.4234 (7)	6.7 (2)
C(14)	0.5393 (6)	0.182 (1)	0.3220 (6)	6.0 (2)

Table 2. *Selected bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

O(1)—C(1)	1.302 (4)	O(2)—C(1)	1.236 (4)
C(1)—C(2)	1.460 (4)	C(3)—C(5)	1.467 (4)
C(2)—C(3)	1.319 (4)	C(3)—C(4)	1.507 (4)
Fe—C(Cp) (mean)	2.035 (4)	C—C(Cp)	1.408 (5)
O(1)—C(1)—O(2)	121.8 (3)	O(1)—C(1)—C(2)	113.2 (3)
O(2)—C(1)—C(2)	125.2 (4)	C(2)—C(3)—C(4)	123.5 (3)
C(1)—C(2)—C(3)	127.1 (3)	C(3)—C(5)—C(9)	126.8 (3)
C(2)—C(3)—C(5)	120.7 (3)	C(3)—C(5)—C(6)	126.6 (3)
C(4)—C(3)—C(5)	115.8 (3)		
C—C—C(Cp) (mean)	108.0 (4)		
C(1)—C(2)—C(3)—C(5)	−176.91	C(1)—C(2)—C(3)—C(4)	2.15
H(2)—C(2)—C(3)—C(4)	−176.83	H(2)—C(2)—C(3)—C(5)	4.11

Fourier map and refined with fixed isotropic thermal parameters ($B = 4.0$ Å^2), but were only included in the structure-factor calculations in the last cycle of refinement. $\sum w(F_o - F_c)^2$ was minimized, where $w = 1/\sigma^2(F_o)$ for all observed reflections. Final $R = 0.061$, $wR = 0.067$, $S = 4.551$, $(\Delta/\sigma)_{\max} = 0.18$ for 154 variables. The largest peak in the final ΔF map was 0.969 e Å^{-3}. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations were performed on a PDP 11/44 computer, using the Enraf–Nonius *SDP* (Frenz, 1984).

The structure of the title compound is shown in Fig. 1. Positional parameters and equivalent values of the anisotropic temperature factors of non-H atoms are given in Table 1,* bond distances, selected bond angles and torsion angles are listed in Table 2.

Related literature. The structure of the butenoic acid group in the title compound is similar to that of crotonic acid $CH_3CH{=}CHCOOH$ (Shimizu, Kekka, Kashino & Haisa, 1974). The structure of the title compound shows the (*E*) stereochemical conformation, which is compared with (*Z*)-(1,2-diphenylenethenyl)ferrocence (Cardin Crawford, Watts & Hathaway, 1979).

The author is grateful to Professor Yin for supplying crystals.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55657 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0554]

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3-Methoxyazetidinium Chloride

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Abstract. $C_4H_{10}NO^+.Cl^-$, $M_r = 123.58$, triclinic, $P\bar{1}$, $a = 4.9016$ (8), $b = 7.975$ (1), $c = 8.4134$ (7) Å, $\alpha = 100.40$ (1), $\beta = 102.417$ (1), $\gamma = 94.083$ (1)°, $V = 313.8$ (2) Å^3, $Z = 2$, $D_x = 1.308$ (2) g cm^{-3}, monochromatized Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 46.08$ cm^{-1}, $F(000) = 132$, $T = 122$ K, final $R = 0.0474$, for 1262

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unique observed reflections. The 3-methoxyazetidinium cations form infinite chains *via* hydrogen bonding with the chloride ions. The chains are directed along the *a* axis. The puckering angle is 168.7 (2)°.

Experimental. *tert*-Butyl 3-hydroxy-1-azetidinylcarboxylate was prepared from azetidin-3-ol (Gajhede, Anthoni, Christophersen & Nielsen, 1989) and 3-pivaloyloxy-2,2-dimethylpropanoic acid according to the procedure of Tarbell, Yamamoto & Pope (1972). An 85% yield of greasy crystals, m.p. 309–310 K, was achieved. Analysis for $C_8H_{15}NO_3$: calculated: C 55.47, H 8.73, N 8.09%; found: C 55.29, H 8.94, N 7.97%. ^{13}C NMR ($CDCl_3$): δ 155.6 (C=O), 78.6 (C′), 59.4 (CH), 58.2 (CH_2), 27.4 (CH_3). Electron-impact mass spectrometry (EIMS): m/z 173 (M^+, 10), 158 ($M - 15$, 40), 130 ($M - 43$, 100%). Methylation of *tert*-butyl 3-hydroxy-1-azetidinylcarboxylate was accomplished with methyl iodide following the procedure outlined by Johnstone & Rose (1979) to give *tert*-butyl 3-methoxy-1-azetidinylcarboxylate. Colourless oil, yield 83%. Analysis for $C_9H_{17}O_3N$: calculated: C 57.73, H 9.15, N 7.48%; found: C 57.79, H 8.94, N 7.42%. ^{13}C NMR ($CDCl_3$): δ 155.5 (C=O), 78.4 (C′), 68.3 (CH_3O), 55.4, *d* 55.1 (CH, CH_2), 27.6 (CH_3). EIMS: m/z 187 (M^+, 15), 155 ($M - 32$, 100%).

3-Methoxyazetidinium chloride was prepared by dissolving 187 mg (1 mmol) of *tert*-butyl 3-hydroxy-1-azetidinylcarboxylate in a mixture of 2 ml ethanol and 1 ml 2 *M* hydrochloric acid. This was stirred for 30 min at ambient temperature. The reaction mixture was taken to dryness and the residue recrystallized from absolute ethanol. Colourless crystals, m.p. 368.0–368.5 K; yield 89%. Analysis for $C_4H_{10}ClNO$: calculated: C 38.87, H 8.10, N 11.34, Cl 28.74%; found: C 38.66, H 8.29, N 11.05, Cl 28.75%. ^{13}C NMR (D_2O): δ 72.3 (CH_3), 58.1 (CH), 55.4 (CH_2). Fast-atom bombardment mass spectrometry (FABMS): m/z 88, ($M + H$).

The crystal used for data collection had the dimensions 0.05 × 0.15 × 0.4 mm. Preliminary film data indicated the triclinic space group $P\bar{1}$. Diffraction data were collected on an Enraf–Nonius CAD-4 diffractometer. The crystal was cooled to 122 ± 0.8 K in a stream of N_2 gas. The unit-cell dimensions were determined from a least-squares fit of 25 reflections, well distributed in reciprocal space, $40 < \theta < 43°$. 2342 reflections were measured with $\omega/2\theta$ scans, $\theta \leq 75.0°$, *hkl* ranges: *h*: −4 to 6, *k*: −10 to 10, *l*: −10 to 10. Three reflections (10$\bar{3}$, 032, 020) were measured every 2.8 h to check for possible decay of the crystal. No change was detected. The intensity data were corrected for Lorentz–polarization and absorption effects. They were averaged to 1302 independent reflections [$R_{int}(I) = 0.034$], 1252 of

Table 1. *Positional and displacement parameters* (Å^2)

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}].$$

	x	*y*	*z*	B_{eq}/B_{iso}
O(5)	0.2023 (2)	0.69425 (13)	0.09758 (12)	0.0138 (4)
N(3)	−0.0356 (3)	0.71734 (18)	0.40891 (16)	0.0186 (5)
C(1)	−0.0557 (3)	0.69132 (16)	0.14574 (16)	0.0142 (5)
C(2*A*)	−0.0678 (3)	0.56779 (18)	0.2637 (2)	0.0221 (4)
C(2*B*)	−0.0681 (3)	0.83931 (17)	0.29015 (18)	0.0159 (5)
C(6)	0.2280 (4)	0.82414 (19)	0.00253 (19)	0.0183 (6)
Cl(1)	0.44676 (6)	0.23497 (4)	0.35914 (3)	0.0003 (2)
H(11)	−0.175 (5)	0.679 (3)	0.045 (3)	0.027 (5)
H(2*A*1)	0.091 (5)	0.502 (3)	0.287 (3)	0.030 (5)
H(2*A*2)	−0.229 (4)	0.486 (3)	0.256 (2)	0.018 (4)
H(2*B*1)	0.100 (5)	0.934 (3)	0.314 (3)	0.033 (6)
H(2*B*2)	−0.252 (5)	0.883 (3)	0.291 (3)	0.027 (5)
H(31)	−0.179 (6)	0.710 (3)	0.454 (3)	0.035 (6)
H(32)	0.161 (7)	0.734 (4)	0.459 (3)	0.049 (7)
H(61)	0.093 (7)	0.810 (4)	−0.096 (4)	0.044 (8)
H(62)	0.252 (6)	0.940 (4)	0.064 (3)	0.034 (6)
H(63)	0.388 (6)	0.821 (4)	−0.038 (3)	0.034 (6)

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

O(5)—C(1)	1.4084 (18)	C(1)—H(11)	0.90 (2)
O(5)—C(6)	1.4326 (19)	C(2*B*)—H(2*B*2)	0.99 (3)
N(3)—C(2*B*)	1.508 (2)	C(2*B*)—H(2*B*1)	1.04 (2)
N(3)—C(2*A*)	1.518 (2)	C(6)—H(61)	0.93 (3)
C(1)—C(2*A*)	1.526 (2)	C(6)—H(62)	0.96 (3)
C(1)—C(2*B*)	1.548 (2)	C(6)—H(63)	0.92 (3)
N(3)—H(31)	0.87 (3)	C(2*A*)—H(2*A*1)	0.97 (2)
N(3)—H(32)	0.95 (3)	C(2*A*)—H(2*A*2)	0.97 (2)
C(1)—O(5)—C(6)	110.96 (12)	N(3)—C(2*B*)—H(2*B*1)	114.1 (13)
C(2)—N(3)—C(2*A*)	89.83 (10)	C(1)—C(2*B*)—H(2*B*2)	117.6 (14)
O(5)—C(1)—C(2*B*)	114.44 (11)	C(1)—C(2*B*)—H(2*B*1)	111.9 (14)
O(5)—C(1)—C(2*A*)	110.78 (11)	H(2*B*2)—C(2*B*)—H(2*B*1)	115 (2)
C(2)—C(1)—C(2*A*)	88.05 (10)	O(5)—C(6)—H(61)	115 (2)
N(3)—C(2*B*)—C(1)	90.26 (10)	O(5)—C(6)—H(62)	115.2 (16)
N(3)—C(2*A*)—C(1)	90.74 (11)	O(5)—C(6)—H(63)	111.9 (19)
C(2*B*)—N(3)—H(31)	112.0 (17)	H(61)—C(6)—H(62)	111 (3)
C(2*B*)—N(3)—H(32)	103.6 (18)	H(61)—C(6)—H(63)	100 (3)
C(2*A*)—N(3)—H(31)	109.7 (16)	H(62)—C(6)—H(63)	102 (3)
C(2*A*)—N(3)—H(32)	104.2 (17)	N(3)—C(2*A*)—H(2*A*1)	108.2 (14)
H(31)—N(3)—H(32)	130 (2)	N(3)—C(2*A*)—H(2*A*2)	111.5 (11)
O(5)—C(1)—H(11)	99.6 (16)	C(1)—C(2*A*)—H(2*A*1)	117.3 (14)
C(2*B*)—C(1)—H(11)	120.8 (16)	C(1)—C(2*A*)—H(2*A*2)	124.5 (11)
C(2*A*)—C(1)—H(11)	123.9 (16)	H(2)—C(2*A*)—H(2*A*2)	103 (2)
C(6)—O(5)—C(1)—C(2*B*)	−75.72 (14)	O(5)—C(1)—C(2*B*)—N(3)	−104.01 (12)
C(6)—O(5)—C(1)—C(2*A*)	−173.33 (11)	C(2*B*)—C(1)—C(2*A*)—N(3)	−7.93 (11)
C(2*A*)—N(3)—C(2*B*)—C(1)	−8.02 (11)	C(2*A*)—C(1)—C(2*B*)—N(3)	7.99 (11)
C(2*B*)—N(3)—C(2*A*)—C(1)	8.14 (11)	O(5)—C(1)—C(2*A*)—N(3)	107.53 (12)

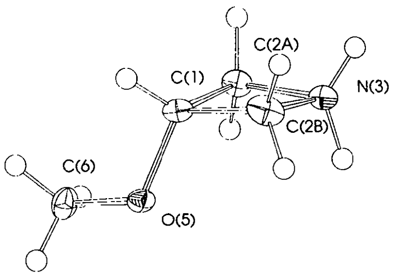

Fig. 1. *PLATON* (Spek, 1990) drawing of the 3-methoxyazetidinium cation. The thermal ellipsoids for non-H atoms are drawn at the 50% probability level.

which with $F_o^2 > 3.0\sigma(F_o^2)$ were considered observed. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and least-squares refined on F with the *SHELX*76 (Sheldrick, 1976) program. The H atoms were located and included in Fourier summations. 104 parameters were refined to $R = 0.047$, $wR = 0.048$ and $S = 0.34$ with $w = [\sigma^2(F) + 0.045F^2]^{-1}$; max. and min. heights in final $\Delta\rho$ map are 0.54 (3) and −0.84 (3) e Å^{-3}, respectively, $(\Delta/\rho)_{\max} = 0.008$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV), except for H atoms which were taken from Stewart, Davidson & Simpson (1965).

Final atomic coordinates and isotropic or equivalent isotropic displacement parameters are listed in Table 1.* Bond distances and angles are given in Table 2. All geometric calculations were calculated with program *PLATON* (Spek, 1990). A perspective drawing of one of the molecules is shown in Fig. 1 giving the atomic numbering scheme.

Each chloride ion accepts two hydrogen bonds: N(3)—H(31)···Cl(1) with angle 159 (2)° and N(3)—H(32)···Cl(1) with angle 162 (2)°. The N(3)···Cl(1) distances are 3.090 (2) and 3.072 (2) Å, respectively. The effects of hydrogen bonding on the puckering angle seems to cancel out in this system owing to the symmetric forces on the N atom.

Related literature. This structure determination is part of an investigation of the conformational properties of the azetidinol system (Gajhede *et al.*, 1989; Hagen, Volden, Gajhede, Anthoni, Christophersen & Nielsen, 1991).

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Structure and Configuration of the Michael Adduct of 2-(Methylthio)thiolane *S*-Oxide and Butenone at 173 K*

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Abstract. (*E*)-2-Methylthio-2-(3-oxobutyl)thiolane 1-oxide, $C_9H_{16}O_2S_2$, $M_r = 220.36$, orthorhombic, $P2_1ab$ (No. 29, non-standard), $a = 7.737$ (3), $b = 9.533$ (2), $c = 14.725$ (5) Å, $V = 1086.1$ (6) Å^3, $Z = 4$, $D_x = 1.348$ g cm^{-3}, $\lambda(\text{Cu } K\alpha_1) = 1.54051$ Å, $\mu = 41.3$ cm^{-1}, $F(000) = 472$, $T = 173$ K, $R = 0.063$ for 990 observed reflections $[|F_o| \geq 4\sigma(F_o)]$. The thiolane ring in (1) exhibits a half-chair conformation with quasi-axial positions of the sulfoxide O atom and the methylthio group. The *E* (*trans*) isomer (1) is the

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* C,C-Coupling with Sulfur-Stabilized Carbanions. 3. Part 2: Böge, Brunck, Schwär & Voss (1992).

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