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(*E*)-Methyl 3-[3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxamido]thiophene-2-carboxylate

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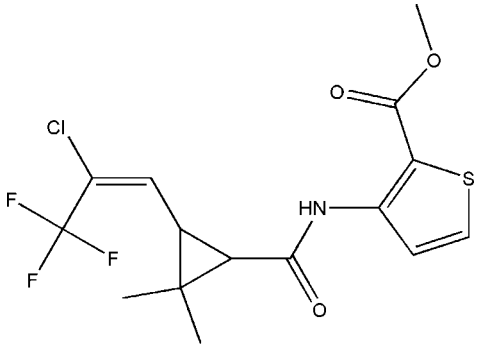
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.055; wR factor = 0.164; data-to-parameter ratio = 14.2.

The title compound, $C_{15}H_{15}ClF_3NO_3S$, was synthesized from 3-[(*E*)-2-chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropanecarboxylic acid and methyl 3-aminothiophene-2-carboxylate. The propenyl and carboxamide substituents lie on the same side of the cyclopropane ring plane, with the two methyl substituents on either side of the plane. The molecular conformation is stabilized by an N—H···O hydrogen bond and the crystal packing shows C—H···O hydrogen bonds.

Related literature

For related literature, see: Liu & Yan (2007); Punja (1981).



Experimental

Crystal data

$C_{15}H_{15}ClF_3NO_3S$
M_r = 381.79
Triclinic, $P\overline{1}$
a = 7.9342 (12) Å
b = 8.4940 (14) Å
c = 13.8971 (16) Å
α = 87.144 (8)°
β = 85.696 (8)°
γ = 77.821 (8)°
V = 912.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 294 (2) K
0.32 × 0.28 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.892, T_{max} = 0.918
4678 measured reflections
3188 independent reflections
2206 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.164
S = 1.03
3188 reflections
224 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2	0.85 (3)	2.12 (3)	2.809 (3)	138 (3)
C5—H5···O2^{i}	0.93	2.45	3.307 (4)	153

Symmetry code: (i) $x-1, y, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997; data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2628).

References

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supplementary materials

Acta Cryst. (2007). E**63**, o4877 [**doi:10.1107/S1600536807061120**]

(*E*)-Methyl 3-[3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxamido]thiophene-2-carboxylate

F.-Y. Yan and D.-Q. Liu

Comment

The title compound is known to possess insecticidal activity (Punja, 1981). The propenyl and carboxamide substituents lie on the same side of the cyclopropane ring plane, with two methyl substituents, on either side of this plane. The molecular conformation is stabilized by a N—H···O hydrogen bond and the crystal packing shows C—H···O hydrogen bonds.

Experimental

The title compound was prepared according to the method of Liu *et al.* (2007). The product was recrystallized from methanol and ethyl acetate (5:1, *v*/*v*) over 4 days at ambient temperature, giving colourless prismatic crystals.

Refinement

H atoms were positioned geometrically with C—H = 0.93–0.98 Å and refined using riding model with U_{iso}(H) = 1.2Ueq(carrier). H atom of N—H was located from difference map and refinded freely.

Figures

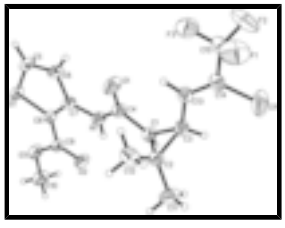


Fig. 1. The molecular structure of the title compound drawn with 30% probability ellipsoids. H atoms are drawn as spheres of arbitrary radius.

(*E*)-Methyl 3-[3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2- dimethylcyclopropanecarboxamido]thiophene-2-carboxylate

Crystal data

$C_{15}H_{15}ClF_3NO_3S$	$Z = 2$
$M_r = 381.79$	$F_{000} = 392$
Triclinic, $P\overline{1}$	$D_x = 1.390$ Mg m^{-3}
$a = 7.9342$ (12) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 8.4940$ (14) Å	Cell parameters from 1854 reflections
$c = 13.8971$ (16) Å	$\theta = 2.8$–$25.1°$
$\alpha = 87.144$ (8)°	$\mu = 0.37$ mm^{-1}

β = 85.696 (8)º	T = 294 (2) K
γ = 77.821 (8)º	Prism, colorless
V = 912.4 (2) Å^3	0.32 × 0.28 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3188 independent reflections
Radiation source: fine-focus sealed tube	2206 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.018
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -9 \rightarrow 8$
T_{min} = 0.892, T_{max} = 0.918	$k = -7 \rightarrow 10$
4678 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.055	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.164	$w = 1/[\sigma^2(F_o^2) + (0.074P)^2 + 0.6746P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
3188 reflections	$\Delta\rho_{max}$ = 0.88 e Å^{-3}
224 parameters	$\Delta\rho_{min}$ = −0.63 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C14	0.7383 (5)	−0.2156 (4)	0.6067 (2)	0.0576 (9)

C15	0.6041 (6)	−0.2568 (5)	0.5474 (3)	0.0746 (11)
Cl1	0.93950 (17)	−0.33682 (19)	0.58260 (10)	0.1170 (6)
F1	0.6345 (4)	−0.2276 (4)	0.45312 (18)	0.1239 (11)
F2	0.5909 (5)	−0.4098 (4)	0.5577 (2)	0.1330 (12)
F3	0.4476 (4)	−0.1713 (4)	0.5698 (2)	0.1175 (10)
S1	0.23568 (10)	0.41449 (12)	1.12716 (6)	0.0591 (3)
N1	0.5903 (3)	0.1716 (3)	0.94587 (19)	0.0509 (7)
H1A	0.681 (4)	0.180 (4)	0.972 (2)	0.045 (9)*
O1	0.4775 (3)	0.1059 (4)	0.81065 (19)	0.0813 (9)
O2	0.7451 (3)	0.2882 (3)	1.09510 (17)	0.0642 (7)
O3	0.5618 (3)	0.4482 (3)	1.19945 (17)	0.0664 (7)
C1	0.7882 (4)	0.0441 (4)	0.8196 (2)	0.0515 (8)
H1	0.8686	0.0144	0.8708	0.062*
C2	0.8663 (4)	0.1144 (4)	0.7273 (2)	0.0568 (9)
C3	0.8370 (4)	−0.0577 (4)	0.7302 (2)	0.0544 (8)
H3	0.9429	−0.1407	0.7348	0.065*
C4	0.6038 (4)	0.1073 (4)	0.8559 (2)	0.0523 (8)
C5	0.1460 (4)	0.3306 (5)	1.0390 (3)	0.0616 (9)
H5	0.0275	0.3411	1.0357	0.074*
C6	0.2653 (4)	0.2472 (4)	0.9744 (3)	0.0565 (9)
H6	0.2377	0.1958	0.9220	0.068*
C7	0.4381 (4)	0.2480 (4)	0.9970 (2)	0.0448 (7)
C8	0.4426 (4)	0.3336 (4)	1.0790 (2)	0.0464 (7)
C9	0.5988 (4)	0.3520 (4)	1.1234 (2)	0.0494 (8)
C10	0.7086 (5)	0.4736 (6)	1.2501 (3)	0.0818 (13)
H10A	0.7623	0.3743	1.2814	0.123*
H10B	0.6688	0.5537	1.2976	0.123*
H10C	0.7909	0.5093	1.2046	0.123*
C11	1.0542 (5)	0.1314 (6)	0.7309 (3)	0.0886 (14)
H11A	1.1142	0.0467	0.7717	0.133*
H11B	1.0566	0.2339	0.7564	0.133*
H11C	1.1094	0.1243	0.6670	0.133*
C12	0.7536 (6)	0.2431 (5)	0.6678 (3)	0.0771 (11)
H12A	0.7961	0.2367	0.6013	0.116*
H12B	0.7560	0.3474	0.6906	0.116*
H12C	0.6370	0.2269	0.6737	0.116*
C13	0.7063 (4)	−0.1026 (4)	0.6711 (2)	0.0584 (9)
H13	0.5928	−0.0461	0.6804	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C14	0.069 (2)	0.053 (2)	0.0512 (19)	−0.0107 (17)	−0.0092 (16)	−0.0099 (16)
C15	0.098 (3)	0.065 (3)	0.068 (3)	−0.025 (2)	−0.024 (2)	−0.014 (2)
Cl1	0.0915 (8)	0.1380 (12)	0.1102 (10)	0.0219 (8)	−0.0186 (7)	−0.0751 (9)
F1	0.140 (3)	0.190 (3)	0.0610 (16)	−0.068 (2)	−0.0300 (15)	−0.0095 (17)
F2	0.191 (3)	0.080 (2)	0.152 (3)	−0.061 (2)	−0.072 (2)	−0.0102 (18)
F3	0.0820 (18)	0.145 (3)	0.136 (2)	−0.0271 (18)	−0.0332 (17)	−0.054 (2)

S1	0.0384 (4)	0.0735 (6)	0.0617 (6)	−0.0048 (4)	0.0053 (4)	−0.0072 (4)
N1	0.0363 (14)	0.0666 (19)	0.0516 (16)	−0.0110 (12)	−0.0062 (12)	−0.0134 (13)
O1	0.0467 (14)	0.124 (2)	0.0735 (17)	−0.0060 (14)	−0.0139 (12)	−0.0396 (16)
O2	0.0357 (12)	0.0900 (18)	0.0681 (15)	−0.0116 (11)	0.0000 (10)	−0.0253 (13)
O3	0.0479 (13)	0.0865 (18)	0.0653 (15)	−0.0075 (12)	−0.0066 (11)	−0.0309 (13)
C1	0.0444 (17)	0.061 (2)	0.0494 (18)	−0.0067 (15)	−0.0102 (14)	−0.0120 (15)
C2	0.0545 (19)	0.065 (2)	0.056 (2)	−0.0200 (17)	−0.0035 (15)	−0.0178 (17)
C3	0.0475 (18)	0.058 (2)	0.056 (2)	−0.0029 (15)	−0.0073 (14)	−0.0193 (16)
C4	0.0440 (17)	0.062 (2)	0.0528 (19)	−0.0110 (15)	−0.0080 (14)	−0.0126 (16)
C5	0.0350 (16)	0.079 (3)	0.070 (2)	−0.0136 (16)	−0.0023 (15)	0.0043 (19)
C6	0.0402 (17)	0.068 (2)	0.065 (2)	−0.0162 (15)	−0.0086 (15)	−0.0029 (17)
C7	0.0362 (15)	0.0507 (18)	0.0481 (17)	−0.0107 (13)	−0.0043 (12)	0.0014 (14)
C8	0.0357 (15)	0.0545 (19)	0.0479 (18)	−0.0083 (13)	0.0019 (12)	−0.0019 (14)
C9	0.0410 (17)	0.060 (2)	0.0483 (18)	−0.0140 (14)	0.0003 (13)	−0.0051 (15)
C10	0.061 (2)	0.108 (3)	0.082 (3)	−0.017 (2)	−0.017 (2)	−0.034 (2)
C11	0.066 (3)	0.130 (4)	0.082 (3)	−0.046 (3)	0.005 (2)	−0.028 (3)
C12	0.095 (3)	0.068 (3)	0.072 (3)	−0.024 (2)	−0.011 (2)	−0.004 (2)
C13	0.056 (2)	0.057 (2)	0.063 (2)	−0.0095 (16)	−0.0102 (16)	−0.0148 (17)

Geometric parameters (Å, °)

C14—C13	1.317 (5)	C2—C3	1.526 (5)
C14—C15	1.501 (5)	C2—C11	1.532 (5)
C14—Cl1	1.726 (4)	C3—C13	1.491 (4)
C15—F3	1.323 (5)	C3—H3	0.9800
C15—F2	1.326 (5)	C5—C6	1.363 (5)
C15—F1	1.335 (5)	C5—H5	0.9300
S1—C5	1.716 (4)	C6—C7	1.430 (4)
S1—C8	1.738 (3)	C6—H6	0.9300
N1—C4	1.377 (4)	C7—C8	1.389 (4)
N1—C7	1.404 (4)	C8—C9	1.465 (4)
N1—H1A	0.85 (3)	C10—H10A	0.9600
O1—C4	1.225 (4)	C10—H10B	0.9600
O2—C9	1.218 (4)	C10—H10C	0.9600
O3—C9	1.345 (4)	C11—H11A	0.9600
O3—C10	1.464 (4)	C11—H11B	0.9600
C1—C4	1.506 (4)	C11—H11C	0.9600
C1—C3	1.528 (4)	C12—H12A	0.9600
C1—C2	1.532 (5)	C12—H12B	0.9600
C1—H1	0.9800	C12—H12C	0.9600
C2—C12	1.512 (5)	C13—H13	0.9300
C13—C14—C15	124.1 (4)	C6—C5—H5	123.3
C13—C14—Cl1	123.7 (3)	S1—C5—H5	123.3
C15—C14—Cl1	112.1 (3)	C5—C6—C7	111.9 (3)
F3—C15—F2	105.7 (4)	C5—C6—H6	124.0
F3—C15—F1	105.5 (4)	C7—C6—H6	124.0
F2—C15—F1	106.7 (3)	C8—C7—N1	121.5 (3)
F3—C15—C14	112.4 (3)	C8—C7—C6	112.2 (3)
F2—C15—C14	113.5 (4)	N1—C7—C6	126.3 (3)

F1—C15—C14	112.4 (4)	C7—C8—C9	125.8 (3)
C5—S1—C8	91.01 (16)	C7—C8—S1	111.4 (2)
C4—N1—C7	126.8 (3)	C9—C8—S1	122.7 (2)
C4—N1—H1A	120 (2)	O2—C9—O3	123.7 (3)
C7—N1—H1A	113 (2)	O2—C9—C8	124.3 (3)
C9—O3—C10	116.6 (3)	O3—C9—C8	112.0 (3)
C4—C1—C3	123.0 (3)	O3—C10—H10A	109.5
C4—C1—C2	121.4 (3)	O3—C10—H10B	109.5
C3—C1—C2	59.8 (2)	H10A—C10—H10B	109.5
C4—C1—H1	114.0	O3—C10—H10C	109.5
C3—C1—H1	114.0	H10A—C10—H10C	109.5
C2—C1—H1	114.0	H10B—C10—H10C	109.5
C12—C2—C3	119.2 (3)	C2—C11—H11A	109.5
C12—C2—C1	119.5 (3)	C2—C11—H11B	109.5
C3—C2—C1	59.9 (2)	H11A—C11—H11B	109.5
C12—C2—C11	115.6 (3)	C2—C11—H11C	109.5
C3—C2—C11	115.9 (3)	H11A—C11—H11C	109.5
C1—C2—C11	115.3 (3)	H11B—C11—H11C	109.5
C13—C3—C2	121.1 (3)	C2—C12—H12A	109.5
C13—C3—C1	122.9 (3)	C2—C12—H12B	109.5
C2—C3—C1	60.2 (2)	H12A—C12—H12B	109.5
C13—C3—H3	114.1	C2—C12—H12C	109.5
C2—C3—H3	114.1	H12A—C12—H12C	109.5
C1—C3—H3	114.1	H12B—C12—H12C	109.5
O1—C4—N1	122.5 (3)	C14—C13—C3	125.4 (3)
O1—C4—C1	124.6 (3)	C14—C13—H13	117.3
N1—C4—C1	112.9 (3)	C3—C13—H13	117.3
C6—C5—S1	113.4 (2)		
C13—C14—C15—F3	−2.7 (6)	C2—C1—C4—N1	−118.3 (3)
Cl1—C14—C15—F3	176.0 (3)	C8—S1—C5—C6	−1.0 (3)
C13—C14—C15—F2	−122.6 (4)	S1—C5—C6—C7	0.8 (4)
Cl1—C14—C15—F2	56.1 (5)	C4—N1—C7—C8	−168.2 (3)
C13—C14—C15—F1	116.2 (5)	C4—N1—C7—C6	12.8 (5)
Cl1—C14—C15—F1	−65.1 (4)	C5—C6—C7—C8	−0.1 (4)
C4—C1—C2—C12	−3.8 (4)	C5—C6—C7—N1	179.0 (3)
C3—C1—C2—C12	108.7 (3)	N1—C7—C8—C9	−1.7 (5)
C4—C1—C2—C3	−112.5 (3)	C6—C7—C8—C9	177.4 (3)
C4—C1—C2—C11	141.0 (3)	N1—C7—C8—S1	−179.8 (2)
C3—C1—C2—C11	−106.5 (4)	C6—C7—C8—S1	−0.7 (3)
C12—C2—C3—C13	3.5 (5)	C5—S1—C8—C7	1.0 (3)
C1—C2—C3—C13	112.7 (3)	C5—S1—C8—C9	−177.2 (3)
C11—C2—C3—C13	−141.7 (3)	C10—O3—C9—O2	−0.7 (5)
C12—C2—C3—C1	−109.2 (3)	C10—O3—C9—C8	179.3 (3)
C11—C2—C3—C1	105.6 (3)	C7—C8—C9—O2	−3.0 (5)
C4—C1—C3—C13	0.3 (5)	S1—C8—C9—O2	175.0 (3)
C2—C1—C3—C13	−109.8 (4)	C7—C8—C9—O3	177.0 (3)
C4—C1—C3—C2	110.0 (4)	S1—C8—C9—O3	−5.0 (4)
C7—N1—C4—O1	−2.5 (6)	C15—C14—C13—C3	−179.3 (3)
C7—N1—C4—C1	176.1 (3)	Cl1—C14—C13—C3	2.2 (6)

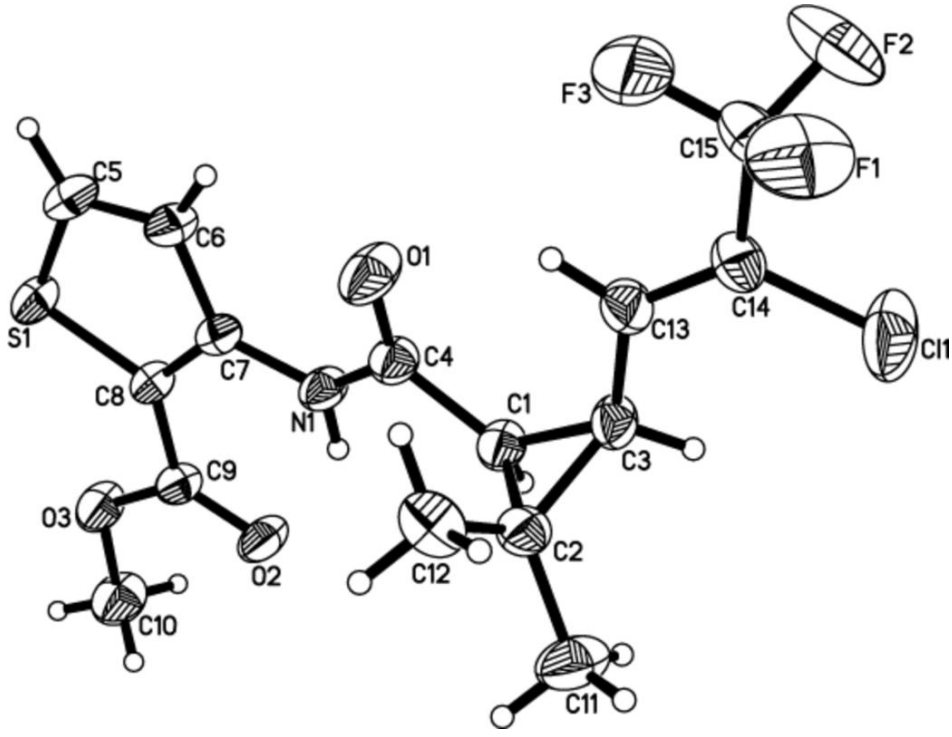
C3—C1—C4—O1	−12.0 (6)	C2—C3—C13—C14	122.6 (4)
C2—C1—C4—O1	60.2 (5)	C1—C3—C13—C14	−164.9 (4)
C3—C1—C4—N1	169.5 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O2	0.85 (3)	2.12 (3)	2.809 (3)	138 (3)
C5—H5···O2^{i}	0.93	2.45	3.307 (4)	153

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1


F2
F3
C15
F1
C5
C6
O1
S1
C7
C4
C13
C14
Cl1
C8
N1
C1
C3
C9
O3
O2
C12
C2
C10
C11