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3-[(*E*)-2-Chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethyl-*N*-(3-pyridyl)cyclopropanecarboxamide acetone hemisolvate

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In the title compound, $C_{14}H_{14}ClF_3N_2O\cdot0.5C_3H_6O$, the pyridine ring makes a dihedral angle of 73.0 (3)° with the cyclopropane ring. The amide NH group and the pyridine N atom are linked by an intermolecular N—H···N hydrogen bond.

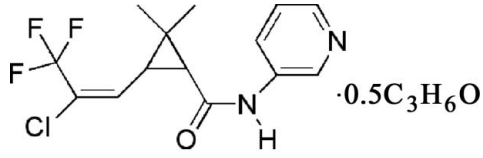
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.059
wR factor = 0.210
Data-to-parameter ratio = 12.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

3-[(*E*)-2-Chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropanecarboxylic acid is a very important intermediate for tefluthrin, an insecticide controlling a wide range of soil insect pests in maize, sugar beet, and other crops (Punja 1981). A pyridine ring is often used as an active component in pesticide discovery (Elbert *et al.*, 2000). The title compound, (I), contains both active parts and may show some insecticidal activity.



(I)

The dihedral angle between the pyridine and cyclopropane rings is 73.0 (3)°. An acetone molecule is found to cocrystallize in the structure and shows twofold disorder about an inversion center. The amide NH group and the pyridine N atom are linked by an intermolecular N—H···N hydrogen bond. The packing can be described as a dimeric arrangement of molecules linked through N—H···N hydrogen bonds (Fig. 2 and Table 1).

Experimental

3-[(*E*)-2-Chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropanecarboxylic acid (0.97 g, 4 mmol) was dispersed in $SOCl_2$ (15 ml) and a drop of anhydrous DMF was added. The mixture was heated under reflux for 4 h. $SOCl_2$ was removed by rotary evaporatio. The crude product was dissolved in anhydrous toluene (20 ml) containing 3-aminopyridine (0.4 g). Triethylamine (0.45 g) was added dropwise to the system, until white fumes ceased to be given off. After 12 h stirring at room temperature, the reaction mixture was treated with *n*-hexane (30 ml). The off-white precipitate was filtered off and purified by silica chromotography (EtOAc–hexene = 1:8) to give the title compound (yield: 1.02 g, 80%). Recrystallization of the product from acetone and a small amount of water (50:1) over a period of 8 d at ambient temperature gave colorless single crystals of (I) suitable for X-ray analysis.

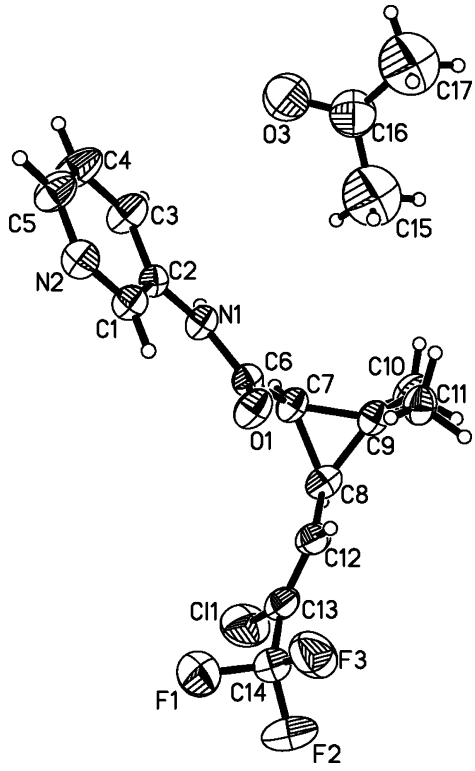


Figure 1
The molecular structure of (I), with 30% probability ellipsoids. H atoms are drawn as spheres of arbitrary radius. Only one component is shown for each disordered unit.

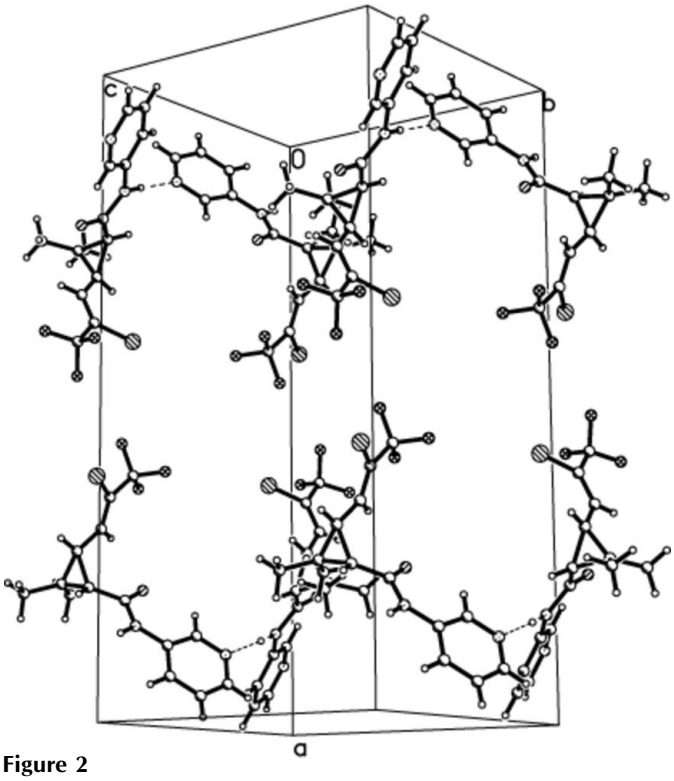


Figure 2
The crystal structure of (I). Only one component is shown for each disordered unit. Dashed lines indicate hydrogen bonds.

Crystal data

$C_{14}H_{14}ClF_3N_2O{\cdot}0.5C_3H_6O$
M_r = 347.76
Monoclinic, $C2/c$
a = 24.274 (5) Å
b = 12.330 (2) Å
c = 11.4763 (18) Å
β = 90.761 (6)°
V = 3434.7 (10) Å^3
Z = 8
D_x = 1.345 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 (2) K
Block, colorless
0.16 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.960, T_{max} = 0.975
8546 measured reflections
3025 independent reflections
1477 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.210
S = 1.03
3025 reflections
250 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1093P)^2 + 0.4942P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2^{i}	0.94 (3)	2.00 (3)	2.939 (4)	176 (3)

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

The amide H atom was located in a difference map and refined freely. Other H atoms were positioned geometrically, with C—H = 0.93–0.98Å, and refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(carrier). The occupancy factors for the two disordered acetone sites are 0.5. The three F atoms are disordered over two sites with refined occupancy factors of 0.743 (5) and 0.257 (5).

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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