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(*E*)-3-(2-Chloro-3,3,3-trifluoroprop-1-enyl)-*N*-(2,5-dimethylphenyl)-2,2-dimethylcyclopropanecarboxamide

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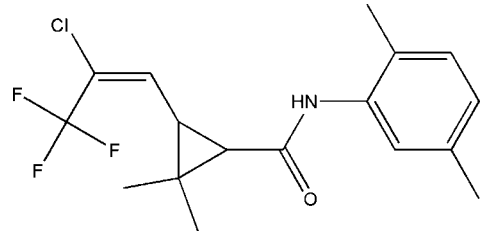
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.056; wR factor = 0.179; data-to-parameter ratio = 14.3.

The benzene ring in the title compound, $C_{17}H_{19}ClF_3NO$, makes a dihedral angle of 66.7 (3)° with the plane of the cyclopropane ring. The crystal packing is stabilized by an N—H···O hydrogen bond.

Related literature

For related literature, see: Liu *et al.* (2006); Punja (1981); Zhang (2005).



Experimental

Crystal data

$C_{17}H_{19}ClF_3NO$
M_r = 345.78
Orthorhombic, *Pbcn*
a = 16.445 (3) Å
b = 9.5147 (17) Å
c = 21.856 (4) Å
V = 3419.9 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.946, T_{max} = 0.965
16642 measured reflections
3027 independent reflections
1746 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.179
S = 1.01
3027 reflections
212 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.44	3.214 (4)	149

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

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*.

This work was supported by the National Natural Science Foundation (No. 20376059)

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2582).

References

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supplementary materials

Acta Cryst. (2007). E**63**, o4637 [**doi:10.1107/S1600536807055924**]

(*E*)-3-(2-Chloro-3,3,3-trifluoroprop-1-enyl)-*N*-(2,5-dimethylphenyl)-2,2-dimethylcyclopropanecarboxamide

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

Comment

3-((*E*)-2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethyl cyclopropanecarboxylic acid is a very important intermediate for tefluthrin, a important insecticide controlling a wide range of soil insect pests in maize, sugar beet, and other crops (Punja, 1981). 2,5-dimethylbenzenamine containing pesticides have the advantage of low toxicity, high activity and low residues (Zhang, 2005). The title compound containing both these active parts may have some insecticide activity. The present X-ray crystal structure analysis was undertaken in order to study the stereochemistry and crystal packing of the title compound. The dihedral angles between the benzene moiety and the cycloprapane group is 66.7 (3)°. The crystal packing is stabilized by an N—H···O hydrogen bond.

Experimental

The title compound was prepared according to the method of Liu *et al.* (2006). 3-((*E*)-2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2- dimethylcyclopropanecarboxylic acid (0.97 g, 4.0 mmol) was dispersed in $SOCl_2$ (15 ml), and a drop of anhydrous DMF was added. The mixture was heated to reflux for 2 h. $SOCl_2$ was removed by rotoevapor. The crude product could be directly disolved in anhydrous toluene, mixed with 2,5-dimethylbenzenamine (0.50 g, 4.1 mmol). Triethylamine was dropped into the system, and a white fume was coming out. After 8 h stirring at room temperature, the reaction mixture was treated with hexane. Recrystallization of the product from methanol and a small amount of water (70:1) overnight at ambient temperature gave colorless rod like crystals.

Refinement

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

Figures

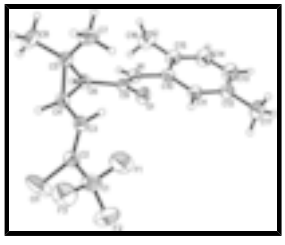

Fig. 1. The molecular structure of the title compound, drawn with 30% probability ellipsoids. H atoms are drawn as spheres of arbitrary radius.

Fig. 2. The packing of the title compund, viewed along the *b* axis.

(*E*)-3-(2-Chloro-3,3,3-trifluoroprop-1-enyl)-*N*-(2,5-dimethylphenyl)- 2,2-dimethylcyclopropanecarboxamide

Crystal data

$C_{17}H_{19}ClF_3NO$	$D_x = 1.343$ Mg m^{-3}
$M_r = 345.78$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, *Pbcn*	Cell parameters from 3270 reflections
$a = 16.445$ (3) Å	$\theta = 2.5$–22.5°
$b = 9.5147$ (17) Å	$\mu = 0.26$ mm^{-1}
$c = 21.856$ (4) Å	$T = 294$ (2) K
$V = 3419.9$ (11) Å^3	Rod, colorless
$Z = 8$	0.22 × 0.20 × 0.14 mm
$F_{000} = 1440$	

Data collection

Bruker SMART CCD area-detector diffractometer	3027 independent reflections
Radiation source: fine-focus sealed tube	1746 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.055$
$T = 294$(2) K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -18\rightarrow19$
$T_{min} = 0.946$, $T_{max} = 0.965$	$k = -11\rightarrow10$
16642 measured reflections	$l = -26\rightarrow25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.057$	H-atom parameters constrained
$wR(F^2) = 0.179$	$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2 + 3.5628P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
3027 reflections	$\Delta\rho_{max} = 0.45$ e Å^{-3}
212 parameters	$\Delta\rho_{min} = -0.43$ 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}
Cl1	0.19174 (10)	0.54485 (17)	0.28229 (5)	0.1191 (6)
F1	0.07036 (18)	0.3181 (3)	0.39600 (12)	0.0933 (9)
F2	0.15906 (16)	0.2580 (3)	0.33078 (13)	0.0954 (9)
F3	0.04769 (16)	0.3457 (3)	0.30072 (12)	0.0953 (9)
O1	0.20727 (14)	0.5688 (2)	0.51113 (11)	0.0520 (6)
N1	0.29927 (15)	0.7368 (3)	0.53797 (12)	0.0448 (7)
H1	0.3173	0.8183	0.5277	0.054*
C1	0.1046 (2)	0.3562 (4)	0.34407 (18)	0.0616 (10)
C2	0.1395 (2)	0.5006 (4)	0.34741 (15)	0.0523 (9)
C3	0.1319 (2)	0.5825 (4)	0.39507 (15)	0.0459 (8)
H3	0.1067	0.5441	0.4293	0.055*
C4	0.1591 (2)	0.7286 (4)	0.40009 (15)	0.0487 (9)
H4	0.1720	0.7731	0.3609	0.058*
C5	0.1220 (2)	0.8267 (4)	0.44675 (16)	0.0512 (9)
C6	0.21059 (19)	0.7814 (3)	0.45415 (14)	0.0435 (8)
H6	0.2503	0.8540	0.4433	0.052*
C7	0.0588 (2)	0.7747 (4)	0.49090 (18)	0.0637 (11)
H7A	0.0560	0.8371	0.5253	0.096*
H7B	0.0731	0.6822	0.5047	0.096*
H7C	0.0068	0.7717	0.4709	0.096*
C8	0.1093 (3)	0.9766 (4)	0.4253 (2)	0.0781 (13)
H8A	0.0592	0.9829	0.4030	0.117*
H8B	0.1536	1.0038	0.3992	0.117*
H8C	0.1072	1.0381	0.4601	0.117*
C9	0.23767 (19)	0.6849 (4)	0.50305 (14)	0.0410 (8)
C10	0.33751 (19)	0.6729 (4)	0.58929 (15)	0.0456 (8)
C11	0.3313 (2)	0.5296 (4)	0.60035 (15)	0.0488 (9)
H11	0.3029	0.4738	0.5727	0.059*
C12	0.3659 (2)	0.4671 (4)	0.65121 (17)	0.0571 (10)
C13	0.4089 (3)	0.5517 (5)	0.6902 (2)	0.0796 (13)
H13	0.4325	0.5129	0.7250	0.096*
C14	0.4178 (3)	0.6923 (5)	0.6789 (2)	0.0810 (13)
H14	0.4488	0.7459	0.7058	0.097*

C15	0.3824 (2)	0.7582 (4)	0.62888 (17)	0.0587 (10)
C16	0.3928 (3)	0.9132 (4)	0.6193 (2)	0.0778 (13)
H16A	0.4129	0.9301	0.5787	0.117*
H16B	0.4308	0.9494	0.6486	0.117*
H16C	0.3413	0.9593	0.6243	0.117*
C17	0.3556 (3)	0.3115 (5)	0.6628 (2)	0.0791 (13)
H17A	0.3987	0.2790	0.6889	0.119*
H17B	0.3576	0.2618	0.6246	0.119*
H17C	0.3042	0.2950	0.6823	0.119*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1729 (15)	0.1255 (12)	0.0590 (7)	−0.0545 (10)	0.0390 (8)	−0.0235 (7)
F1	0.129 (2)	0.0727 (17)	0.0780 (17)	−0.0332 (16)	0.0157 (16)	−0.0098 (14)
F2	0.0903 (19)	0.0702 (17)	0.126 (2)	0.0183 (14)	−0.0112 (16)	−0.0312 (16)
F3	0.0857 (18)	0.105 (2)	0.0954 (18)	−0.0110 (15)	−0.0415 (15)	−0.0257 (15)
O1	0.0549 (15)	0.0447 (14)	0.0564 (15)	−0.0047 (11)	−0.0145 (11)	0.0057 (12)
N1	0.0439 (16)	0.0439 (16)	0.0465 (15)	−0.0038 (13)	−0.0052 (13)	−0.0002 (13)
C1	0.059 (2)	0.065 (3)	0.061 (2)	0.002 (2)	−0.009 (2)	−0.019 (2)
C2	0.050 (2)	0.064 (2)	0.043 (2)	−0.0020 (18)	−0.0063 (16)	−0.0035 (18)
C3	0.046 (2)	0.051 (2)	0.0409 (18)	0.0008 (16)	−0.0068 (15)	−0.0007 (16)
C4	0.053 (2)	0.052 (2)	0.0409 (18)	0.0003 (17)	−0.0049 (16)	0.0019 (16)
C5	0.052 (2)	0.048 (2)	0.054 (2)	0.0089 (17)	−0.0099 (18)	−0.0058 (17)
C6	0.0458 (19)	0.0408 (18)	0.0439 (18)	−0.0038 (15)	−0.0015 (15)	0.0024 (15)
C7	0.043 (2)	0.076 (3)	0.072 (3)	0.0070 (19)	−0.0031 (19)	−0.017 (2)
C8	0.085 (3)	0.053 (2)	0.096 (3)	0.017 (2)	−0.033 (3)	−0.001 (2)
C9	0.0412 (19)	0.041 (2)	0.0408 (17)	0.0035 (15)	0.0006 (15)	−0.0031 (15)
C10	0.0355 (18)	0.057 (2)	0.0437 (19)	0.0046 (16)	−0.0036 (15)	−0.0055 (17)
C11	0.0459 (19)	0.052 (2)	0.048 (2)	0.0040 (17)	−0.0074 (16)	−0.0024 (17)
C12	0.046 (2)	0.070 (3)	0.055 (2)	0.0105 (18)	−0.0089 (18)	0.003 (2)
C13	0.077 (3)	0.094 (4)	0.068 (3)	0.013 (3)	−0.032 (2)	0.008 (3)
C14	0.078 (3)	0.086 (3)	0.079 (3)	0.003 (3)	−0.040 (3)	−0.014 (3)
C15	0.052 (2)	0.063 (3)	0.061 (2)	0.0004 (19)	−0.0147 (19)	−0.008 (2)
C16	0.068 (3)	0.068 (3)	0.098 (3)	−0.009 (2)	−0.032 (2)	−0.014 (2)
C17	0.084 (3)	0.076 (3)	0.077 (3)	0.013 (2)	−0.016 (2)	0.019 (2)

Geometric parameters (Å, °)

Cl1—C2	1.715 (4)	C7—H7C	0.9600
F1—C1	1.318 (4)	C8—H8A	0.9600
F2—C1	1.326 (4)	C8—H8B	0.9600
F3—C1	1.336 (4)	C8—H8C	0.9600
O1—C9	1.226 (4)	C10—C11	1.389 (5)
N1—C9	1.361 (4)	C10—C15	1.398 (5)
N1—C10	1.422 (4)	C11—C12	1.383 (5)
N1—H1	0.8600	C11—H11	0.9300
C1—C2	1.490 (5)	C12—C13	1.370 (6)
C2—C3	1.307 (4)	C12—C17	1.511 (6)

C3—C4	1.464 (5)	C13—C14	1.369 (6)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.511 (5)	C14—C15	1.388 (5)
C4—C6	1.538 (4)	C14—H14	0.9300
C4—H4	0.9800	C15—C16	1.499 (6)
C5—C7	1.502 (5)	C16—H16A	0.9600
C5—C8	1.516 (5)	C16—H16B	0.9600
C5—C6	1.528 (4)	C16—H16C	0.9600
C6—C9	1.478 (4)	C17—H17A	0.9600
C6—H6	0.9800	C17—H17B	0.9600
C7—H7A	0.9600	C17—H17C	0.9600
C7—H7B	0.9600		
C9—N1—C10	128.0 (3)	C5—C8—H8A	109.5
C9—N1—H1	116.0	C5—C8—H8B	109.5
C10—N1—H1	116.0	H8A—C8—H8B	109.5
F1—C1—F2	106.5 (4)	C5—C8—H8C	109.5
F1—C1—F3	106.9 (3)	H8A—C8—H8C	109.5
F2—C1—F3	105.4 (3)	H8B—C8—H8C	109.5
F1—C1—C2	112.1 (3)	O1—C9—N1	123.4 (3)
F2—C1—C2	113.6 (3)	O1—C9—C6	122.8 (3)
F3—C1—C2	111.9 (3)	N1—C9—C6	113.9 (3)
C3—C2—C1	123.5 (3)	C11—C10—C15	120.1 (3)
C3—C2—Cl1	124.2 (3)	C11—C10—N1	121.6 (3)
C1—C2—Cl1	112.2 (3)	C15—C10—N1	118.3 (3)
C2—C3—C4	126.6 (3)	C12—C11—C10	122.1 (3)
C2—C3—H3	116.7	C12—C11—H11	118.9
C4—C3—H3	116.7	C10—C11—H11	118.9
C3—C4—C5	120.9 (3)	C13—C12—C11	117.4 (4)
C3—C4—C6	122.4 (3)	C13—C12—C17	121.9 (4)
C5—C4—C6	60.1 (2)	C11—C12—C17	120.7 (4)
C3—C4—H4	114.3	C14—C13—C12	121.1 (4)
C5—C4—H4	114.3	C14—C13—H13	119.4
C6—C4—H4	114.3	C12—C13—H13	119.4
C7—C5—C4	120.6 (3)	C13—C14—C15	122.7 (4)
C7—C5—C8	114.4 (3)	C13—C14—H14	118.7
C4—C5—C8	115.3 (3)	C15—C14—H14	118.7
C7—C5—C6	120.0 (3)	C14—C15—C10	116.5 (4)
C4—C5—C6	60.8 (2)	C14—C15—C16	120.5 (4)
C8—C5—C6	115.4 (3)	C10—C15—C16	123.0 (3)
C9—C6—C5	122.6 (3)	C15—C16—H16A	109.5
C9—C6—C4	121.2 (3)	C15—C16—H16B	109.5
C5—C6—C4	59.1 (2)	H16A—C16—H16B	109.5
C9—C6—H6	114.3	C15—C16—H16C	109.5
C5—C6—H6	114.3	H16A—C16—H16C	109.5
C4—C6—H6	114.3	H16B—C16—H16C	109.5
C5—C7—H7A	109.5	C12—C17—H17A	109.5
C5—C7—H7B	109.5	C12—C17—H17B	109.5
H7A—C7—H7B	109.5	H17A—C17—H17B	109.5
C5—C7—H7C	109.5	C12—C17—H17C	109.5

H7A—C7—H7C	109.5	H17A—C17—H17C	109.5
H7B—C7—H7C	109.5	H17B—C17—H17C	109.5
F1—C1—C2—C3	−4.2 (5)	C3—C4—C6—C5	−109.7 (4)
F2—C1—C2—C3	−125.0 (4)	C10—N1—C9—O1	−2.0 (5)
F3—C1—C2—C3	115.8 (4)	C10—N1—C9—C6	178.2 (3)
F1—C1—C2—Cl1	174.7 (3)	C5—C6—C9—O1	54.5 (5)
F2—C1—C2—Cl1	54.0 (4)	C4—C6—C9—O1	−16.5 (5)
F3—C1—C2—Cl1	−65.2 (4)	C5—C6—C9—N1	−125.7 (3)
C1—C2—C3—C4	−175.2 (3)	C4—C6—C9—N1	163.4 (3)
Cl1—C2—C3—C4	6.0 (5)	C9—N1—C10—C11	18.3 (5)
C2—C3—C4—C5	158.0 (3)	C9—N1—C10—C15	−161.9 (3)
C2—C3—C4—C6	−129.9 (4)	C15—C10—C11—C12	2.5 (5)
C3—C4—C5—C7	2.6 (5)	N1—C10—C11—C12	−177.8 (3)
C6—C4—C5—C7	−109.5 (3)	C10—C11—C12—C13	−1.6 (6)
C3—C4—C5—C8	−141.7 (3)	C10—C11—C12—C17	178.1 (3)
C6—C4—C5—C8	106.2 (3)	C11—C12—C13—C14	−0.5 (6)
C3—C4—C5—C6	112.1 (3)	C17—C12—C13—C14	179.8 (4)
C7—C5—C6—C9	1.1 (5)	C12—C13—C14—C15	1.9 (7)
C4—C5—C6—C9	−109.5 (3)	C13—C14—C15—C10	−1.0 (7)
C8—C5—C6—C9	144.4 (3)	C13—C14—C15—C16	178.8 (4)
C7—C5—C6—C4	110.6 (4)	C11—C10—C15—C14	−1.2 (5)
C8—C5—C6—C4	−106.1 (4)	N1—C10—C15—C14	179.1 (3)
C3—C4—C6—C9	2.1 (5)	C11—C10—C15—C16	179.0 (4)
C5—C4—C6—C9	111.8 (3)	N1—C10—C15—C16	−0.7 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.44	3.214 (4)	149

Symmetry codes: (i) −*x*+1/2, *y*+1/2, *z*.

Fig. 1

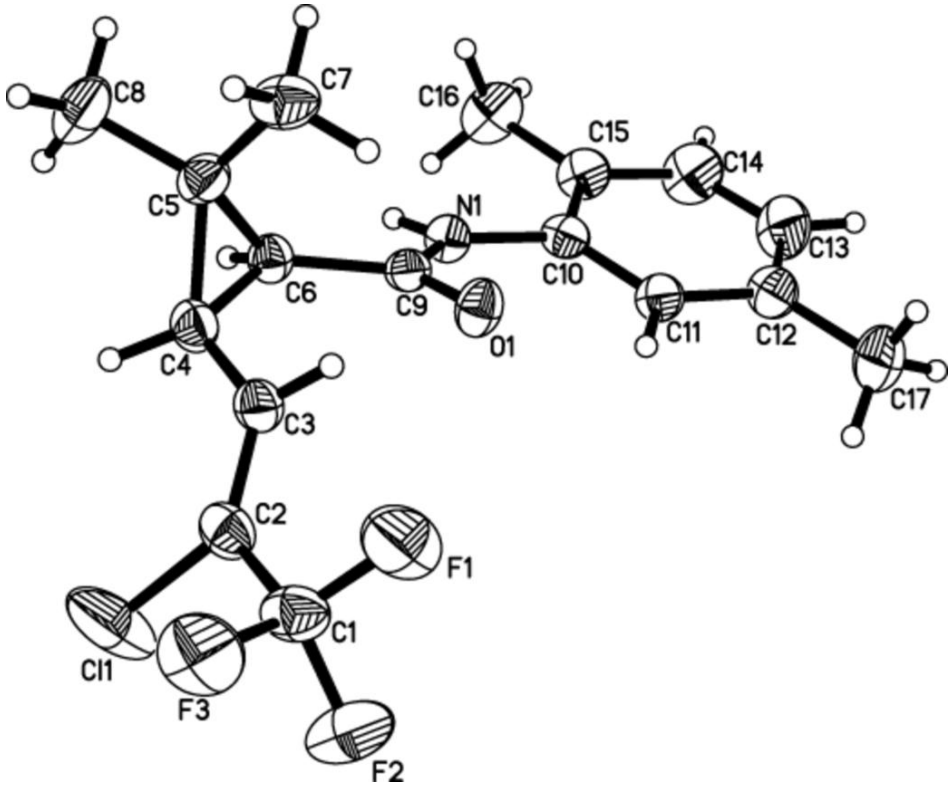
C8
C7
C16
C15
C14
C5
N1
C13
C10
C6
C9
C11
C12
O1
C4
C17
C3
C2
F1
C1
Cl1
F3
F2

Fig. 2