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(*E*)-*N*-(2-Bromophenyl)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxamide

Dong-Qing Liu and Fan-Yong Yan*

School of Materials Science and Chemical Engineering, Tianjin Polytechnic University, Tianjin 300160, People's Republic of China
Correspondence e-mail: yfany@163.com

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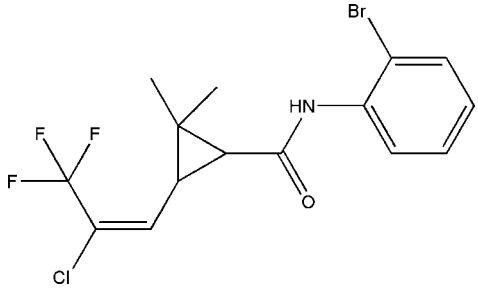
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.011 Å; R factor = 0.054; wR factor = 0.205; data-to-parameter ratio = 14.9.

In the title compound, $C_{15}H_{14}BrClF_3NO$, the benzene and cyclopropane rings make a dihedral angle of 76.3 (3)°. The amide and methine H atoms are both linked to the amide O atom in an adjacent molecule by a pair of intermolecular hydrogen bonds, N—H···O···H—C, leading to the formation of chains along the *b* axis.

Related literature

For related literature, see: Punja (1981); Zhang (2005).



Experimental

Crystal data

$C_{15}H_{14}BrClF_3NO$
M_r = 396.63
Orthorhombic, *Pbca*
a = 18.701 (3) Å
b = 9.4180 (16) Å
c = 19.356 (3) Å
V = 3409.2 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.60 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.575, T_{max} = 0.652
16318 measured reflections
3004 independent reflections
1493 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.205
S = 1.04
3004 reflections
201 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 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	2.27	3.110 (7)	164
C6—H6···O1^{i}	0.98	2.48	2.39	137

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, 1990); 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: BV2066).

References

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Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
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  doi:10.1107/S1600536807045916

supplementary materials

Acta Cryst. (2007). E**63**, o4202 [**doi:10.1107/S1600536807045916**]

(*E*)-*N*-(2-Bromophenyl)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxamide

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

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). Bromine containing pesticides have the advantage of low toxicity, high activity and low residues (Zhang 2005). The present structure contains two active parts and thus might be expected to show some insecticide activity. The X-ray crystal structure analysis was undertaken in order to study the stereochemistry and crystal packing of the title compound, (I) In this paper, the title compound, (*E*)—*N*-(2-bromophenyl)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2, 2-dimethylcyclopropanecarboxamide, (I), was synthesized and its structure is illustrated in Fig. 1. The dihedral angle between the benzene moiety and the cyclopropane group is 76.3 (3)°. The amide hydrogen and methine hydrogen link with the same amide oxygen in an adjacent molecule *via* an intermolecular N—H···O···H—C hydrogen bond. The packing can be described as a dimeric arrangement of molecules linked through N—H···O···H—C hydrogen bonds as shown in Fig. 2 and Table 1.

Experimental

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 4 h. $SOCl_2$ was removed by rotoevaporation. The crude product could be directly disolved in anhydrous toluene, already mixed with 2-bromoaniline (0.71 g, 4.1 mmol). Triethylamine was aded droppwise to the system to prevent white fumes coming out. After 12 h stirring at room temperature, the reaction mixture was treated with hexane. Recrystallization of the off-white product from methanol and a small amount of water (50:1) overnight at ambient temperature gave colorless single crystals of (*E*)—*N*-(2-bromophenyl)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2, 2-dimethylcyclopropanecarboxamide, suitable for X-ray analysis.

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 refined freely.

Figures

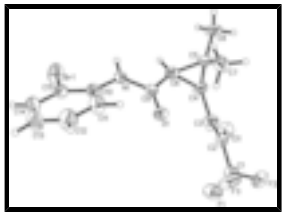

Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms are drawn as spheres of arbitrary radius.

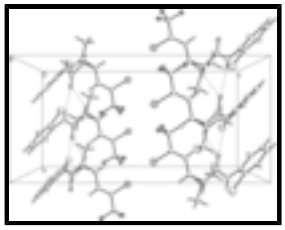

Fig. 2. The crystal structure of (I), viewed along *a* axis

(*E*)-*N*-(2-Bromophenyl)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)- 2,2-dimethylcyclopropanecarboxamide

Crystal data

$C_{15}H_{14}BrClF_3NO$	$D_x = 1.546$ Mg m^{-3}
$M_r = 396.63$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 2447 reflections
$a = 18.701$ (3) Å	$\theta = 2.4$–19.3º
$b = 9.4180$ (16) Å	$\mu = 2.60$ mm^{-1}
$c = 19.356$ (3) Å	$T = 294$ (2) K
$V = 3409.2$ (10) Å^3	Prism, colourless
$Z = 8$	0.24 × 0.22 × 0.18 mm
$F_{000} = 1584$	

Data collection

Bruker SMART CCD area-detector diffractometer	3004 independent reflections
Radiation source: fine-focus sealed tube	1493 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.075$
$T = 294$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -19 \rightarrow 22$
$T_{min} = 0.575$, $T_{max} = 0.652$	$k = -9 \rightarrow 11$
16318 measured reflections	$l = -17 \rightarrow 23$

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-atom parameters constrained
$wR(F^2) = 0.205$	$w = 1/[\sigma^2(F_o^2) + (0.0915P)^2 + 5.7059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
3004 reflections	$\Delta\rho_{max} = 0.46$ e Å^{-3}
201 parameters	$\Delta\rho_{min} = -0.71$ e Å^{-3}

6 restraints
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}
Br1	0.05485 (5)	−0.07635 (11)	0.13989 (5)	0.0912 (4)
Cl1	0.34565 (12)	0.1240 (3)	0.44233 (11)	0.0843 (7)
F1	0.3114 (3)	0.4319 (6)	0.4070 (3)	0.1145 (19)
F2	0.4226 (3)	0.4018 (5)	0.4192 (3)	0.1077 (18)
F3	0.3800 (3)	0.4432 (4)	0.3209 (3)	0.1038 (18)
O1	0.2896 (2)	0.1847 (4)	0.1711 (3)	0.0588 (13)
N1	0.2144 (3)	0.0086 (5)	0.1403 (3)	0.0464 (13)
H1	0.2044	−0.0797	0.1461	0.056*
C1	0.3690 (4)	0.3721 (9)	0.3778 (5)	0.070 (2)
C2	0.3568 (3)	0.2181 (7)	0.3663 (4)	0.0515 (17)
C3	0.3552 (4)	0.1605 (7)	0.3053 (4)	0.0552 (18)
H3	0.3631	0.2197	0.2676	0.066*
C4	0.3420 (4)	0.0089 (7)	0.2909 (4)	0.061 (2)
H4	0.3343	−0.0487	0.3324	0.073*
C5	0.3773 (4)	−0.0685 (7)	0.2336 (4)	0.070 (2)
C6	0.2990 (4)	−0.0395 (6)	0.2287 (4)	0.0567 (19)
H6	0.2687	−0.1226	0.2374	0.068*
C7	0.4275 (4)	0.0051 (12)	0.1865 (5)	0.099 (3)
H7A	0.4270	−0.0407	0.1422	0.148*
H7B	0.4749	0.0013	0.2054	0.148*
H7C	0.4132	0.1024	0.1813	0.148*
C8	0.3977 (5)	−0.2211 (8)	0.2502 (6)	0.120 (4)
H8A	0.4153	−0.2666	0.2092	0.180*
H8B	0.3565	−0.2712	0.2669	0.180*
H8C	0.4343	−0.2217	0.2850	0.180*
C9	0.2686 (3)	0.0626 (6)	0.1782 (3)	0.0464 (15)
C10	0.1729 (3)	0.0846 (7)	0.0919 (3)	0.0479 (16)
C11	0.2031 (4)	0.1875 (8)	0.0496 (4)	0.069 (2)
H11	0.2520	0.2049	0.0519	0.083*
C12	0.1613 (5)	0.2639 (9)	0.0045 (5)	0.088 (3)

H12	0.1826	0.3307	−0.0243	0.106*
C13	0.0887 (5)	0.2433 (10)	0.0011 (5)	0.091 (3)
H13	0.0607	0.2990	−0.0279	0.109*
C14	0.0579 (4)	0.1393 (9)	0.0411 (4)	0.071 (2)
H14	0.0092	0.1212	0.0379	0.086*
C15	0.0995 (3)	0.0624 (7)	0.0859 (3)	0.0514 (17)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0698 (6)	0.1091 (8)	0.0947 (8)	−0.0272 (5)	−0.0041 (5)	0.0263 (5)
Cl1	0.0942 (16)	0.0951 (16)	0.0636 (13)	−0.0040 (13)	0.0020 (11)	0.0041 (11)
F1	0.110 (3)	0.095 (3)	0.139 (4)	0.022 (3)	0.014 (3)	−0.040 (3)
F2	0.103 (4)	0.089 (4)	0.130 (5)	−0.012 (3)	−0.040 (4)	−0.035 (3)
F3	0.161 (5)	0.042 (2)	0.109 (4)	−0.022 (3)	0.000 (4)	−0.007 (3)
O1	0.064 (3)	0.034 (3)	0.078 (3)	−0.004 (2)	−0.016 (3)	0.003 (2)
N1	0.048 (3)	0.035 (3)	0.056 (3)	0.001 (2)	−0.005 (3)	0.003 (3)
C1	0.064 (5)	0.060 (5)	0.086 (6)	0.005 (4)	0.000 (5)	−0.021 (5)
C2	0.046 (4)	0.049 (4)	0.060 (5)	0.000 (3)	0.000 (3)	−0.002 (3)
C3	0.073 (5)	0.041 (4)	0.052 (4)	0.004 (3)	−0.004 (4)	0.002 (3)
C4	0.086 (5)	0.032 (4)	0.065 (5)	−0.002 (4)	−0.027 (4)	−0.002 (3)
C5	0.080 (6)	0.043 (4)	0.086 (6)	0.018 (4)	−0.031 (5)	−0.022 (4)
C6	0.066 (5)	0.031 (3)	0.073 (5)	−0.002 (3)	−0.021 (4)	0.002 (3)
C7	0.068 (6)	0.118 (8)	0.109 (8)	0.032 (6)	−0.015 (6)	−0.049 (7)
C8	0.148 (9)	0.046 (5)	0.166 (10)	0.046 (5)	−0.098 (8)	−0.044 (6)
C9	0.044 (4)	0.036 (4)	0.059 (4)	0.003 (3)	0.002 (3)	−0.005 (3)
C10	0.053 (4)	0.045 (4)	0.046 (4)	0.002 (3)	−0.003 (3)	0.001 (3)
C11	0.062 (5)	0.076 (5)	0.069 (5)	−0.009 (4)	−0.004 (4)	0.025 (4)
C12	0.103 (7)	0.077 (6)	0.085 (6)	−0.012 (5)	−0.020 (5)	0.038 (5)
C13	0.089 (6)	0.081 (6)	0.102 (7)	0.006 (6)	−0.033 (6)	0.026 (5)
C14	0.062 (5)	0.080 (5)	0.073 (5)	0.007 (4)	−0.013 (4)	0.001 (5)
C15	0.048 (4)	0.054 (4)	0.053 (4)	0.002 (3)	0.001 (3)	−0.004 (3)

Geometric parameters (Å, °)

Br1—C15	1.870 (7)	C6—C9	1.484 (9)
Cl1—C2	1.730 (7)	C6—H6	0.9800
F1—C1	1.341 (9)	C7—H7A	0.9600
F2—C1	1.313 (9)	C7—H7B	0.9600
F3—C1	1.306 (10)	C7—H7C	0.9600
O1—C9	1.223 (7)	C8—H8A	0.9600
N1—C9	1.351 (8)	C8—H8B	0.9600
N1—C10	1.412 (8)	C8—H8C	0.9600
N1—H1	0.8600	C10—C11	1.389 (9)
C1—C2	1.485 (10)	C10—C15	1.393 (9)
C2—C3	1.300 (9)	C11—C12	1.375 (10)
C3—C4	1.475 (9)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.373 (11)
C4—C5	1.482 (11)	C12—H12	0.9300

C4—C6	1.518 (9)	C13—C14	1.375 (11)
C4—H4	0.9800	C13—H13	0.9300
C5—C7	1.482 (13)	C14—C15	1.370 (10)
C5—C6	1.492 (10)	C14—H14	0.9300
C5—C8	1.521 (10)		
C9—N1—C10	125.7 (5)	C5—C7—H7A	109.5
C9—N1—H1	117.2	C5—C7—H7B	109.5
C10—N1—H1	117.2	H7A—C7—H7B	109.5
F3—C1—F2	106.6 (7)	C5—C7—H7C	109.5
F3—C1—F1	105.5 (7)	H7A—C7—H7C	109.5
F2—C1—F1	105.5 (7)	H7B—C7—H7C	109.5
F3—C1—C2	113.4 (7)	C5—C8—H8A	109.5
F2—C1—C2	114.6 (7)	C5—C8—H8B	109.5
F1—C1—C2	110.5 (7)	H8A—C8—H8B	109.5
C3—C2—C1	123.2 (7)	C5—C8—H8C	109.5
C3—C2—Cl1	123.8 (5)	H8A—C8—H8C	109.5
C1—C2—Cl1	113.0 (6)	H8B—C8—H8C	109.5
C2—C3—C4	125.4 (7)	O1—C9—N1	122.3 (6)
C2—C3—H3	117.3	O1—C9—C6	124.1 (6)
C4—C3—H3	117.3	N1—C9—C6	113.6 (5)
C3—C4—C5	122.9 (7)	C11—C10—C15	117.2 (6)
C3—C4—C6	122.0 (6)	C11—C10—N1	121.3 (6)
C5—C4—C6	59.6 (5)	C15—C10—N1	121.4 (6)
C3—C4—H4	113.9	C12—C11—C10	120.5 (7)
C5—C4—H4	113.9	C12—C11—H11	119.8
C6—C4—H4	113.9	C10—C11—H11	119.8
C7—C5—C4	120.8 (6)	C13—C12—C11	121.3 (8)
C7—C5—C6	119.8 (7)	C13—C12—H12	119.4
C4—C5—C6	61.4 (5)	C11—C12—H12	119.4
C7—C5—C8	114.4 (8)	C12—C13—C14	119.2 (8)
C4—C5—C8	114.8 (8)	C12—C13—H13	120.4
C6—C5—C8	115.7 (7)	C14—C13—H13	120.4
C9—C6—C5	122.4 (6)	C15—C14—C13	119.7 (7)
C9—C6—C4	122.0 (5)	C15—C14—H14	120.2
C5—C6—C4	59.0 (5)	C13—C14—H14	120.2
C9—C6—H6	114.2	C14—C15—C10	122.1 (7)
C5—C6—H6	114.2	C14—C15—Br1	118.0 (5)
C4—C6—H6	114.2	C10—C15—Br1	119.9 (5)
F3—C1—C2—C3	−1.8 (11)	C5—C4—C6—C9	−111.2 (8)
F2—C1—C2—C3	−124.6 (8)	C3—C4—C6—C5	112.1 (8)
F1—C1—C2—C3	116.4 (9)	C10—N1—C9—O1	−4.1 (10)
F3—C1—C2—Cl1	177.7 (5)	C10—N1—C9—C6	175.6 (6)
F2—C1—C2—Cl1	54.9 (9)	C5—C6—C9—O1	−51.3 (10)
F1—C1—C2—Cl1	−64.1 (8)	C4—C6—C9—O1	19.8 (11)
C1—C2—C3—C4	−178.8 (7)	C5—C6—C9—N1	129.0 (6)
Cl1—C2—C3—C4	1.7 (11)	C4—C6—C9—N1	−159.9 (6)
C2—C3—C4—C5	−144.9 (7)	C9—N1—C10—C11	37.4 (10)
C2—C3—C4—C6	142.9 (8)	C9—N1—C10—C15	−140.7 (7)

C3—C4—C5—C7	−1.1 (11)	C15—C10—C11—C12	0.5 (11)
C6—C4—C5—C7	109.5 (8)	N1—C10—C11—C12	−177.7 (7)
C3—C4—C5—C6	−110.7 (7)	C10—C11—C12—C13	1.6 (14)
C3—C4—C5—C8	142.4 (7)	C11—C12—C13—C14	−3.4 (15)
C6—C4—C5—C8	−107.0 (7)	C12—C13—C14—C15	2.9 (14)
C7—C5—C6—C9	−0.6 (10)	C13—C14—C15—C10	−0.8 (12)
C4—C5—C6—C9	110.5 (7)	C13—C14—C15—Br1	179.3 (7)
C8—C5—C6—C9	−144.0 (8)	C11—C10—C15—C14	−0.9 (10)
C7—C5—C6—C4	−111.1 (8)	N1—C10—C15—C14	177.3 (6)
C8—C5—C6—C4	105.5 (8)	C11—C10—C15—Br1	179.0 (5)
C3—C4—C6—C9	0.9 (11)	N1—C10—C15—Br1	−2.8 (9)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.27	3.110 (7)	164
C6—H6···01^{i}	0.98	2.48	2.39	137

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

Fig. 1

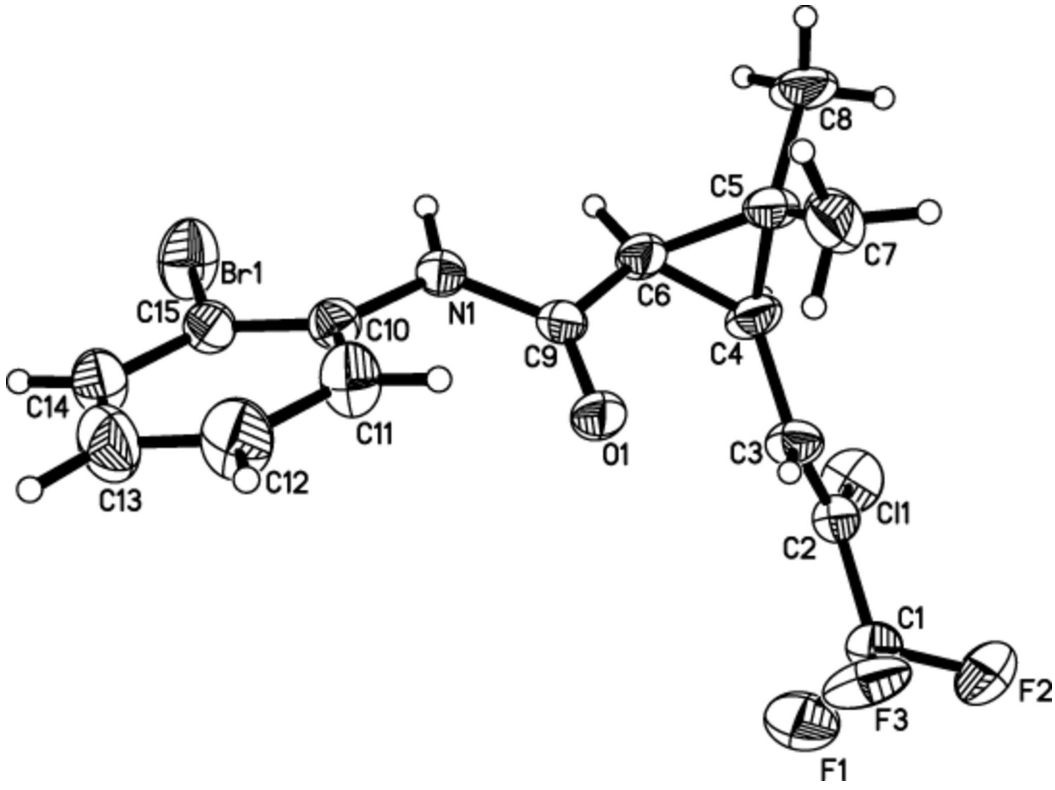
C8
C5
C7
Br1
C15
C10
N1
C6
C9
C4
C14
C11
O1
C3
C13
C12
Cl1
C2
C1
F2
F3
F1

Fig. 2