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2-Amino-1*H*-benzoimidazol-3-ium 4,4,4-trifluoro-1,3-dioxo-1-phenylbutan-2-ide

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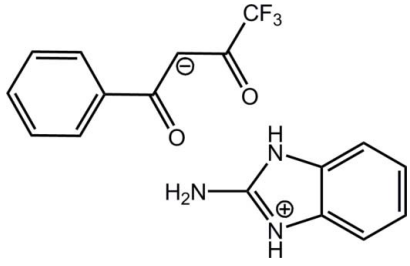
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.107; data-to-parameter ratio = 11.9.

In the title compound, $C_7H_8N_3^+\cdot C_{10}H_6F_3O_2^-$, 1*H*-benzoimidazol-2-amine system adopts a planar conformation with an r.m.s. deviation of 0.0174 Å. The cation and anion in the asymmetric unit are linked by N—H···O hydrogen bonds. There are also additional intermolecular N—H···O hydrogen bonds and π–π stacking interactions between the phenyl rings of neighbouring anions with centroid–centroid distances of 4.0976 (13) Å.

Related literature

For details of the bioactivity of organofluorine compounds, see: Hermann *et al.* (2003); Ulrich (2004).



Experimental

Crystal data

$C_7H_8N_3^+\cdot C_{10}H_6F_3O_2^-$
M_r = 349.31
Orthorhombic, *Pbca*
a = 16.755 (3) Å
b = 10.552 (2) Å
c = 18.497 (4) Å
V = 3270.0 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 113 (2) K
0.12 × 0.10 × 0.06 mm

Data collection

Rigaku Saturn CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2002)
T_{min} = 0.986, T_{max} = 0.993
31132 measured reflections
2881 independent reflections
2609 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.06
2881 reflections
243 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.877 (9)	2.004 (11)	2.7803 (18)	146.8 (16)
N3—H3A···O1^{i}	0.895 (9)	1.949 (13)	2.7651 (17)	150.9 (19)
N3—H3A···O2^{i}	0.895 (9)	2.363 (18)	2.9912 (18)	127.3 (17)
N1—H1B···O1	0.879 (9)	2.030 (10)	2.8662 (18)	158.4 (16)
N2—H2A···O2	0.905 (10)	1.862 (11)	2.7360 (18)	161.8 (19)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: SJ2549).

References

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supplementary materials

Acta Cryst. (2008). E**64**, o2460 [**doi:10.1107/S1600536808037483**]

2-Amino-1*H*-benzoimidazol-3-ium 4,4,4-trifluoro-1,3-dioxo-1-phenylbutan-2-ide

G.-C. Li, F.-L. Yang and C.-S. Yao

Comment

Compounds that contain fluorine exhibit particular bioactivity, for example, flumioxazin is a widely used herbicide (Hermann *et al.*, 2003; Ulrich,2004). This led us to pay much attention to the synthesis and structure of similar fluoro-compounds. During the synthesis of trifluoromethylated heterocyclic compounds, an intermediate, the title compound,(I), Fig.1, was isolated and we report its crystal structure here.

The 1*H*-benzo[*d*]imizol-2-amine cation adopts a planar conformation with an rms deviation of 0.0174 for the fitted atoms. The phenyl ring is almost perpendicular to the fused heterocyclic rings, with a dihedral angle of 81.84 (4)° between them. The cation and anion in the asymmetric unit are linked by N1—H1B···O1 and N2—H2A···O2 hydrogen bonds.

The crystal packing is further stabilized by additional intermolecular N—H···O hydrogen bonds (Table 1, Fig. 2) and intermolecular π···π stacking interactions between the C5-C10 phenyl rings (symmetry code: 2-x, -y, -z) of neighbouring anions with centroid-to-centroid distances, plane-plane distances and displacement distances of 4.0976 (13), 3.786 and 1.565Å respectively.

Experimental

The title compound was synthesized by the reaction of 4,4,4-trifluoro-1- phenylbutane-1,3-dione (1 mmol) and 1*H*-benzo[*d*]imidazol-2-amine (1 mmol) in refluxing ethanol (20 mL) for a certain time (monitored by TLC). Cooling, the reaction mixture slowly to room temperature, gave single crystals suitable for X-ray diffraction.

Refinement

Hydrogen atoms bound to nitrogen atoms were located in a difference Fourier map and were refined with d(N—H) restrained to 0.90 (1) Å and d(H1A···H1B) restrained to 1.50 (1) Å. Other H atoms were placed in calculated positions, d(C—H) = 0.93 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

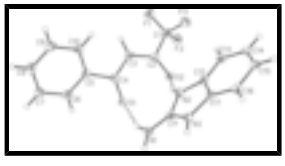

Fig. 1. The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are drawn as dashed lines.

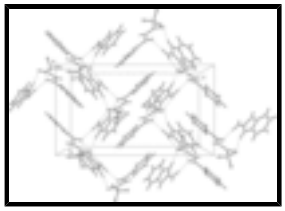

Fig. 2. The packing diagram of the title compound. Intermolecular hydrogen bonds are shown as dashed lines.

2-Amino-1*H*-benzoimidazol-3-ium 4,4,4-trifluoro-1,3-dioxo-1-phenylbutan-2-ide

Crystal data

$C_7H_8N_3^+ \cdot C_{10}H_6F_3O_2^-$	F_{000} = 1440
M_r = 349.31	D_x = 1.419 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 6974 reflections
a = 16.755 (3) Å	θ = 2.2–27.9º
b = 10.552 (2) Å	μ = 0.12 mm^{-1}
c = 18.497 (4) Å	*T* = 113 (2) K
V = 3270.0 (11) Å^3	Block, yellow
Z = 8	0.12 × 0.10 × 0.06 mm

Data collection

Rigaku Saturn CCD diffractometer	2881 independent reflections
Radiation source: rotating anode	2609 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.050
T = 113(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2002)	*h* = −19→19
T_{min} = 0.986, T_{max} = 0.993	*k* = −12→12
31132 measured reflections	*l* = −22→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.107	$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 0.895P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
2881 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
243 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}

5 restraints

Primary atom site location: structure-invariant direct methods

Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0073 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
F1	0.74257 (7)	0.54639 (12)	0.05657 (5)	0.0552 (3)
F2	0.77552 (6)	0.66729 (9)	0.14532 (6)	0.0473 (3)
F3	0.71669 (6)	0.49082 (11)	0.16580 (6)	0.0467 (3)
O1	1.01117 (6)	0.37243 (10)	0.11875 (6)	0.0301 (3)
O2	0.88568 (6)	0.49842 (10)	0.19269 (6)	0.0303 (3)
N1	1.03832 (8)	0.19158 (15)	0.23172 (8)	0.0379 (4)
N2	0.90662 (8)	0.26462 (13)	0.25377 (7)	0.0298 (3)
N3	0.94668 (8)	0.09546 (13)	0.31266 (7)	0.0295 (3)
C1	0.77211 (10)	0.54628 (16)	0.12397 (9)	0.0335 (4)
C2	0.85307 (9)	0.48052 (14)	0.13136 (8)	0.0277 (3)
C3	0.88043 (9)	0.40780 (15)	0.07420 (8)	0.0290 (4)
H3	0.8467	0.3955	0.0349	0.035*
C4	0.95774 (9)	0.35055 (14)	0.07232 (8)	0.0264 (3)
C5	0.97828 (9)	0.26266 (14)	0.01141 (8)	0.0263 (3)
C6	1.05759 (10)	0.25313 (16)	−0.01066 (9)	0.0324 (4)
H6	1.0962	0.3030	0.0116	0.039*
C7	1.07968 (10)	0.17084 (17)	−0.06508 (10)	0.0378 (4)
H7	1.1327	0.1665	−0.0797	0.045*
C8	1.02270 (11)	0.09443 (16)	−0.09797 (9)	0.0368 (4)
H8	1.0376	0.0379	−0.1341	0.044*
C9	0.94389 (10)	0.10278 (15)	−0.07679 (9)	0.0346 (4)
H9	0.9057	0.0518	−0.0989	0.042*
C10	0.92111 (10)	0.18696 (15)	−0.02263 (8)	0.0308 (4)
H10	0.8678	0.1928	−0.0091	0.037*
C11	0.96806 (9)	0.18451 (15)	0.26443 (8)	0.0296 (4)
C12	0.84329 (9)	0.22790 (15)	0.29854 (8)	0.0275 (4)
C13	0.76879 (9)	0.28105 (17)	0.31021 (8)	0.0321 (4)
H13	0.7518	0.3527	0.2853	0.038*
C14	0.72078 (10)	0.22158 (17)	0.36124 (9)	0.0349 (4)

H14	0.6705	0.2548	0.3710	0.042*
C15	0.74579 (10)	0.11347 (17)	0.39833 (9)	0.0339 (4)
H15	0.7118	0.0763	0.4319	0.041*
C16	0.82038 (10)	0.06027 (15)	0.38614 (8)	0.0309 (4)
H16	0.8371	−0.0120	0.4106	0.037*
C17	0.86862 (9)	0.11999 (14)	0.33575 (8)	0.0273 (3)
H1A	1.0783 (9)	0.1439 (16)	0.2457 (10)	0.046 (5)*
H1B	1.0438 (10)	0.2461 (14)	0.1961 (8)	0.043 (5)*
H2A	0.9093 (12)	0.3377 (13)	0.2283 (10)	0.051 (6)*
H3A	0.9769 (11)	0.0317 (15)	0.3290 (11)	0.051 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0509 (7)	0.0792 (8)	0.0356 (6)	0.0313 (6)	−0.0129 (5)	−0.0053 (6)
F2	0.0462 (6)	0.0331 (6)	0.0628 (7)	0.0093 (4)	0.0013 (5)	−0.0034 (5)
F3	0.0293 (5)	0.0561 (7)	0.0546 (7)	−0.0022 (5)	0.0024 (5)	0.0043 (5)
O1	0.0292 (6)	0.0319 (6)	0.0291 (6)	−0.0017 (5)	−0.0052 (5)	−0.0008 (5)
O2	0.0307 (6)	0.0322 (6)	0.0279 (6)	−0.0019 (5)	−0.0042 (4)	−0.0015 (5)
N1	0.0316 (8)	0.0461 (9)	0.0358 (8)	0.0102 (7)	0.0039 (6)	0.0122 (7)
N2	0.0306 (7)	0.0313 (7)	0.0274 (7)	0.0048 (6)	−0.0006 (5)	0.0055 (6)
N3	0.0298 (7)	0.0304 (7)	0.0284 (7)	0.0056 (6)	−0.0008 (5)	0.0029 (6)
C1	0.0353 (9)	0.0355 (9)	0.0296 (8)	0.0026 (7)	−0.0016 (7)	0.0005 (7)
C2	0.0275 (8)	0.0253 (8)	0.0301 (8)	−0.0030 (6)	−0.0033 (6)	0.0048 (6)
C3	0.0295 (8)	0.0312 (8)	0.0263 (8)	0.0009 (6)	−0.0056 (6)	0.0006 (6)
C4	0.0296 (8)	0.0239 (8)	0.0256 (7)	−0.0038 (6)	−0.0027 (6)	0.0053 (6)
C5	0.0291 (8)	0.0251 (7)	0.0249 (7)	−0.0002 (6)	−0.0030 (6)	0.0044 (6)
C6	0.0285 (8)	0.0337 (9)	0.0350 (9)	−0.0024 (7)	−0.0036 (7)	−0.0011 (7)
C7	0.0309 (9)	0.0418 (10)	0.0407 (9)	0.0010 (7)	0.0045 (7)	−0.0035 (8)
C8	0.0452 (10)	0.0324 (9)	0.0327 (9)	0.0025 (7)	0.0014 (7)	−0.0036 (7)
C9	0.0391 (9)	0.0286 (8)	0.0360 (9)	−0.0057 (7)	−0.0058 (7)	−0.0025 (7)
C10	0.0287 (8)	0.0306 (8)	0.0330 (8)	−0.0030 (6)	−0.0021 (6)	0.0020 (7)
C11	0.0301 (9)	0.0336 (8)	0.0250 (8)	0.0036 (7)	−0.0028 (6)	0.0009 (6)
C12	0.0293 (8)	0.0305 (8)	0.0227 (7)	−0.0001 (6)	−0.0027 (6)	−0.0021 (6)
C13	0.0300 (9)	0.0369 (9)	0.0292 (8)	0.0055 (7)	−0.0047 (6)	−0.0011 (7)
C14	0.0260 (8)	0.0453 (10)	0.0332 (9)	0.0007 (7)	−0.0029 (6)	−0.0045 (8)
C15	0.0300 (8)	0.0421 (10)	0.0298 (8)	−0.0074 (7)	−0.0005 (7)	−0.0015 (7)
C16	0.0348 (9)	0.0300 (8)	0.0279 (8)	−0.0032 (7)	−0.0049 (6)	0.0018 (6)
C17	0.0279 (8)	0.0290 (8)	0.0249 (7)	0.0008 (6)	−0.0056 (6)	−0.0033 (6)

Geometric parameters (Å, °)

F1—C1	1.3415 (19)	C5—C10	1.397 (2)
F2—C1	1.338 (2)	C6—C7	1.380 (2)
F3—C1	1.3429 (19)	C6—H6	0.9300
O1—C4	1.2618 (18)	C7—C8	1.390 (3)
O2—C2	1.2732 (18)	C7—H7	0.9300
N1—C11	1.326 (2)	C8—C9	1.380 (2)
N1—H1A	0.877 (9)	C8—H8	0.9300

N1—H1B	0.879 (9)	C9—C10	1.392 (2)
N2—C11	1.346 (2)	C9—H9	0.9300
N2—C12	1.401 (2)	C10—H10	0.9300
N2—H2A	0.905 (10)	C12—C13	1.385 (2)
N3—C11	1.344 (2)	C12—C17	1.396 (2)
N3—C17	1.400 (2)	C13—C14	1.390 (2)
N3—H3A	0.895 (9)	C13—H13	0.9300
C1—C2	1.530 (2)	C14—C15	1.395 (2)
C2—C3	1.385 (2)	C14—H14	0.9300
C3—C4	1.430 (2)	C15—C16	1.388 (2)
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.499 (2)	C16—C17	1.385 (2)
C5—C6	1.394 (2)	C16—H16	0.9300
C11—N1—H1A	120.8 (12)	C8—C7—H7	120.0
C11—N1—H1B	118.1 (11)	C9—C8—C7	119.73 (16)
H1A—N1—H1B	121.1 (14)	C9—C8—H8	120.1
C11—N2—C12	108.60 (13)	C7—C8—H8	120.1
C11—N2—H2A	125.0 (13)	C8—C9—C10	120.49 (15)
C12—N2—H2A	125.5 (13)	C8—C9—H9	119.8
C11—N3—C17	108.79 (13)	C10—C9—H9	119.8
C11—N3—H3A	126.8 (14)	C9—C10—C5	120.07 (15)
C17—N3—H3A	124.4 (14)	C9—C10—H10	120.0
F2—C1—F1	106.81 (13)	C5—C10—H10	120.0
F2—C1—F3	105.99 (13)	N1—C11—N3	125.36 (14)
F1—C1—F3	106.30 (13)	N1—C11—N2	125.23 (15)
F2—C1—C2	111.64 (13)	N3—C11—N2	109.40 (14)
F1—C1—C2	114.24 (14)	C13—C12—C17	121.81 (14)
F3—C1—C2	111.35 (13)	C13—C12—N2	131.50 (15)
O2—C2—C3	128.36 (14)	C17—C12—N2	106.66 (13)
O2—C2—C1	113.13 (14)	C12—C13—C14	116.39 (15)
C3—C2—C1	118.47 (14)	C12—C13—H13	121.8
C2—C3—C4	123.55 (14)	C14—C13—H13	121.8
C2—C3—H3	118.2	C13—C14—C15	121.95 (15)
C4—C3—H3	118.2	C13—C14—H14	119.0
O1—C4—C3	123.29 (14)	C15—C14—H14	119.0
O1—C4—C5	117.51 (13)	C16—C15—C14	121.39 (15)
C3—C4—C5	119.18 (13)	C16—C15—H15	119.3
C6—C5—C10	118.71 (14)	C14—C15—H15	119.3
C6—C5—C4	118.92 (13)	C17—C16—C15	116.78 (15)
C10—C5—C4	122.34 (14)	C17—C16—H16	121.6
C7—C6—C5	120.98 (15)	C15—C16—H16	121.6
C7—C6—H6	119.5	C16—C17—C12	121.68 (14)
C5—C6—H6	119.5	C16—C17—N3	131.78 (14)
C6—C7—C8	120.01 (16)	C12—C17—N3	106.52 (13)
C6—C7—H7	120.0		
F2—C1—C2—O2	48.38 (18)	C4—C5—C10—C9	−177.14 (14)
F1—C1—C2—O2	169.71 (14)	C17—N3—C11—N1	179.40 (15)
F3—C1—C2—O2	−69.86 (17)	C17—N3—C11—N2	−1.38 (17)

F2—C1—C2—C3	−133.69 (15)	C12—N2—C11—N1	−179.13 (15)
F1—C1—C2—C3	−12.4 (2)	C12—N2—C11—N3	1.64 (18)
F3—C1—C2—C3	108.07 (16)	C11—N2—C12—C13	177.01 (16)
O2—C2—C3—C4	−7.8 (3)	C11—N2—C12—C17	−1.26 (17)
C1—C2—C3—C4	174.64 (14)	C17—C12—C13—C14	0.3 (2)
C2—C3—C4—O1	−8.1 (2)	N2—C12—C13—C14	−177.71 (16)
C2—C3—C4—C5	173.70 (14)	C12—C13—C14—C15	−0.6 (2)
O1—C4—C5—C6	−27.7 (2)	C13—C14—C15—C16	0.3 (2)
C3—C4—C5—C6	150.58 (15)	C14—C15—C16—C17	0.3 (2)
O1—C4—C5—C10	150.25 (14)	C15—C16—C17—C12	−0.5 (2)
C3—C4—C5—C10	−31.4 (2)	C15—C16—C17—N3	177.26 (15)
C10—C5—C6—C7	0.1 (2)	C13—C12—C17—C16	0.2 (2)
C4—C5—C6—C7	178.13 (15)	N2—C12—C17—C16	178.68 (14)
C5—C6—C7—C8	−1.0 (3)	C13—C12—C17—N3	−178.06 (14)
C6—C7—C8—C9	1.1 (3)	N2—C12—C17—N3	0.42 (16)
C7—C8—C9—C10	−0.1 (3)	C11—N3—C17—C16	−177.45 (16)
C8—C9—C10—C5	−0.8 (2)	C11—N3—C17—C12	0.57 (16)
C6—C5—C10—C9	0.9 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O2^{i}	0.877 (9)	2.004 (11)	2.7803 (18)	146.8 (16)
N3—H3A···O1^{i}	0.895 (9)	1.949 (13)	2.7651 (17)	150.9 (19)
N3—H3A···O2^{i}	0.895 (9)	2.363 (18)	2.9912 (18)	127.3 (17)
N1—H1B···O1	0.879 (9)	2.030 (10)	2.8662 (18)	158.4 (16)
N2—H2A···O2	0.905 (10)	1.862 (11)	2.7360 (18)	161.8 (19)

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

Fig. 1

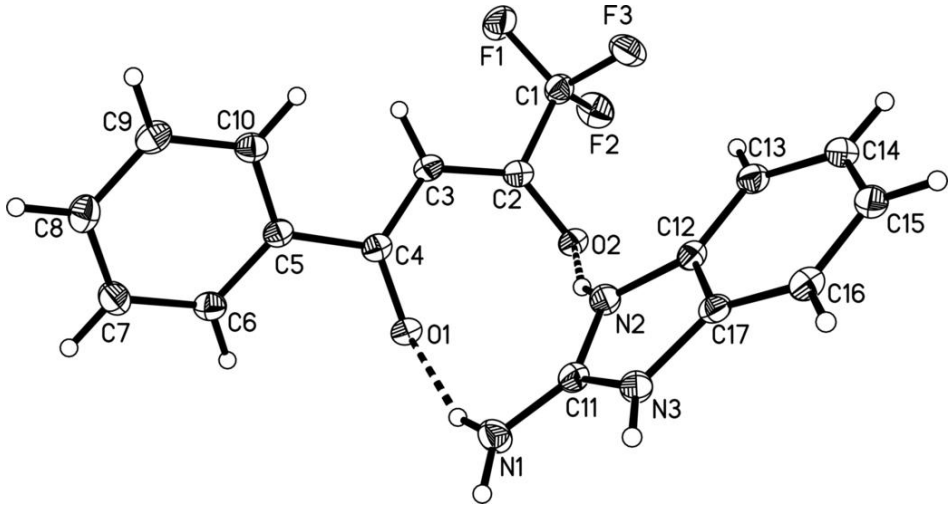
F3
F1
C1
F2
C9
C10
C13
C14
C3
C2
C8
C12
C15
O2
C5
C4
C16
N2
C7
C6
O1
C17
C11
N3
N1

Fig. 2