Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[(2,4-Dinitrophenyl)hydrazono]-4,4,4-trifluoro-1-phenylbutan-1-one

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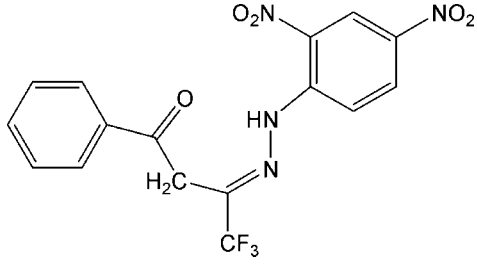
Received 8 June 2007; accepted 12 June 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.118; data-to-parameter ratio = 13.6.

The crystal structure of the title compound, $C_{16}H_{11}F_3N_4O_5$, contains two aromatic rings, which are bridged by a C=N unit. The molecular structure is stabilized by intramolecular N—H···O hydrogen bonds. The chains are linked through weak intermolecular interactions, resulting in a zigzag packing arrangement.

Related literature

For general background, see: Belloni *et al.* (2005); Kahwa *et al.* (1986); Parashar *et al.* (1988); Santos *et al.* (2001); Tynan *et al.* (2005).



Experimental

Crystal data

$C_{16}H_{11}F_3N_4O_5$
M_r = 396.29
Monoclinic, $P2_1/n$
a = 12.670 (2) Å
b = 8.8499 (14) Å
c = 15.846 (3) Å
β = 108.080 (3)°
V = 1689.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.970, T_{max} = 0.978
9400 measured reflections
3451 independent reflections
2112 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.118
S = 0.98
3451 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.86	1.98	2.604 (2)	129
N2—H2···O1	0.86	2.52	3.031 (2)	119

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2327).

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 doi:10.1107/S1600536807028747

supplementary materials

Acta Cryst. (2007). E**63**, o3203 [**doi:10.1107/S1600536807028747**]

3-[(2,4-Dinitrophenyl)hydrazono]-4,4,4-trifluoro-1-phenylbutan-1-one

Z.-L. Jing, S.-J. Zhang, N. Yang and S.-C. Feng

Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988), the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Shiff bases functioning as ligands, we report the synthesis and structure of the title compound, (I). In the molecular structure of the title compound, (I) (Fig. 1), the expected geometric parameters are observed.

The 4,4,4-trifluoro-1-phenyl-butane-1,3-dione ring system (C1—C6) is planar, with an r.m.s. deviation for the fitted atoms of 0.0023 (4) Å, as are the (2,4-dinitro-phenyl)-hydrazine group (C11—C16), with an r.m.s. deviation of 0.0150 (3) Å, and the dihedral angles formed between these planes is 82.74 (6)°. Intramolecular N—H···O hydrogen bond stabilizes the molecular conformation, and the molecules are linked *via* weak intermolecular N—H···O hydrogen bonds to forming a zigzag packing arrangement.

Experimental

An anhydrous ethanol solution (50 ml) of 4,4,4-trifluoro-1-phenyl- butane-1,3-dione (2.16 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of (2,4-dinitro-phenyl)-hydrazine (1.98 g, 10 mmol) and the mixture was stirred at 350 K for 6 h under N_2, whereupon a red solution appeared. The solvent was removed and the residue recrystallized from anhydrous ethanol. The product was isolated and then dried *in vacuo* to give pure (I) in 84% yield. Red single crystals suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution of (I).

Refinement

The N-bound H atom was located in a difference Fourier map and were refined with $U_{iso}(H) = 1.2U_{eq}(N)$. C-bound H atoms were included in calculated positions, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

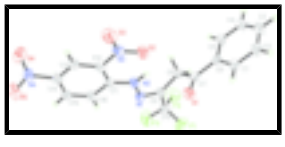

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level.

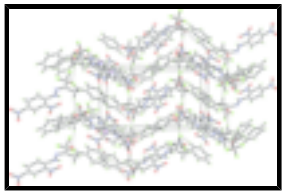

Fig. 2. Packing view of (I), showing the intermolecular hydrogen bonds (dashed lines).

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Crystal data

$C_{16}H_{11}F_3N_4O_5$	F_{000} = 808
M_r = 396.29	D_x = 1.558 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2407 reflections
a = 12.670 (2) Å	θ = 2.7–23.2º
b = 8.8499 (14) Å	μ = 0.14 mm^{-1}
c = 15.846 (3) Å	T = 294 (2) K
β = 108.080 (3)º	Block, red
V = 1689.0 (5) Å^3	0.22 × 0.20 × 0.16 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3451 independent reflections
Radiation source: fine-focus sealed tube	2112 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 294(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→15
T_{min} = 0.970, T_{max} = 0.978	k = −11→10
9400 measured reflections	l = −19→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H-atom parameters constrained
$wR(F^2)$ = 0.118	$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.2886P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.98	$(\Delta/\sigma)_{max}$ = 0.001
3451 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
253 parameters	$\Delta\rho_{min}$ = −0.19 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.52280 (12)	0.60190 (15)	0.76335 (10)	0.0816 (5)
F2	0.41425 (12)	0.78857 (19)	0.72284 (12)	0.1009 (6)
F3	0.46161 (13)	0.71459 (19)	0.85655 (11)	0.1002 (6)
O1	0.79723 (12)	0.77365 (17)	0.77580 (10)	0.0630 (4)
O2	0.90737 (12)	1.09368 (18)	0.89456 (11)	0.0637 (4)
O3	1.00769 (13)	1.2392 (2)	0.99578 (12)	0.0780 (5)
O4	0.92171 (15)	1.3453 (2)	1.25985 (12)	0.0799 (5)
O5	0.83158 (16)	1.1765 (2)	1.30921 (11)	0.0870 (6)
N1	0.64300 (13)	0.86642 (18)	0.89153 (11)	0.0476 (4)
N2	0.73460 (13)	0.95719 (18)	0.91460 (11)	0.0497 (4)
H2	0.7683	0.9782	0.8766	0.060*
N3	0.93045 (14)	1.1511 (2)	0.96872 (12)	0.0515 (4)
N4	0.86534 (16)	1.2290 (2)	1.25123 (13)	0.0619 (5)
C1	0.84995 (17)	0.8257 (2)	0.61940 (15)	0.0527 (5)
H1	0.8928	0.7563	0.6597	0.063*
C2	0.8773 (2)	0.8615 (3)	0.54414 (16)	0.0668 (7)
H2A	0.9378	0.8154	0.5334	0.080*
C3	0.8150 (2)	0.9655 (3)	0.48503 (17)	0.0743 (7)
H3	0.8338	0.9900	0.4345	0.089*
C4	0.7249 (2)	1.0337 (3)	0.50012 (15)	0.0693 (7)
H4	0.6832	1.1042	0.4599	0.083*
C5	0.69638 (19)	0.9972 (2)	0.57511 (14)	0.0558 (5)
H5	0.6352	1.0429	0.5850	0.067*
C6	0.75850 (15)	0.8929 (2)	0.63559 (13)	0.0439 (5)
C7	0.73331 (16)	0.8507 (2)	0.71830 (13)	0.0439 (5)
C8	0.62590 (16)	0.9075 (2)	0.73105 (13)	0.0458 (5)
H8A	0.6309	1.0164	0.7380	0.055*
H8B	0.5654	0.8861	0.6774	0.055*
C9	0.59704 (16)	0.8416 (2)	0.80879 (14)	0.0450 (5)
C10	0.49893 (19)	0.7371 (3)	0.78885 (16)	0.0568 (6)
C11	0.77211 (15)	1.0141 (2)	0.99852 (12)	0.0433 (5)

C12	0.86413 (15)	1.1134 (2)	1.02618 (13)	0.0435 (5)
C13	0.89433 (16)	1.1825 (2)	1.10864 (13)	0.0474 (5)
H13	0.9537	1.2496	1.1250	0.057*
C14	0.83583 (17)	1.1508 (2)	1.16547 (13)	0.0482 (5)
C15	0.74946 (18)	1.0470 (2)	1.14354 (14)	0.0536 (5)
H15	0.7135	1.0222	1.1847	0.064*
C16	0.71734 (18)	0.9813 (2)	1.06135 (14)	0.0518 (5)
H16	0.6582	0.9137	1.0467	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0895 (10)	0.0567 (8)	0.1060 (12)	−0.0184 (7)	0.0410 (9)	−0.0213 (8)
F2	0.0541 (9)	0.1086 (12)	0.1215 (14)	−0.0132 (8)	0.0002 (9)	0.0215 (10)
F3	0.0975 (11)	0.1294 (13)	0.0980 (12)	−0.0536 (10)	0.0659 (10)	−0.0294 (10)
O1	0.0653 (10)	0.0730 (10)	0.0542 (9)	0.0225 (8)	0.0235 (8)	0.0121 (8)
O2	0.0675 (10)	0.0758 (11)	0.0578 (10)	−0.0087 (8)	0.0342 (9)	−0.0039 (8)
O3	0.0616 (10)	0.0989 (13)	0.0766 (12)	−0.0297 (10)	0.0259 (9)	−0.0022 (10)
O4	0.0753 (11)	0.0800 (12)	0.0771 (12)	−0.0021 (10)	0.0130 (9)	−0.0242 (10)
O5	0.0947 (13)	0.1202 (16)	0.0483 (10)	0.0047 (11)	0.0251 (10)	−0.0046 (10)
N1	0.0494 (10)	0.0469 (10)	0.0513 (11)	−0.0034 (8)	0.0226 (9)	−0.0026 (8)
N2	0.0531 (10)	0.0553 (10)	0.0455 (10)	−0.0096 (8)	0.0222 (8)	−0.0023 (8)
N3	0.0457 (10)	0.0551 (11)	0.0560 (12)	0.0005 (9)	0.0191 (9)	0.0110 (9)
N4	0.0583 (12)	0.0754 (14)	0.0483 (12)	0.0172 (11)	0.0113 (10)	−0.0016 (11)
C1	0.0468 (12)	0.0590 (13)	0.0552 (13)	−0.0029 (10)	0.0198 (10)	−0.0034 (11)
C2	0.0639 (15)	0.0778 (17)	0.0712 (17)	−0.0067 (13)	0.0390 (13)	−0.0081 (14)
C3	0.0924 (19)	0.0799 (18)	0.0636 (16)	−0.0149 (15)	0.0434 (15)	−0.0004 (14)
C4	0.0914 (18)	0.0643 (15)	0.0580 (15)	0.0050 (13)	0.0318 (14)	0.0112 (12)
C5	0.0642 (14)	0.0529 (12)	0.0528 (13)	0.0058 (11)	0.0219 (11)	0.0007 (11)
C6	0.0462 (11)	0.0414 (10)	0.0456 (11)	−0.0060 (9)	0.0167 (9)	−0.0055 (9)
C7	0.0492 (11)	0.0385 (10)	0.0457 (12)	−0.0003 (9)	0.0171 (10)	−0.0039 (9)
C8	0.0470 (11)	0.0447 (11)	0.0470 (12)	0.0034 (9)	0.0168 (9)	−0.0010 (9)
C9	0.0455 (11)	0.0427 (11)	0.0520 (13)	0.0019 (9)	0.0227 (10)	−0.0017 (9)
C10	0.0562 (13)	0.0592 (14)	0.0601 (14)	−0.0081 (11)	0.0254 (12)	−0.0046 (11)
C11	0.0466 (11)	0.0439 (11)	0.0411 (11)	0.0039 (9)	0.0162 (9)	0.0046 (9)
C12	0.0421 (10)	0.0458 (11)	0.0440 (11)	0.0047 (9)	0.0156 (9)	0.0080 (9)
C13	0.0415 (11)	0.0485 (12)	0.0483 (12)	0.0048 (9)	0.0084 (9)	0.0047 (10)
C14	0.0489 (12)	0.0512 (12)	0.0423 (12)	0.0092 (10)	0.0109 (10)	0.0024 (10)
C15	0.0613 (13)	0.0597 (13)	0.0463 (12)	0.0061 (11)	0.0262 (11)	0.0092 (10)
C16	0.0562 (12)	0.0526 (12)	0.0515 (13)	−0.0067 (10)	0.0237 (10)	0.0037 (10)

Geometric parameters (Å, °)

F1—C10	1.328 (3)	C3—H3	0.9300
F2—C10	1.325 (3)	C4—C5	1.383 (3)
F3—C10	1.315 (2)	C4—H4	0.9300
O1—C7	1.222 (2)	C5—C6	1.386 (3)
O2—N3	1.229 (2)	C5—H5	0.9300
O3—N3	1.221 (2)	C6—C7	1.490 (3)

O4—N4	1.235 (2)	C7—C8	1.522 (3)
O5—N4	1.220 (2)	C8—C9	1.507 (3)
N1—C9	1.278 (2)	C8—H8A	0.9700
N1—N2	1.365 (2)	C8—H8B	0.9700
N2—C11	1.362 (2)	C9—C10	1.502 (3)
N2—H2	0.8600	C11—C16	1.409 (3)
N3—C12	1.456 (2)	C11—C12	1.417 (3)
N4—C14	1.466 (3)	C12—C13	1.385 (3)
C1—C2	1.378 (3)	C13—C14	1.361 (3)
C1—C6	1.395 (3)	C13—H13	0.9300
C1—H1	0.9300	C14—C15	1.388 (3)
C2—C3	1.374 (3)	C15—C16	1.368 (3)
C2—H2A	0.9300	C15—H15	0.9300
C3—C4	1.375 (3)	C16—H16	0.9300
C9—N1—N2	117.07 (16)	C9—C8—H8A	108.3
C11—N2—N1	119.28 (16)	C7—C8—H8A	108.3
C11—N2—H2	120.4	C9—C8—H8B	108.3
N1—N2—H2	120.4	C7—C8—H8B	108.3
O3—N3—O2	121.79 (17)	H8A—C8—H8B	107.4
O3—N3—C12	118.81 (19)	N1—C9—C10	113.94 (18)
O2—N3—C12	119.40 (17)	N1—C9—C8	128.54 (18)
O5—N4—O4	124.1 (2)	C10—C9—C8	117.47 (18)
O5—N4—C14	118.0 (2)	F3—C10—F2	106.75 (19)
O4—N4—C14	117.9 (2)	F3—C10—F1	106.58 (19)
C2—C1—C6	120.5 (2)	F2—C10—F1	105.94 (19)
C2—C1—H1	119.8	F3—C10—C9	113.91 (19)
C6—C1—H1	119.8	F2—C10—C9	111.65 (18)
C3—C2—C1	119.9 (2)	F1—C10—C9	111.53 (18)
C3—C2—H2A	120.0	N2—C11—C16	120.96 (18)
C1—C2—H2A	120.0	N2—C11—C12	122.39 (17)
C2—C3—C4	120.5 (2)	C16—C11—C12	116.59 (18)
C2—C3—H3	119.8	C13—C12—C11	121.73 (18)
C4—C3—H3	119.8	C13—C12—N3	116.41 (18)
C3—C4—C5	119.9 (2)	C11—C12—N3	121.84 (18)
C3—C4—H4	120.0	C14—C13—C12	119.04 (19)
C5—C4—H4	120.0	C14—C13—H13	120.5
C4—C5—C6	120.4 (2)	C12—C13—H13	120.5
C4—C5—H5	119.8	C13—C14—C15	121.31 (19)
C6—C5—H5	119.8	C13—C14—N4	118.8 (2)
C5—C6—C1	118.84 (19)	C15—C14—N4	119.93 (19)
C5—C6—C7	122.99 (18)	C16—C15—C14	119.89 (19)
C1—C6—C7	118.16 (18)	C16—C15—H15	120.1
O1—C7—C6	121.51 (17)	C14—C15—H15	120.1
O1—C7—C8	119.90 (17)	C15—C16—C11	121.3 (2)
C6—C7—C8	118.58 (17)	C15—C16—H16	119.4
C9—C8—C7	115.74 (16)	C11—C16—H16	119.4
C9—N1—N2—C11	165.70 (17)	C8—C9—C10—F1	75.6 (2)
C6—C1—C2—C3	0.8 (3)	N1—N2—C11—C16	−0.4 (3)

C1—C2—C3—C4	−0.4 (4)	N1—N2—C11—C12	−177.63 (16)
C2—C3—C4—C5	−0.2 (4)	N2—C11—C12—C13	173.26 (17)
C3—C4—C5—C6	0.4 (4)	C16—C11—C12—C13	−4.1 (3)
C4—C5—C6—C1	0.0 (3)	N2—C11—C12—N3	−5.3 (3)
C4—C5—C6—C7	178.9 (2)	C16—C11—C12—N3	177.36 (17)
C2—C1—C6—C5	−0.6 (3)	O3—N3—C12—C13	0.2 (3)
C2—C1—C6—C7	−179.60 (19)	O2—N3—C12—C13	−179.73 (17)
C5—C6—C7—O1	−170.44 (19)	O3—N3—C12—C11	178.87 (18)
C1—C6—C7—O1	8.5 (3)	O2—N3—C12—C11	−1.1 (3)
C5—C6—C7—C8	8.7 (3)	C11—C12—C13—C14	1.8 (3)
C1—C6—C7—C8	−172.37 (17)	N3—C12—C13—C14	−179.53 (17)
O1—C7—C8—C9	−9.9 (3)	C12—C13—C14—C15	2.3 (3)
C6—C7—C8—C9	170.94 (17)	C12—C13—C14—N4	−177.54 (17)
N2—N1—C9—C10	178.27 (16)	O5—N4—C14—C13	−163.28 (19)
N2—N1—C9—C8	−4.3 (3)	O4—N4—C14—C13	17.7 (3)
C7—C8—C9—N1	71.9 (3)	O5—N4—C14—C15	16.9 (3)
C7—C8—C9—C10	−110.8 (2)	O4—N4—C14—C15	−162.20 (19)
N1—C9—C10—F3	14.0 (3)	C13—C14—C15—C16	−4.0 (3)
C8—C9—C10—F3	−163.70 (18)	N4—C14—C15—C16	175.84 (19)
N1—C9—C10—F2	135.0 (2)	C14—C15—C16—C11	1.5 (3)
C8—C9—C10—F2	−42.7 (3)	N2—C11—C16—C15	−175.03 (18)
N1—C9—C10—F1	−106.7 (2)	C12—C11—C16—C15	2.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.86	1.98	2.604 (2)	129
N2—H2···O1	0.86	2.52	3.031 (2)	119

Fig. 1

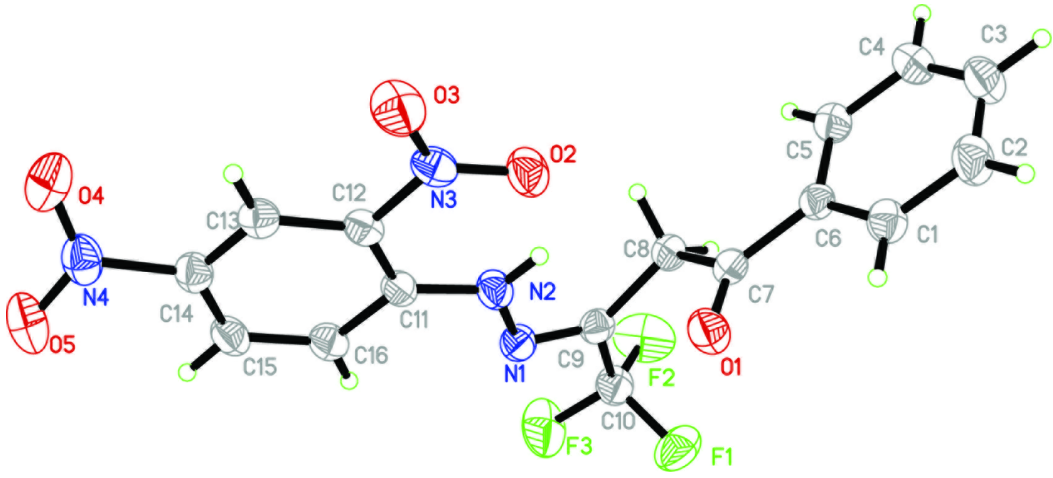
O3
O2
O4
N3
C12
C13
C4
C3
C5
C2
C6
C1
C8
C7
N4
C14
C11
N2
O5
C15
C16
N1
C9
F2
O1
C10
F3
F1

Fig. 2