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Key indicators

Single-crystal X-ray study
 $T = 120\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.021
 wR factor = 0.050
 Data-to-parameter ratio = 15.6

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

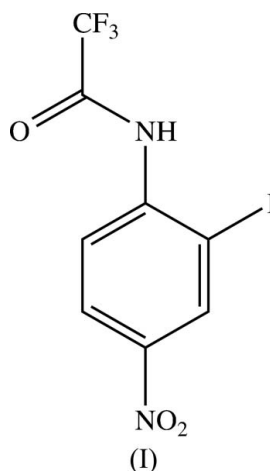
2-Iodo-4-nitro-*N*-(trifluoroacetyl)aniline: sheets built from iodo–nitro and nitro–nitro interactions

In the title compound, $\text{C}_8\text{H}_4\text{F}_3\text{IN}_2\text{O}_3$, the molecules are linked into sheets by a combination of a nearly symmetrical three-centre iodo–nitro interaction and a dipolar nitro–nitro interaction. Hydrogen bonds are absent from the structure.

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Comment

We have recently reported the supramolecular structures of a range of iodonitroanilines, which exhibit a wide variety of intermolecular interactions including hydrogen bonds, iodo–nitro interactions and aromatic π – π stacking interactions (Garden *et al.*, 2001, 2002, 2004, 2005; McWilliam *et al.*, 2001). Continuing this study, we now report the structure of 2-iodo-4-nitro-*N*-(trifluoroacetyl)aniline, (I) (Fig. 1).



With the exception of the trifluoromethyl group, the molecule of (I) is approximately planar, as shown by the key torsion angles (Table 1); the bond distances and inter-bond angles show no unusual values.

The molecules of (I) are linked by a nearly symmetrical three-centre iodo–nitro interaction. Atom I2 in the molecule at (x, y, z) makes contacts with both nitro atoms O41 and O42 in the molecule at $(-\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z)$, with geometric parameters $\text{I2} \cdots \text{O41}^i = 3.3853(16)\text{ \AA}$, $\text{I2} \cdots \text{O42}^i = 3.4159(18)\text{ \AA}$, $\text{C2}-\text{I2} \cdots \text{O41}^i = 158.24(6)^\circ$, $\text{C2}-\text{I2} \cdots \text{O42}^i = 155.32(6)^\circ$ and $\text{O41}^i \cdots \text{I2} \cdots \text{O42}^i = 37.06(4)^\circ$ [symmetry code: (i) $-\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z$]. Propagation of this interaction then produces a $C(6)C(6)[R_2^1(4)]$ chain of rings (Bernstein *et al.*, 1995; Starbuck *et al.*, 1999) running parallel to the [101] direction and generated by the n -glide plane at $y = \frac{3}{4}$ (Fig. 2).

Two chains of this type, which are related to one another by inversion and which are thus anti-parallel, pass through each unit cell. The [101] chains are linked into sheets by a dipolar

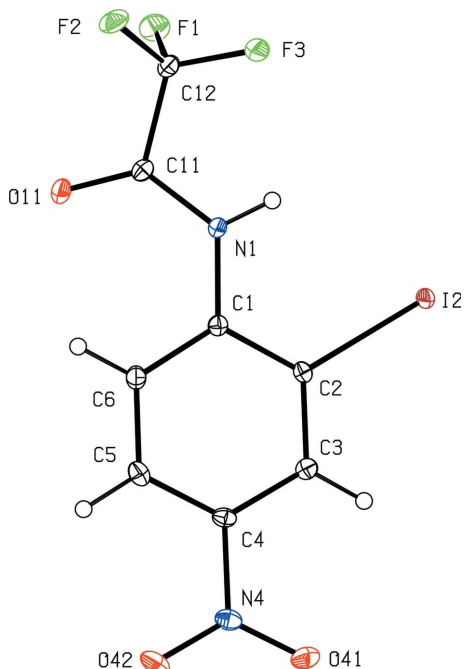


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

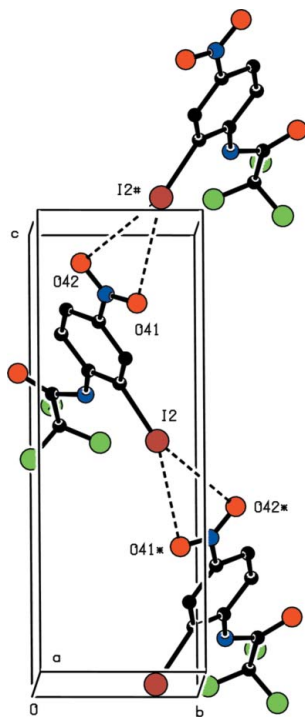


Figure 2
Part of the crystal structure of compound (I), showing the formation of a chain of rings along the [101] direction. For the sake of clarity, H atoms have all been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(-\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z)$ and $(\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z)$, respectively.

nitro–nitro interaction. Nitro atom O41 in the molecule at (x, y, z) makes a short dipolar contact with nitro atom N4 in the molecule at $(\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$, with geometric parameters $O41 \cdots N4^{ii} = 2.872(2) \text{ \AA}$ and $N4-O41 \cdots N4^{ii} = 148.26(14)^\circ$

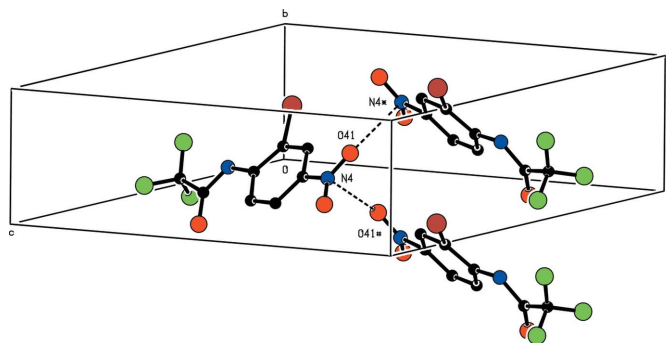


Figure 3
Part of the crystal structure of compound (I), showing the formation of a chain along the [010] direction. For the sake of clarity, H atoms have all been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$ and $(\frac{3}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z)$, respectively.

[symmetry code: (ii) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$]. Propagation of this interaction, which resembles the type I (perpendicular) carbonyl–carbonyl interaction (Allen *et al.*, 1998), produces a $C(2)$ chain running parallel to the [010] direction and generated by the 2_1 screw axis along $(\frac{3}{2}, y, \frac{3}{2})$ (Fig. 3).

The combination of [101] and [010] chains generates a sheet parallel to $(10\bar{1})$, but there are no direction-specific interactions between adjacent sheets; in particular, hydrogen bonds of all types and aromatic π – π stacking interactions are absent from the structure of (I). The absence of any participation by the amide group in any significant intermolecular interactions is unexpected.

Experimental

2-Iodo-4-nitro-*N*-(trifluoroacetyl)aniline was prepared according to a published method (Latham & Stanforth, 1997) and recrystallized from ethanol (m.p. 399–400 K).

Crystal data

$C_8H_4F_3IN_2O_5$
 $M_r = 360.03$
Monoclinic, $P2_1/n$
 $a = 14.6372(3) \text{ \AA}$
 $b = 5.0029(2) \text{ \AA}$
 $c = 15.2723(3) \text{ \AA}$
 $\beta = 110.318(2)^\circ$
 $V = 1048.78(5) \text{ \AA}^3$

$Z = 4$
 $D_x = 2.280 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
 $\mu = 3.10 \text{ mm}^{-1}$
 $T = 120(2) \text{ K}$
Block, yellow
 $0.30 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
Absorption correction: multi-scan
(*DENZO-SMN*; Otwinowski & Minor, 1997)
 $T_{\min} = 0.425$, $T_{\max} = 0.536$

13862 measured reflections
2402 independent reflections
2214 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$
 $\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.021$
 $wR(F^2) = 0.050$
 $S = 1.14$
2402 reflections
154 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0133P)^2 + 1.0182P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.62 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.83 \text{ e \AA}^{-3}$

Table 1

Selected torsion angles (°).

C2–C1–N1–C11	164.14 (19)	C3–C4–N4–O41	4.7 (3)
C1–N1–C11–C12	–178.11 (18)		

All H atoms were located in difference maps and then treated as riding, with C–H = 0.95 Å, N–H = 0.88 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC National X-ray Crystallography Service, University of Southampton, England; the authors thank the staff of the Service for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

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Acta Cryst. (2006). E62, o3762–o3764 [https://doi.org/10.1107/S1600536806029072]

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$C_8H_4F_3IN_2O_3$

$M_r = 360.03$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 14.6372$ (3) Å

$b = 5.0029$ (2) Å

$c = 15.2723$ (3) Å

$\beta = 110.318$ (2)°

$V = 1048.78$ (5) Å³

$Z = 4$

$F(000) = 680$

$D_x = 2.280$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2402 reflections

$\theta = 3.0$ – 27.5 °

$\mu = 3.10$ mm⁻¹

$T = 120$ K

Block, yellow

$0.30 \times 0.20 \times 0.20$ mm

Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed X-ray tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(DENZO-SMN; Otwinowski & Minor, 1997)

$T_{\min} = 0.425$, $T_{\max} = 0.536$

13862 measured reflections

2402 independent reflections

2214 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.0$ °

$h = -19 \rightarrow 18$

$k = -6 \rightarrow 6$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.021$

$wR(F^2) = 0.050$

$S = 1.14$

2402 reflections

154 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0133P)^2 + 1.0182P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.62$ e Å⁻³

$\Delta\rho_{\min} = -0.83$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.41248 (14)	0.3324 (4)	0.68419 (13)	0.0136 (4)

C2	0.46717 (14)	0.5180 (4)	0.65374 (12)	0.0132 (4)
C3	0.56627 (14)	0.5485 (4)	0.70208 (13)	0.0157 (4)
C4	0.60949 (14)	0.3870 (4)	0.77944 (13)	0.0158 (4)
C5	0.55859 (17)	0.1934 (4)	0.80803 (15)	0.0187 (4)
C6	0.45995 (16)	0.1672 (4)	0.76057 (14)	0.0177 (4)
N1	0.31119 (13)	0.3194 (4)	0.63785 (12)	0.0148 (3)
C11	0.25016 (14)	0.1210 (4)	0.64240 (13)	0.0152 (4)
O11	0.27013 (11)	-0.0869 (3)	0.68607 (10)	0.0210 (3)
C12	0.14369 (16)	0.1693 (4)	0.57965 (15)	0.0180 (4)
F1	0.11944 (10)	-0.0034 (3)	0.50804 (9)	0.0278 (3)
F2	0.08382 (9)	0.1259 (3)	0.62684 (9)	0.0274 (3)
F3	0.12630 (9)	0.4152 (3)	0.54381 (10)	0.0269 (3)
I2	0.402878 (9)	0.74612 (2)	0.532577 (8)	0.01443 (7)
N4	0.71340 (13)	0.4257 (4)	0.83326 (12)	0.0199 (4)
O41	0.75675 (11)	0.6106 (3)	0.81166 (11)	0.0288 (4)
O42	0.75250 (14)	0.2737 (3)	0.89876 (13)	0.0293 (4)
H3	0.6035	0.6762	0.6828	0.019*
H5	0.5910	0.0805	0.8595	0.022*
H6	0.4239	0.0361	0.7798	0.021*
H1	0.2844	0.4559	0.6017	0.018*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0157 (9)	0.0122 (9)	0.0130 (8)	-0.0006 (8)	0.0050 (7)	-0.0015 (7)
C2	0.0163 (9)	0.0122 (9)	0.0107 (8)	0.0016 (7)	0.0044 (7)	-0.0002 (7)
C3	0.0160 (9)	0.0176 (10)	0.0142 (9)	-0.0014 (8)	0.0061 (7)	-0.0014 (7)
C4	0.0138 (9)	0.0166 (9)	0.0149 (9)	0.0008 (8)	0.0026 (7)	-0.0036 (7)
C5	0.0222 (11)	0.0175 (9)	0.0147 (9)	0.0031 (9)	0.0044 (8)	0.0019 (8)
C6	0.0215 (10)	0.0151 (9)	0.0154 (9)	-0.0022 (9)	0.0048 (8)	0.0012 (8)
N1	0.0136 (8)	0.0131 (7)	0.0175 (8)	-0.0009 (7)	0.0053 (7)	0.0023 (7)
C11	0.0158 (9)	0.0151 (10)	0.0172 (9)	-0.0015 (8)	0.0087 (7)	-0.0026 (7)
O11	0.0215 (7)	0.0149 (7)	0.0269 (8)	-0.0020 (6)	0.0091 (6)	0.0038 (6)
C12	0.0173 (10)	0.0172 (10)	0.0199 (10)	-0.0037 (9)	0.0070 (8)	-0.0003 (9)
F1	0.0294 (7)	0.0271 (7)	0.0224 (6)	-0.0056 (6)	0.0034 (5)	-0.0084 (5)
F2	0.0174 (6)	0.0378 (8)	0.0303 (7)	-0.0064 (6)	0.0126 (5)	0.0008 (6)
F3	0.0183 (6)	0.0202 (6)	0.0376 (7)	-0.0003 (5)	0.0039 (5)	0.0059 (6)
I2	0.01406 (9)	0.01624 (10)	0.01339 (9)	0.00049 (4)	0.00527 (6)	0.00346 (4)
N4	0.0169 (8)	0.0241 (9)	0.0156 (8)	0.0002 (7)	0.0017 (7)	-0.0032 (7)
O41	0.0199 (8)	0.0331 (9)	0.0274 (8)	-0.0092 (7)	0.0006 (6)	0.0005 (7)
O42	0.0215 (9)	0.0359 (10)	0.0232 (9)	0.0055 (7)	-0.0017 (7)	0.0069 (6)

Geometric parameters (Å, °)

C1—C6	1.401 (3)	C6—H6	0.95
C1—N1	1.405 (3)	N1—C11	1.353 (3)
C1—C2	1.406 (3)	N1—H1	0.88
C2—C3	1.389 (3)	C11—O11	1.215 (2)

C2—I2	2.0982 (18)	C11—C12	1.539 (3)
C3—C4	1.389 (3)	C12—F2	1.332 (2)
C3—H3	0.95	C12—F3	1.335 (3)
C4—C5	1.382 (3)	C12—F1	1.341 (2)
C4—N4	1.469 (2)	N4—O42	1.228 (2)
C5—C6	1.379 (3)	N4—O41	1.230 (2)
C5—H5	0.95		
C6—C1—N1	121.56 (18)	C1—C6—H6	119.7
C6—C1—C2	119.34 (18)	C11—N1—C1	127.44 (18)
N1—C1—C2	119.09 (17)	C11—N1—H1	116.3
C3—C2—C1	120.40 (17)	C1—N1—H1	116.3
C3—C2—I2	118.36 (14)	O11—C11—N1	128.23 (19)
C1—C2—I2	121.21 (14)	O11—C11—C12	118.34 (18)
C2—C3—C4	118.13 (18)	N1—C11—C12	113.37 (17)
C2—C3—H3	120.9	F2—C12—F3	107.75 (18)
C4—C3—H3	120.9	F2—C12—F1	107.37 (17)
C5—C4—C3	122.69 (19)	F3—C12—F1	107.51 (17)
C5—C4—N4	118.85 (18)	F2—C12—C11	110.60 (17)
C3—C4—N4	118.47 (18)	F3—C12—C11	114.07 (17)
C6—C5—C4	118.80 (19)	F1—C12—C11	109.30 (17)
C6—C5—H5	120.6	O42—N4—O41	123.11 (18)
C4—C5—H5	120.6	O42—N4—C4	118.25 (18)
C5—C6—C1	120.5 (2)	O41—N4—C4	118.64 (17)
C5—C6—H6	119.7		
C6—C1—C2—C3	-3.8 (3)	C2—C1—N1—C11	164.14 (19)
N1—C1—C2—C3	175.52 (18)	C1—N1—C11—O11	-1.1 (3)
C6—C1—C2—I2	174.30 (15)	C1—N1—C11—C12	-178.11 (18)
N1—C1—C2—I2	-6.4 (2)	O11—C11—C12—F2	50.4 (3)
C1—C2—C3—C4	1.4 (3)	N1—C11—C12—F2	-132.29 (19)
I2—C2—C3—C4	-176.70 (14)	O11—C11—C12—F3	171.99 (18)
C2—C3—C4—C5	2.0 (3)	N1—C11—C12—F3	-10.7 (3)
C2—C3—C4—N4	-177.50 (17)	O11—C11—C12—F1	-67.6 (2)
C3—C4—C5—C6	-3.0 (3)	N1—C11—C12—F1	109.71 (19)
N4—C4—C5—C6	176.49 (18)	C5—C4—N4—O42	4.1 (3)
C4—C5—C6—C1	0.6 (3)	C3—C4—N4—O42	-176.38 (19)
N1—C1—C6—C5	-176.51 (19)	C5—C4—N4—O41	-174.9 (2)
C2—C1—C6—C5	2.7 (3)	C3—C4—N4—O41	4.7 (3)
C6—C1—N1—C11	-16.6 (3)		
