

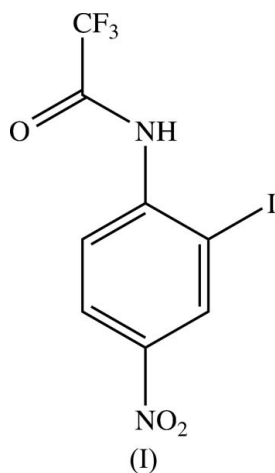
Simon J. Garden,^a James L. Wardell,^a John N. Low,^b Janet M. S. Skakle^b and Christopher Glidewell^{c*}^aInstituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, CP 68563, 21945-970 Rio de Janeiro, RJ, Brazil, ^bDepartment of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and ^cSchool of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland

Correspondence e-mail: cg@st-andrews.ac.uk

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 = 15.6For 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 interactionsIn 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.Received 5 July 2006
Accepted 26 July 2006

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

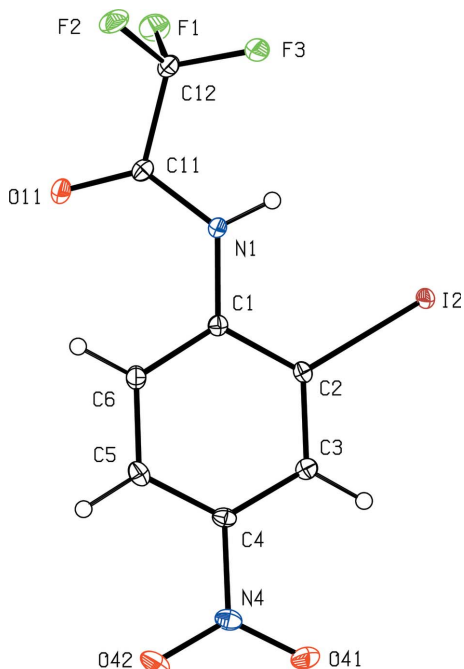


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

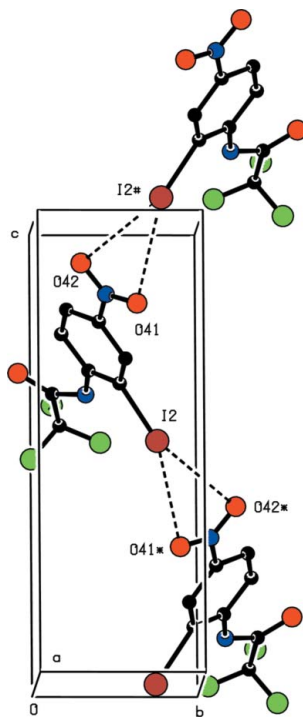


Figure 2
Part of the crystal structure of compound (I), showing the formation of a chain of rings along the [101] direction. For the sake of clarity, H atoms have all been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(-\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z)$ and $(\frac{3}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z)$, 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 $N4-O41 \cdots N4^{ii} = 148.26(14)^\circ$

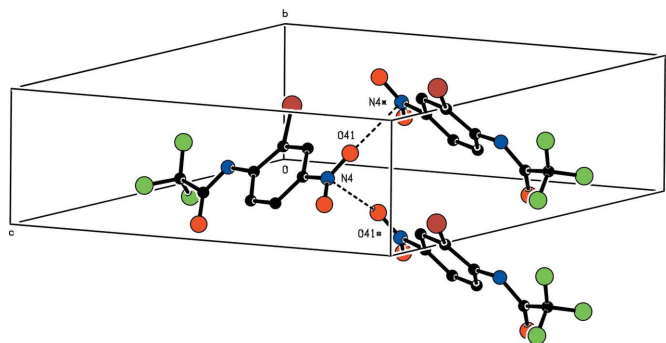


Figure 3
Part of the crystal structure of compound (I), showing the formation of a chain along the [010] direction. For the sake of clarity, H atoms have all been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$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

References

- Allen, F. H., Baalham, C. A., Lommerse, J. P. M. & Raithby, P. R. (1998). *Acta Cryst.* **B54**, 320–329.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
- Garden, S. J., Fontes, S. P., Wardell, J. L., Skakle, J. M. S., Low, J. N. & Glidewell, C. (2002). *Acta Cryst.* **B58**, 701–709.
- Garden, S. J., Glidewell, C., Low, J. N., McWilliam, S. A., Pinto, A. C., Skakle, J. M. S., Torres, J. C. & Wardell, J. L. (2001). *Acta Cryst.* **C57**, 1212–1214.
- Garden, S. J., Glidewell, C., Low, J. N., Skakle, J. M. S. & Wardell, J. L. (2005). *Acta Cryst.* **C61**, o145–o147.
- Garden, S. J., Wardell, J. L., Skakle, J. M. S., Low, J. N. & Glidewell, C. (2004). *Acta Cryst.* **C60**, o328–o330.
- Latham, E. J. & Stanforth, S. P. (1997). *J. Chem. Soc. Perkin Trans. 1*, pp. 2059–2063.
- McWilliam, S. A., Skakle, J. M. S., Low, J. N., Wardell, J. L., Garden, S. J., Pinto, A. C., Torres, J. C. & Glidewell, C. (2001). *Acta Cryst.* **C57**, 942–945.
- Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version, Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Starbuck, J., Norman, N. C. & Orpen, A. G. (1999). *New J. Chem.* **23**, 969–972.