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1-Iodo-4-methoxy-2-nitrobenzene

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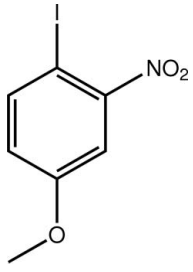
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.014; wR factor = 0.039; data-to-parameter ratio = 16.7.

In the title compound, $C_7H_6INO_3$, the 12 non-H atoms are planar, with an r.m.s. deviation of 0.016 Å. A close intramolecular I···O interaction [3.0295 (13) Å] is present. Intermolecular I···O interactions [3.3448 (13) Å] lead to the formation of zigzag chains along the b axis. These are assembled into layers by weak π–π interactions [centroid–centroid distance = 3.8416 (9) Å], and the layers stack along the a axis, being connected by C—H···O contacts.

Related literature

For general background to halogen bonding, see: Metrangolo *et al.* (2008); Pennington *et al.* (2008). For previous structural studies probing iodo–nitro interactions, see: Glidewell *et al.* (2002, 2004); Garden *et al.* (2002). For van der Waals radii, see: Bondi (1964).



Experimental

Crystal data

$C_7H_6INO_3$
M_r = 279.03
Orthorhombic, *Pbca*
a = 18.6370 (13) Å
b = 11.6257 (5) Å
c = 7.4740 (3) Å
V = 1619.38 (15) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.92 mm^{-1}
T = 100 K
0.13 × 0.09 × 0.01 mm

Data collection

Rigaku Saturn724+ (2 × 2 bin mode) diffractometer
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2011)
T_{min} = 0.755, T_{max} = 1.000
8053 measured reflections
1841 independent reflections
1615 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.014
$wR(F^2)$ = 0.039
S = 1.04
1841 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7B···O1^{i}	0.98	2.58	3.4503 (19)	148

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

Data collection: *CrystalClear-SM Expert* (Rigaku, 2011); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5176).

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supplementary materials

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Comment

In connection with previous structural studies of iodo···nitro interactions (Glidewell *et al.*, 2002; Garden *et al.*, 2002; Glidewell *et al.*, 2004), the crystal and molecular structure of the title compound (I) was undertaken in order to probe the structure for possible I···O halogen bonding (Metrangolo *et al.*, 2008; Pennington *et al.*, 2008).

The 12 non-hydrogen atoms comprising (I), Fig. 1, are co-planar with a rm.s. deviation = 0.016 Å; the maximum deviations of ±0.026 Å being found for the nitro-O atoms. A close intramolecular I1···O1 interaction of 3.0295 (13) Å is noted that is significantly less than the sum of the van der Waals radii for these atoms, *i.e*. 3.50 Å (Bondi, 1964). There are also notable intermolecular I1···O interactions, the shortest of 3.3448 (13) Å occurs with the O1^{i} atom [symmetry operation *i*: 3/2 - *x*, 1/2 + *y*, *z*]. A longer interaction [3.4530 (13) Å] is formed with the O2 atom of the same nitro group. The I1···O1^{i} interactions lead to the formation of zigzag chains along the *b* axis, Fig. 2. The supramolecular chains are assembled into layers in the *bc* plane by weak π–π interactions [ring centroid···centroid distance = 3.8416 (9) Å for symmetry operation *x*, 3/2 - *y*, -1/2 + *z*]. Layers stack along the *a* axis and are connected by C—H···O contacts, Fig. 3 and Table 1.

Experimental

The commercial compound (Aldrich) was recrystallized from EtOH; *M*.pt: 334–335 K.

Refinement

The *C*-bound H atoms were geometrically placed (C—H = 0.95–0.98 Å) and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C).

Computing details

Data collection: *CrystalClear*-SM Expert (Rigaku, 2011); cell refinement: *CrystalClear*-SM Expert (Rigaku, 2011); data reduction: *CrystalClear*-SM Expert (Rigaku, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

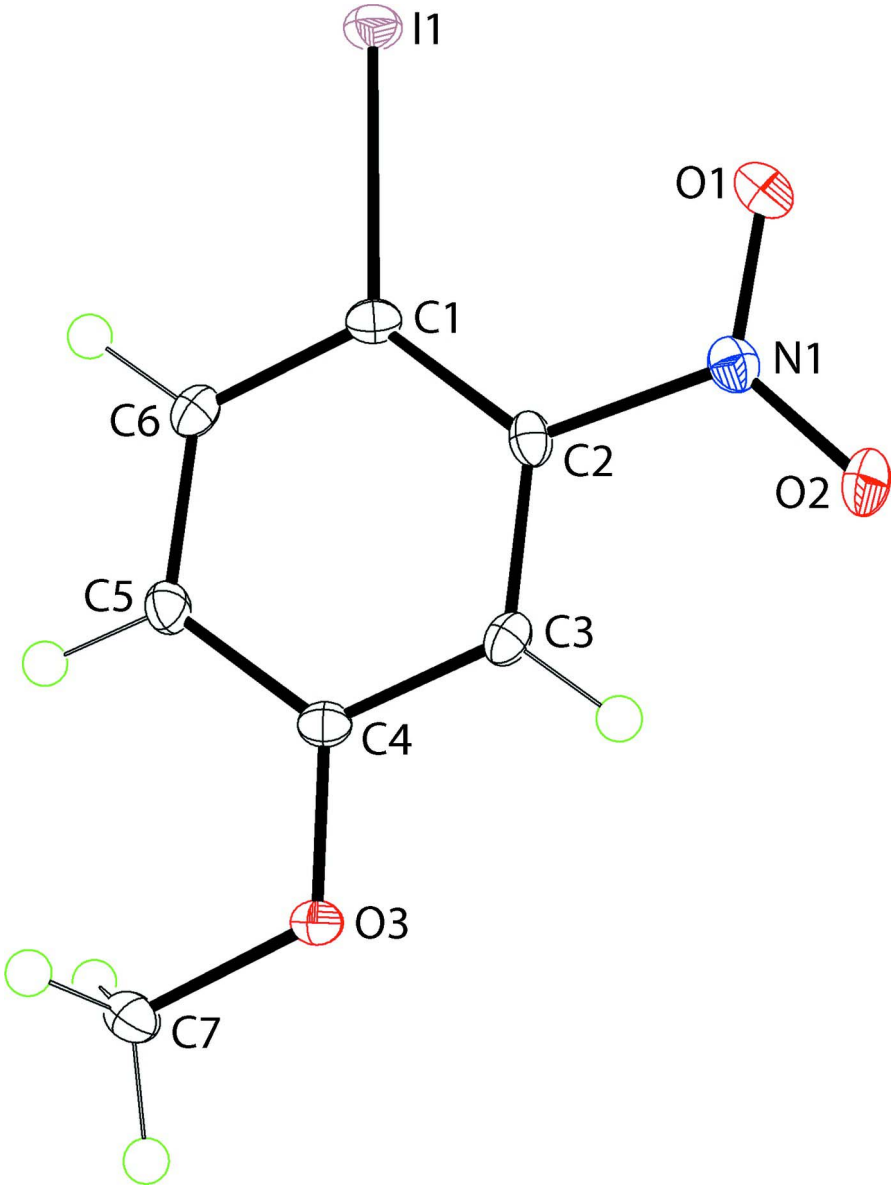

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

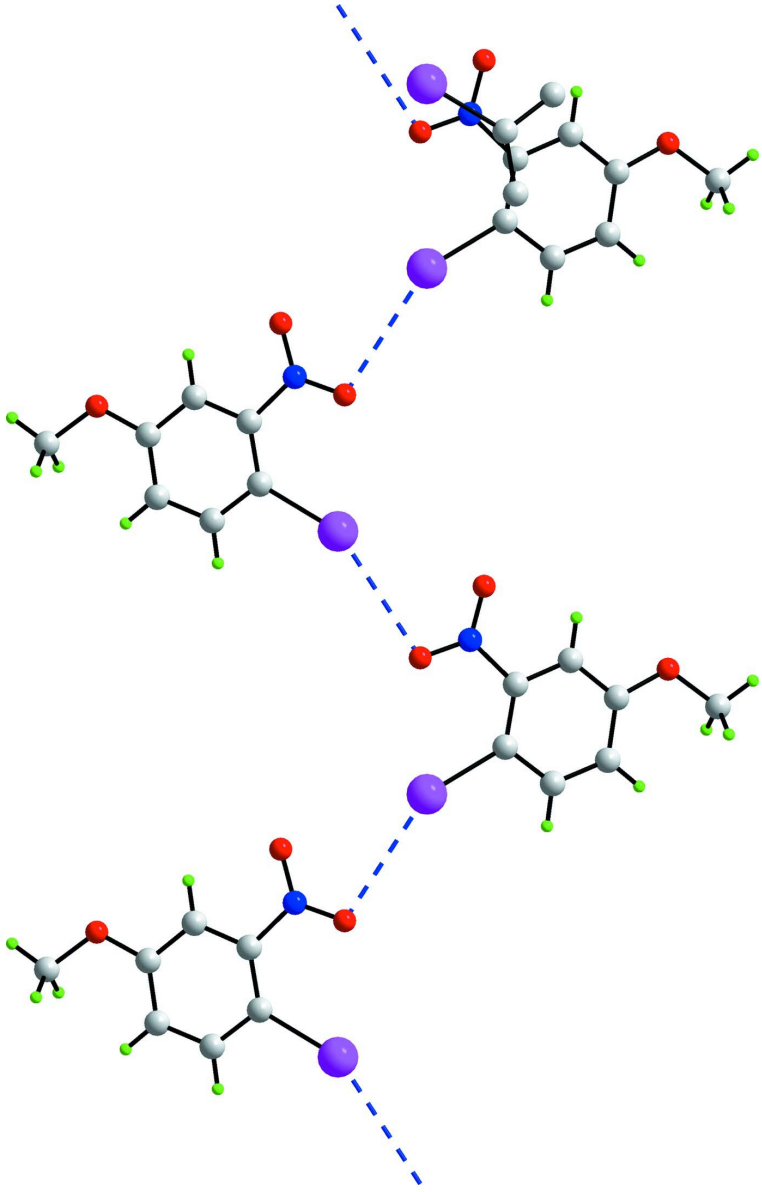

Figure 2

A view of the linear supramolecular chain along the *b* axis in (I). The I···O interactions are shown as blue dashed lines.

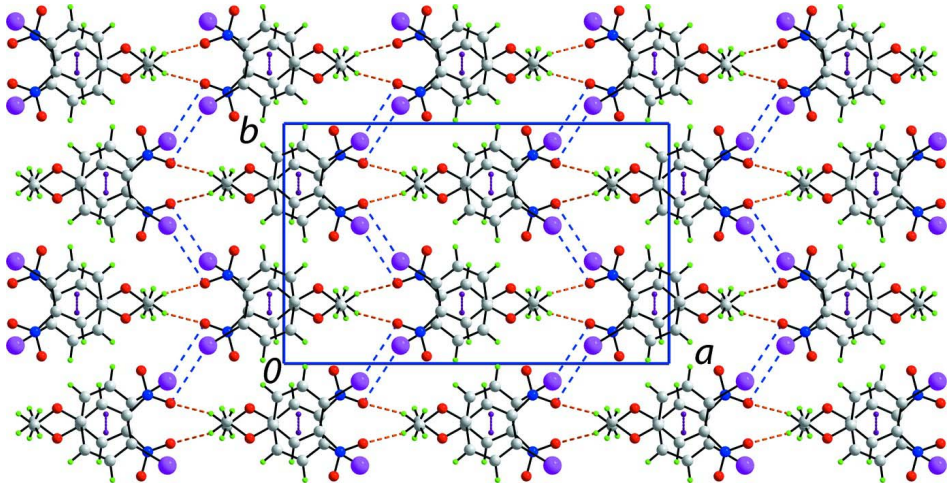

Figure 3

A view in projection down the *c* axis of the packing of supramolecular chains in (I). The I···O, C—H···O and π–π interactions are shown as blue, orange and purple dashed lines, respectively.

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Crystal data

$C_7H_6INO_3$
$M_r = 279.03$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 18.6370$ (13) Å
$b = 11.6257$ (5) Å
$c = 7.4740$ (3) Å
$V = 1619.38$ (15) Å^3
$Z = 8$
$F(000) = 1056$
$D_x = 2.289$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6840 reflections
$\theta = 3.4$–27.4°
$\mu = 3.92$ mm^{-1}
$T = 100$ K
Plate, dark-orange
0.13 × 0.09 × 0.01 mm

Data collection

Rigaku Saturn724+ (2x2 bin mode) diffractometer
Radiation source: Rotating Anode
Confocal monochromator
Detector resolution: 28.5714 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (*CrystalClear*-SM Expert; Rigaku, 2011)
$T_{min} = 0.755$, $T_{max} = 1.000$
8053 measured reflections
1841 independent reflections
1615 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.4°$
$h = -15\rightarrow24$
$k = -14\rightarrow14$
$l = -9\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.014$
$wR(F^2) = 0.039$
$S = 1.04$
1841 reflections
110 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
$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 0.9351P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.697282 (5)	0.925416 (9)	−0.003362 (13)	0.01546 (5)
O1	0.70514 (6)	0.66598 (11)	−0.03589 (18)	0.0231 (3)
O2	0.62939 (7)	0.52986 (10)	0.01236 (15)	0.0214 (3)
O3	0.41118 (6)	0.68759 (8)	0.23820 (15)	0.0148 (2)
N1	0.64607 (7)	0.63180 (12)	0.01188 (16)	0.0133 (3)
C1	0.60441 (8)	0.83551 (12)	0.0734 (2)	0.0119 (3)
C2	0.59202 (8)	0.71660 (12)	0.0719 (2)	0.0116 (3)
C3	0.52698 (8)	0.67047 (12)	0.12652 (19)	0.0122 (3)
H3	0.5199	0.5896	0.1236	0.015*
C4	0.47214 (8)	0.74173 (12)	0.1854 (2)	0.0115 (3)
C5	0.48273 (8)	0.86045 (12)	0.1870 (2)	0.0128 (3)
H5	0.4455	0.9104	0.2254	0.015*
C6	0.54812 (8)	0.90498 (12)	0.1319 (2)	0.0131 (3)
H6	0.5548	0.9860	0.1342	0.016*
C7	0.35196 (8)	0.75849 (13)	0.2943 (2)	0.0157 (3)
H7A	0.3658	0.8025	0.4007	0.024*
H7B	0.3107	0.7096	0.3228	0.024*
H7C	0.3391	0.8116	0.1977	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.01244 (8)	0.01476 (8)	0.01918 (8)	−0.00280 (4)	0.00198 (3)	0.00187 (4)
O1	0.0129 (6)	0.0209 (6)	0.0354 (7)	0.0015 (5)	0.0054 (5)	−0.0037 (5)
O2	0.0223 (7)	0.0104 (5)	0.0315 (7)	0.0033 (5)	0.0035 (5)	−0.0004 (4)
O3	0.0109 (5)	0.0121 (5)	0.0214 (6)	−0.0013 (4)	0.0029 (4)	−0.0001 (4)
N1	0.0128 (6)	0.0149 (6)	0.0124 (6)	0.0025 (5)	−0.0020 (4)	−0.0002 (5)
C1	0.0108 (7)	0.0137 (7)	0.0113 (7)	−0.0019 (6)	−0.0011 (5)	0.0018 (5)
C2	0.0122 (7)	0.0115 (6)	0.0111 (7)	0.0038 (6)	−0.0016 (5)	−0.0007 (5)
C3	0.0146 (7)	0.0091 (6)	0.0130 (7)	0.0003 (5)	−0.0024 (5)	0.0004 (5)
C4	0.0101 (6)	0.0135 (7)	0.0109 (7)	−0.0013 (5)	−0.0014 (5)	0.0007 (5)
C5	0.0116 (7)	0.0121 (6)	0.0148 (7)	0.0027 (6)	−0.0009 (5)	−0.0010 (5)
C6	0.0154 (7)	0.0097 (6)	0.0143 (7)	−0.0006 (6)	−0.0014 (6)	0.0003 (6)
C7	0.0110 (7)	0.0170 (7)	0.0191 (8)	0.0009 (6)	0.0019 (6)	0.0001 (6)

Geometric parameters (Å, º)

I1—C1	2.1017 (14)	C3—C4	1.387 (2)
O1—N1	1.2238 (18)	C3—H3	0.9500
O2—N1	1.225 (2)	C4—C5	1.394 (2)
O3—C4	1.3574 (18)	C5—C6	1.386 (2)
O3—C7	1.4398 (18)	C5—H5	0.9500
N1—C2	1.4791 (19)	C6—H6	0.9500
C1—C6	1.395 (2)	C7—H7A	0.9800
C1—C2	1.4017 (19)	C7—H7B	0.9800
C2—C3	1.387 (2)	C7—H7C	0.9800
C4—O3—C7	117.44 (11)	O3—C4—C5	125.10 (13)
O1—N1—O2	122.90 (14)	C3—C4—C5	119.30 (13)
O1—N1—C2	119.00 (13)	C6—C5—C4	119.44 (13)
O2—N1—C2	118.10 (13)	C6—C5—H5	120.3
C6—C1—C2	116.72 (13)	C4—C5—H5	120.3
C6—C1—I1	114.66 (10)	C5—C6—C1	122.56 (13)
C2—C1—I1	128.62 (11)	C5—C6—H6	118.7
C3—C2—C1	121.54 (13)	C1—C6—H6	118.7
C3—C2—N1	115.27 (12)	O3—C7—H7A	109.5
C1—C2—N1	123.19 (13)	O3—C7—H7B	109.5
C2—C3—C4	120.42 (13)	H7A—C7—H7B	109.5
C2—C3—H3	119.8	O3—C7—H7C	109.5
C4—C3—H3	119.8	H7A—C7—H7C	109.5
O3—C4—C3	115.59 (12)	H7B—C7—H7C	109.5
C6—C1—C2—C3	−0.2 (2)	C7—O3—C4—C3	−177.84 (13)
I1—C1—C2—C3	−179.68 (11)	C7—O3—C4—C5	2.3 (2)
C6—C1—C2—N1	179.41 (13)	C2—C3—C4—O3	−179.07 (13)
I1—C1—C2—N1	−0.1 (2)	C2—C3—C4—C5	0.8 (2)
O1—N1—C2—C3	−178.97 (13)	O3—C4—C5—C6	179.05 (14)
O2—N1—C2—C3	1.04 (19)	C3—C4—C5—C6	−0.9 (2)
O1—N1—C2—C1	1.5 (2)	C4—C5—C6—C1	0.4 (2)
O2—N1—C2—C1	−178.54 (14)	C2—C1—C6—C5	0.1 (2)
C1—C2—C3—C4	−0.3 (2)	I1—C1—C6—C5	179.73 (12)
N1—C2—C3—C4	−179.93 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···O1^{i}	0.98	2.58	3.4503 (19)	148

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