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2,4-Diiodo-3-nitroanisole

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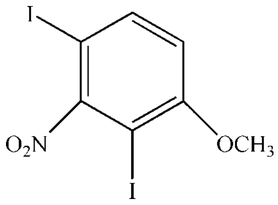
Key indicators: single-crystal X-ray study; *T* = 296 K; mean σ(C–C) = 0.007 Å; *R* factor = 0.027; *wR* factor = 0.065; data-to-parameter ratio = 15.6.

In the title compound (systematic name: 1,3-diiodo-4-methoxy-2-nitrobenzene), $C_7H_5I_2NO_3$, the dihedral angle between the benzene ring and the nitro group is 88.0 (3)°, and the methyl group lies almost in the same plane as the ring [deviation = 0.034 (6) Å]. In the crystal, aromatic π–π stacking occurs between inversion-related rings [centroid–centroid separation = 3.865 (3) Å and slippage = 0.642 Å]. A possible weak C—I$\cdots\pi$ interaction occurs [I$\cdots\pi$ = 3.701 (2) Å and C—I$\cdots\pi$ = 130.18 (13)°], but there are no significant intermolecular I$\cdots$I contacts.

Related literature

For the crystal structures of isomers of the title compound, see: Garden *et al.* (2002, 2004).



Experimental

Crystal data

$C_7H_5I_2NO_3$
M_r = 404.92
Monoclinic, $P2_1/c$
a = 9.264 (2) Å
b = 8.756 (2) Å
c = 13.549 (3) Å
β = 108.835 (2)°
V = 1040.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.02 mm^{-1}
T = 296 K
0.36 × 0.33 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min}$ = 0.220, $T_{\max}$ = 0.486
7459 measured reflections
1937 independent reflections
1712 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.065
S = 1.00
2689 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 1.06 e Å^{-3}
$\Delta\rho_{\min}$ = −1.25 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; 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: HB6662).

References

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supporting information

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2,4-Diiodo-3-nitroanisole

Xianfei Li, Lei Cao, Chuansheng Ruan, Baoming Ji and Le Zhou

S1. Comment

We report here the molecular and supramolecular structures of the title compound, (I), which is isomeric with 2,6-diiodo-4-nitroanisole (Garden *etal.*, 2002) and 2,4-diiodo-6-nitroanisole (Garden *et al.*, 2003). The changed position of iodo, nitro and methoxy may lead to different interactions such as iodo-nitro interactions, and aromatic $\pi\cdots\pi$ stacking interactions.

The asymmetric unit of the title compound comprise a whole molecule of 2,4-diiodo-3-nitroanisole (Fig. 1). Atoms I1, I2, C7 and O3 are almost coplanar with the benzene ring. On the contrary, the plane defined by the nitro group is almost perpendicular to the plane of the aromatic ring and form a dihedral angle of 88.0 (4)°. In contrast with 2,6-diiodo-4-nitroanisole (Garden *et al.*, 2002) and 2,4-diiodo-6-nitroanisole (Garden *et al.*, 2003), there is no iodo-nitro interaction in the compound, each molecule link three others by $\pi\cdots\pi$ stacking interaction and C—I$\cdots\pi$ interaction, leading to the formation of a sheet (Fig. 2). The aryl ring planes (centroid *Cg*1) of two molecules are parallel, show a $\pi\cdots\pi$ stacking interaction $Cg1\cdots Cg1^{viii}$ [symmetry codes: (viii) 1 - *x*, 2 - *y*, -*z*), and the centroid distance is 3.865 (3) Å. C—I$\cdots\pi$ interaction also occurs in the compound, I1 aim to the phenyl ring [$I1\cdots Cg1^{ix}$ 3.701 (2) Å, C2—$I1\cdots Cg1^{ix}$ 130.1 (1)°; symmetry code: (ix) 1 - *x*, 1/2 + *y*, 1/2 - *z*].

S2. Experimental

The title compound was obtained from 2-iodo-3-nitrophenol, a solution of 2-iodo-3-nitrophenol (2 mmol) in acetone (20 ml) was added K_2CO_3 (5 mmol). The mixture was stirred at room temperature for 30 min, then CH_3I (5 mmol) was added at once. The resulting solution was then stirred at 343 K for 3 h. The addition of ice (20 g) prompted the precipitation of the title compound, which was collected by filtration and crystallized from ethyl acetate as yellow blocks (yield 90%, m.p. 406–408 K).

S3. Refinement

All H atoms were located from difference maps and were treated as riding atoms with C—H distances of 0.93 Å (aromatic) and 0.96 Å (methyl).

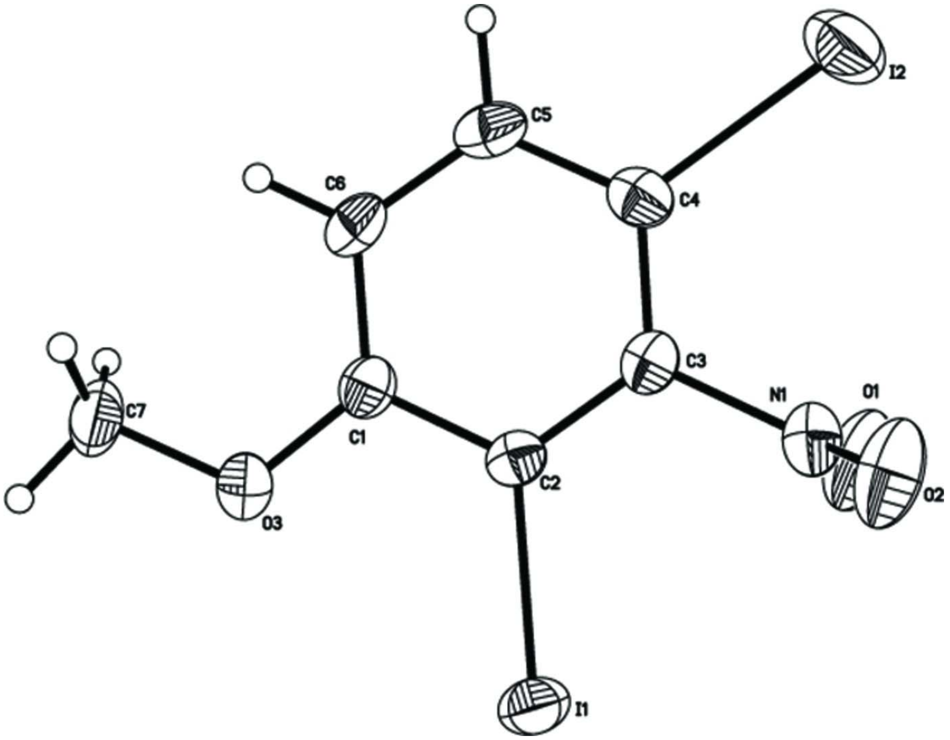

Figure 1

The moleuclar sturcture of (I) with displacement ellipsoids drawn at the 30% probability level.

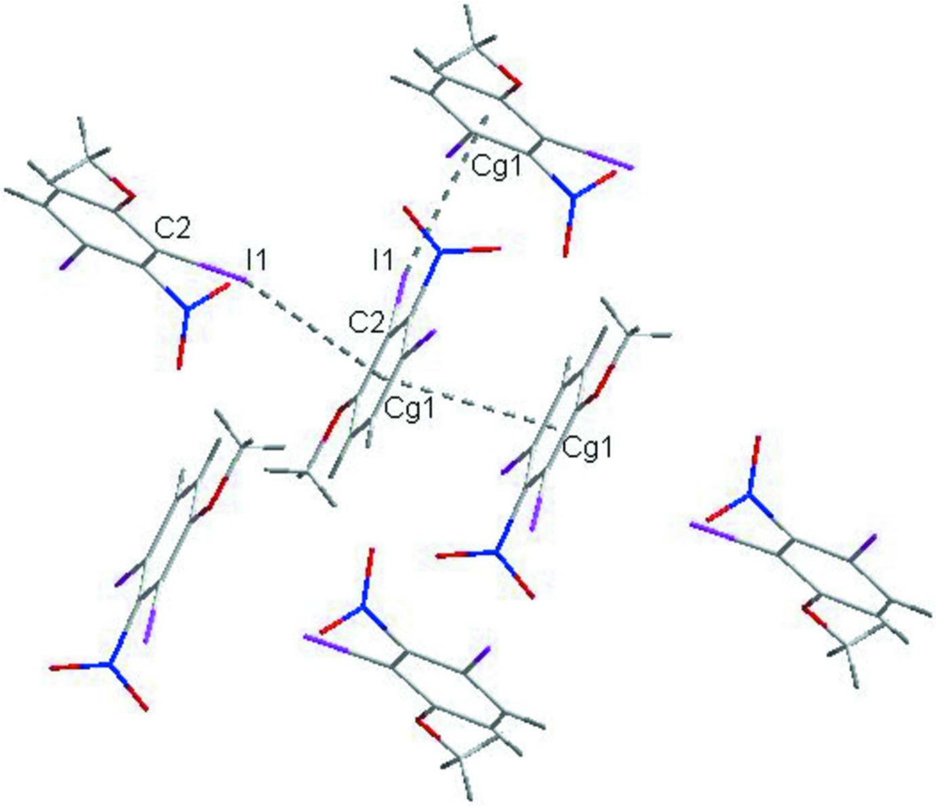

Figure 2

Part of the crystal structure of the title compound, showing formation of a sheet built from $\pi\cdots\pi$ stacking interactions, and C—I$\cdots\pi$ interactions.

1,3-diiodo-4-methoxy-2-nitrobenzene

Crystal data

$C_7H_5I_2NO_3$	$F(000) = 736$
$M_r = 404.92$	$D_x = 2.586$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.264$ (2) Å	Cell parameters from 3452 reflections
$b = 8.756$ (2) Å	$\theta = 2.8$–27.0°
$c = 13.549$ (3) Å	$\mu = 6.02$ mm^{-1}
$\beta = 108.835$ (2)°	$T = 296$ K
$V = 1040.2$ (4) Å^3	Block, yellow
$Z = 4$	0.36 × 0.33 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer	7459 measured reflections
Radiation source: fine-focus sealed tube	1937 independent reflections
Graphite monochromator	1712 reflections with $I > 2\sigma(I)$
phi and ω scans	$R_{\rm int} = 0.018$
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)	$\theta_{\rm max} = 25.5°$, $\theta_{\rm min} = 2.3°$
$T_{\rm min} = 0.220$, $T_{\rm max} = 0.486$	$h = -11\rightarrow11$
	$k = -10\rightarrow10$
	$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.065$
$S = 1.00$
2689 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0264P)^2 + 3.0364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.06$ e Å^{-3}
$\Delta\rho_{min} = -1.25$ e Å^{-3}

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 > 2sigma(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}
C1	0.3646 (5)	0.8010 (5)	0.0510 (3)	0.0431 (10)
C2	0.4466 (5)	0.8963 (5)	0.1324 (3)	0.0391 (9)
C3	0.6036 (5)	0.9031 (5)	0.1575 (3)	0.0405 (9)
C4	0.6821 (5)	0.8208 (6)	0.1041 (3)	0.0465 (11)
C5	0.5990 (6)	0.7285 (6)	0.0233 (3)	0.0519 (12)
H5	0.6490	0.6724	−0.0141	0.062*
C6	0.4412 (6)	0.7182 (6)	−0.0030 (3)	0.0488 (11)
H6	0.3870	0.6549	−0.0574	0.059*
C7	0.1252 (6)	0.7011 (7)	−0.0503 (4)	0.0659 (15)
H7A	0.1457	0.7238	−0.1138	0.099*
H7B	0.0185	0.7152	−0.0607	0.099*
H7C	0.1529	0.5971	−0.0306	0.099*
N1	0.6918 (5)	1.0000 (5)	0.2475 (3)	0.0507 (10)
O1	0.7319 (6)	0.9427 (5)	0.3316 (3)	0.0860 (14)
O2	0.7193 (5)	1.1291 (5)	0.2286 (3)	0.0831 (13)
O3	0.2116 (4)	0.8002 (4)	0.0299 (2)	0.0571 (9)
I1	0.33063 (4)	1.02858 (4)	0.21050 (3)	0.05654 (12)
I2	0.91883 (4)	0.82973 (6)	0.14145 (3)	0.08415 (18)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.048 (2)	0.046 (2)	0.034 (2)	−0.002 (2)	0.0103 (18)	0.0012 (19)
C2	0.047 (2)	0.037 (2)	0.034 (2)	0.0034 (19)	0.0143 (18)	0.0028 (18)
C3	0.048 (2)	0.039 (2)	0.032 (2)	−0.0018 (19)	0.0096 (18)	0.0047 (18)
C4	0.044 (2)	0.056 (3)	0.041 (2)	0.007 (2)	0.0163 (19)	0.009 (2)
C5	0.067 (3)	0.054 (3)	0.040 (2)	0.009 (2)	0.024 (2)	−0.002 (2)
C6	0.062 (3)	0.049 (3)	0.033 (2)	0.000 (2)	0.011 (2)	−0.0057 (19)
C7	0.054 (3)	0.093 (4)	0.046 (3)	−0.021 (3)	0.009 (2)	−0.017 (3)
N1	0.050 (2)	0.056 (3)	0.043 (2)	−0.0101 (19)	0.0105 (18)	0.0048 (18)
O1	0.119 (4)	0.081 (3)	0.039 (2)	−0.024 (3)	−0.001 (2)	0.005 (2)
O2	0.105 (3)	0.064 (3)	0.065 (2)	−0.034 (2)	0.007 (2)	0.000 (2)
O3	0.0453 (18)	0.070 (2)	0.0505 (19)	−0.0050 (16)	0.0083 (15)	−0.0121 (17)
I1	0.0635 (2)	0.0536 (2)	0.0582 (2)	0.00317 (16)	0.02756 (16)	−0.01141 (15)
I2	0.0470 (2)	0.1373 (4)	0.0684 (3)	0.0114 (2)	0.01898 (18)	−0.0001 (2)

Geometric parameters (Å, º)

C1—O3	1.353 (5)	C5—C6	1.391 (7)
C1—C6	1.378 (6)	C5—H5	0.9300
C1—C2	1.396 (6)	C6—H6	0.9300
C2—C3	1.384 (6)	C7—O3	1.419 (6)
C2—I1	2.086 (4)	C7—H7A	0.9600
C3—C4	1.382 (6)	C7—H7B	0.9600
C3—N1	1.493 (6)	C7—H7C	0.9600
C4—C5	1.379 (7)	N1—O1	1.189 (5)
C4—I2	2.087 (5)	N1—O2	1.205 (5)
O3—C1—C6	124.7 (4)	C6—C5—H5	119.6
O3—C1—C2	115.9 (4)	C1—C6—C5	120.6 (4)
C6—C1—C2	119.4 (4)	C1—C6—H6	119.7
C3—C2—C1	118.7 (4)	C5—C6—H6	119.7
C3—C2—I1	121.6 (3)	O3—C7—H7A	109.5
C1—C2—I1	119.6 (3)	O3—C7—H7B	109.5
C4—C3—C2	122.5 (4)	H7A—C7—H7B	109.5
C4—C3—N1	118.9 (4)	O3—C7—H7C	109.5
C2—C3—N1	118.6 (4)	H7A—C7—H7C	109.5
C5—C4—C3	117.9 (4)	H7B—C7—H7C	109.5
C5—C4—I2	119.2 (3)	O1—N1—O2	125.2 (4)
C3—C4—I2	122.9 (3)	O1—N1—C3	117.6 (4)
C4—C5—C6	120.8 (4)	O2—N1—C3	117.2 (4)
C4—C5—H5	119.6	C1—O3—C7	117.3 (4)
O3—C1—C2—C3	−179.6 (4)	C3—C4—C5—C6	−0.4 (7)
C6—C1—C2—C3	−1.0 (6)	I2—C4—C5—C6	179.2 (4)
O3—C1—C2—I1	−1.1 (5)	O3—C1—C6—C5	178.8 (4)
C6—C1—C2—I1	177.5 (3)	C2—C1—C6—C5	0.3 (7)

C1—C2—C3—C4	1.0 (6)	C4—C5—C6—C1	0.4 (7)
I1—C2—C3—C4	−177.5 (3)	C4—C3—N1—O1	−90.3 (6)
C1—C2—C3—N1	−177.4 (4)	C2—C3—N1—O1	88.1 (6)
I1—C2—C3—N1	4.2 (5)	C4—C3—N1—O2	88.1 (6)
C2—C3—C4—C5	−0.3 (7)	C2—C3—N1—O2	−93.5 (5)
N1—C3—C4—C5	178.0 (4)	C6—C1—O3—C7	3.3 (7)
C2—C3—C4—I2	−179.9 (3)	C2—C1—O3—C7	−178.2 (4)
N1—C3—C4—I2	−1.5 (6)		