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4-Chloro-1-iodo-2-nitrobenzene

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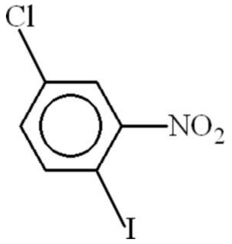
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.023; wR factor = 0.051; data-to-parameter ratio = 16.9.

In the molecule of the title compound, $C_6H_3ClINO_2$, the nitro group is disordered over two sites with occupancies of 0.506 (6) and 0.494 (6). The dihedral angles between the benzene ring and the two disordered components of the nitro group are 29.0 (2) and 51.0 (3)°. The disordering avoids short O$\cdots$O intermolecular contacts in the crystal.

Related literature

For background, see: Arshad *et al.* (2008, 2009). For related structures, see: Meriles *et al.* (1999).



Experimental

Crystal data

$C_6H_3ClINO_2$
M_r = 283.44
Monoclinic, $P2_1/c$
a = 4.1583 (2) Å
b = 14.5213 (7) Å
c = 13.7990 (6) Å
β = 93.361 (2)°
V = 831.81 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.12 mm^{-1}
T = 296 K
0.26 × 0.12 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.554, T_{max} = 0.664
9922 measured reflections
2157 independent reflections
1684 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.051
S = 1.02
2157 reflections
128 parameters
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2905).

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supplementary materials

Acta Cryst. (2009). E**65**, o535   [**doi:10.1107/S1600536809004930**]

4-Chloro-1-iodo-2-nitrobenzene

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Comment

The title compound (I), (Fig 1), has been prepared as an intermediate for the synthesis of sulfonamides (Arshad *et al.*, 2009) and benzothiazines (Arshad *et al.*, 2008). The crystal structures of *p*-chlorobromobenzene and *p*-chloroiodobenzene (Meriles *et al.*, 1999) have been published.

In (I), the iodo and chloro moiety is in plane with the benzene ring. The nitro group is disordered over two sites with nearly equal occupancy ratio of 0.506 (6):0.494 (6). The behaviour of nitro groups is very different from each other. The distance between the symmetry related O-atoms of nitro groups have nearly equal value of 2.110 (9) Å. One group [O1B···O2B (*x* - 1, *y*, *z*)] interact in *trans* form while the other [O2A···O2A (-*x*, -*y*, -*z*)] remains in *cis* form. The dihedral angle between the benzene ring and two nitro groups is 29.03 (23)° and 51.03 (31)°, respectively. The dihedral angle between the disordered nitro groups is 79.76 (37)°. There does not exist any classical H-bond or any kind of π-interaction.

Experimental

4-Chloro-2-nitroaniline (2 g, 0.0116 mol) was dissolved in conc. HCl (10 ml) in a flask. The mixture was put in ice to attain 273-78 K. $NaNO_2$ (0.96 g, 0.14 mol) was added in the solution under stirring. After 5 minutes the solution of KI (2.17 g, 0.0134 mol) was added and stirred for 10 minutes at the same temperature i.e 273-278 K. Then ice was removed and allowed to stirr till the room temperature was attained. After this mixture was heated to remove the nitrogen and reduce the volume. The resulting mixture was cooled in ice overnight. The obtained precipitate was filtered and washed with distilled water. The dried filterate was recrystalized in dicloromethane and methanol to obtain crystals of (I) as yellow needles.

Refinement

The O atoms of the nitro group are disordered over two sets of sites in a 0.506 (6):0.496 (6) ratio. The H atoms were located in a difference map and their positions were refined with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

Fig. 1. View of the molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level. H-atoms are shown by spheres of arbitrary radius.

4-Chloro-1-iodo-2-nitrobenzene

Crystal data

$C_6H_3ClINO_2$

$M_r = 283.44$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 4.1583$ (2) Å

$b = 14.5213$ (7) Å

$c = 13.7990$ (6) Å

$\beta = 93.361$ (2)°

$V = 831.81$ (7) Å^3

$Z = 4$

$F_{000} = 528$

$D_x = 2.263$ Mg m^{-3}

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

Cell parameters from 2157 reflections

$\theta = 2.8$–28.7°

$\mu = 4.12$ mm^{-1}

$T = 296$ K

Needle, yellow

0.26 × 0.12 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 7.40 pixels mm^{-1}

$T = 296$ K

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.554$, $T_{\max} = 0.664$

9922 measured reflections

2157 independent reflections

1684 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 28.7$°

$\theta_{\min} = 2.8$°

$h = -5 \rightarrow 3$

$k = -18 \rightarrow 19$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.023$

$wR(F^2) = 0.051$

$S = 1.02$

2157 reflections

128 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Only H-atom coordinates refined

$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2 + 0.626P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.66$ e Å^{-3}

$\Delta\rho_{\min} = -0.60$ e Å^{-3}

Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > \sigma(F^2)$ 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*	$U_{\rm iso}$*/$U_{\rm eq}$	Occ. (<1)
I1	−0.08606 (5)	0.03022 (1)	0.36564 (1)	0.0520 (1)	
Cl1	0.5423 (2)	0.29672 (6)	0.03919 (7)	0.0739 (3)	
O1A	0.1842 (14)	−0.0814 (3)	0.2012 (4)	0.0693 (19)	0.506 (6)
O2A	0.1343 (15)	−0.0393 (3)	0.0522 (4)	0.077 (2)	0.506 (6)
N1	0.1689 (7)	−0.02093 (18)	0.14257 (19)	0.0521 (9)	
C1	0.2016 (6)	0.07685 (18)	0.16917 (19)	0.0395 (8)	
C2	0.1064 (6)	0.10984 (18)	0.25738 (18)	0.0394 (8)	
C3	0.1504 (8)	0.2032 (2)	0.2764 (2)	0.0525 (10)	
C4	0.2833 (8)	0.2599 (2)	0.2098 (3)	0.0554 (11)	
C5	0.3754 (7)	0.2250 (2)	0.1233 (2)	0.0487 (9)	
C6	0.3366 (7)	0.1329 (2)	0.1020 (2)	0.0460 (9)	
O1B	−0.0923 (15)	−0.0561 (3)	0.1528 (4)	0.0721 (19)	0.494 (6)
O2B	0.4074 (16)	−0.0577 (3)	0.1171 (4)	0.082 (3)	0.494 (6)
H6	0.392 (7)	0.109 (2)	0.045 (2)	0.0552*	
H3	0.086 (8)	0.228 (2)	0.336 (2)	0.0630*	
H4	0.325 (8)	0.321 (2)	0.223 (2)	0.0666*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0531 (1)	0.0648 (1)	0.0392 (1)	0.0006 (1)	0.0124 (1)	0.0053 (1)
Cl1	0.0847 (6)	0.0649 (5)	0.0724 (6)	−0.0142 (5)	0.0073 (5)	0.0265 (4)
O1A	0.098 (4)	0.038 (3)	0.074 (3)	0.000 (2)	0.024 (3)	0.007 (2)
O2A	0.109 (5)	0.074 (3)	0.050 (3)	−0.023 (3)	0.013 (3)	−0.024 (2)
N1	0.0660 (16)	0.0438 (15)	0.0478 (15)	−0.0018 (12)	0.0137 (13)	−0.0032 (12)
C1	0.0427 (13)	0.0359 (14)	0.0398 (14)	0.0018 (11)	0.0015 (11)	−0.0004 (11)
C2	0.0406 (13)	0.0450 (15)	0.0325 (13)	0.0061 (11)	0.0010 (10)	0.0014 (11)
C3	0.0650 (18)	0.0492 (18)	0.0432 (16)	0.0097 (14)	0.0029 (14)	−0.0066 (14)
C4	0.070 (2)	0.0375 (16)	0.058 (2)	0.0011 (14)	−0.0022 (16)	−0.0001 (15)
C5	0.0504 (15)	0.0462 (17)	0.0490 (17)	−0.0004 (12)	−0.0010 (13)	0.0131 (14)
C6	0.0508 (15)	0.0496 (17)	0.0382 (15)	0.0019 (12)	0.0072 (12)	0.0004 (13)
O1B	0.096 (4)	0.057 (3)	0.066 (3)	−0.027 (3)	0.027 (3)	−0.012 (2)
O2B	0.100 (5)	0.055 (3)	0.095 (5)	0.019 (3)	0.037 (4)	−0.017 (3)

Geometric parameters (Å, °)

I1—C2	2.086 (3)	C1—C2	1.387 (4)
Cl1—C5	1.734 (3)	C2—C3	1.391 (4)
O1A—N1	1.193 (6)	C3—C4	1.374 (5)
O1B—N1	1.216 (7)	C4—C5	1.372 (5)
O2A—N1	1.275 (6)	C5—C6	1.377 (4)
O2B—N1	1.197 (7)	C3—H3	0.95 (3)
N1—C1	1.471 (4)	C4—H4	0.92 (3)
C1—C6	1.378 (4)	C6—H6	0.90 (3)
O2B···O1B^{i}	2.110 (9)		
O1A—N1—O2A	120.5 (4)	C2—C3—C4	120.8 (3)
O1A—N1—C1	122.7 (3)	C3—C4—C5	120.3 (3)
O2A—N1—C1	116.7 (3)	Cl1—C5—C4	120.2 (2)
O1B—N1—C1	116.5 (3)	Cl1—C5—C6	119.1 (2)
O2B—N1—C1	116.0 (3)	C4—C5—C6	120.6 (3)
O1B—N1—O2B	127.4 (4)	C1—C6—C5	118.5 (3)
N1—C1—C2	121.7 (2)	C2—C3—H3	119.5 (18)
N1—C1—C6	116.0 (2)	C4—C3—H3	119.8 (18)
C2—C1—C6	122.3 (2)	C3—C4—H4	121.6 (18)
I1—C2—C1	125.33 (19)	C5—C4—H4	118.0 (18)
I1—C2—C3	117.19 (19)	C1—C6—H6	119.6 (19)
C1—C2—C3	117.5 (2)	C5—C6—H6	121.8 (19)
O1A—N1—C1—C2	−30.2 (5)	C2—C1—C6—C5	−0.5 (4)
O1A—N1—C1—C6	148.8 (4)	I1—C2—C3—C4	178.4 (2)
O2A—N1—C1—C2	152.2 (4)	C1—C2—C3—C4	0.0 (4)
O2A—N1—C1—C6	−28.8 (5)	C2—C3—C4—C5	−0.1 (5)
N1—C1—C2—I1	1.0 (4)	C3—C4—C5—Cl1	179.9 (3)
N1—C1—C2—C3	179.3 (3)	C3—C4—C5—C6	−0.1 (5)
C6—C1—C2—I1	−177.9 (2)	Cl1—C5—C6—C1	−179.5 (2)
C6—C1—C2—C3	0.3 (4)	C4—C5—C6—C1	0.4 (4)
N1—C1—C6—C5	−179.5 (3)		

Symmetry codes: (i) $x+1, y, z$.

Fig. 1