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4-Iodobenzohydrazide

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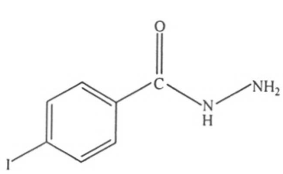
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.006 Å; R factor = 0.029; wR factor = 0.106; data-to-parameter ratio = 19.0.

In the structure of the title compound, $C_7H_7IN_2O$, the hydrazide group is inclined at 13.3 (3)° with respect to the benzene ring. The structure is stabilized by intermolecular N—H···N and N—H···O hydrogen bonds involving the hydrazide group, resulting in six- and ten-membered rings with $R_2^2(6)$ and $R_2^2(10)$ graph-set notations, respectively.

Related literature

For related structures, see: Kallel *et al.* (1992); Saraogi *et al.* (2002); Ashiq, Jamal *et al.* (2008). For related literature, see: Ara *et al.* (2007); Ashiq, Ara *et al.* (2008); Bernstein *et al.* (1994).



Experimental

Crystal data

$C_7H_7IN_2O$
M_r = 262.05
Monoclinic, $C2/c$
a = 28.4394 (18) Å
b = 4.4514 (3) Å
c = 13.3216 (9) Å
β = 94.292 (2)°
V = 1681.72 (19) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.76 mm^{-1}
T = 296 (2) K
0.12 × 0.08 × 0.06 mm

Data collection

Bruker KappaAPEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.581, T_{max} = 0.806
9236 measured reflections
2069 independent reflections
1645 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.106
S = 1.05
2069 reflections
109 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −1.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2^{i}	0.857 (10)	2.19 (3)	2.964 (5)	151 (5)
N2—H2A···O1^{ii}	0.862 (10)	2.240 (14)	3.094 (5)	170 (5)
C3—H3···O1^{iii}	0.93	2.56	3.257 (5)	132

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x, y, -z+\frac{1}{2}$; (iii) $x, -y+1, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2007); 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); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2109).

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supplementary materials

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4-Iodobenzohydrazide

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Comment

The title compound and its oxovanadium(IV) complex were investigated for their α-glucosidase inhibitory and urease activities. Free hydrazide ligand was found to be inactive, whereas its oxovanadium(IV) complex was found to be a potent inhibitor of α-glucosidase (Ashiq, Ara *et al.*, 2008) and urease (Ara *et al.*, 2007). Continuing our studies on the enzyme inhibition behavior of the title compound, (I), and to investigate the change in its activity due to complexation with vanadium center, we have synthesized (I) and report its crystal structure in this paper. The structures of benzhydrazide (Kallel *et al.*, 1992), *para*-chloro (Saraogi *et al.*, 2002) and *para*-bromo (Ashiq, Jamal *et al.*, 2008) analogues of (I) have already been reported.

The molecule of the title compound (Fig. 1) is far from planar as is evident from the dihedral angle of 13.3 (3)° between the mean-planes of the phenyl ring (C1-C6) and the hydrazide moiety (N1/N2/O1/C7). The bond distances and bond angles in (I) are similar to the corersponding distances and angles reported in the structures quoted above. The molecules of (I) are involved in two types of hydrogen bonds involving hydrazide moiety. On one hand, the molecules lying about inversion centers form six membered rings *via* N1—H1A···N2^{i} hydrogen bonding. On the other hand, the molecules related by c-glide form ten membered rings *via* N2—H2A···O1^{ii}; detail of the hydrogen bonding have been presented in Table 1 and depicted in Fig. 2. The six and ten membered rings represent $R_2^2(6)$ and $R_2^2(10)$ graph set patterns, respectively (Bernstein *et al.*, 1994).

Experimental

All reagent-grade chemicals were obtained from Aldrich and Sigma Chemical companies and were used without further purification. To a solution of ethyl-4-iodobenzoate (5.5 g, 20 mmol) in 75 ml ethanol, hydrazine hydrate (5.0 ml, 100 mmol) was added. The mixture was refluxed for 5 h and a solid was obtained upon removal of the solvent by rotary evaporation. The resulting solid was washed with hexane to afford 4-iodobenzohydrazide (yield 84%).

Refinement

H-atoms bonded to N-atoms were located from a difference map and were included in the refinement at those positions (using DFIX command with N—H = 0.86 (1) Å) while the aryl H-atoms were positioned geometrically in a riding mode, with C—H = 0.93 Å; for all H-atoms, U_{iso} = 1.2 times U_{eq} of the parent atoms.

Figures

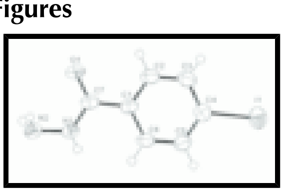

Fig. 1. *ORTEP* plot of the title compound with the ellipsoids drawn at the 50% probability level.

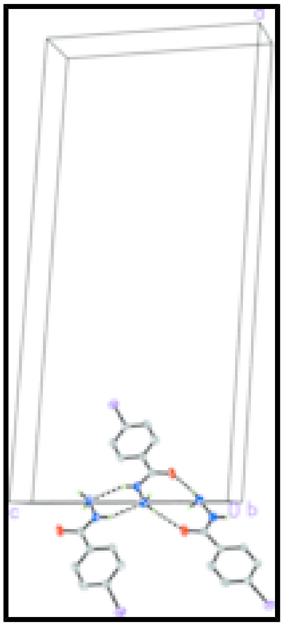

Fig. 2. The hydrogen bonding patterns of (I) represented by dashed lines in the unit cell; H-atoms not involved in H-bonds have been excluded.

4-Iodobenzohydrazide

Crystal data

$C_7H_7IN_2O$	F_{000} = 992
M_r = 262.05	D_x = 2.072 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3495 reflections
a = 28.4394 (18) Å	θ = 1.4–28.3°
b = 4.4514 (3) Å	μ = 3.76 mm^{-1}
c = 13.3216 (9) Å	*T* = 296 (2) K
β = 94.292 (2)°	Needle, colorless
V = 1681.72 (19) Å^3	0.12 × 0.08 × 0.06 mm
Z = 8	

Data collection

Bruker KappaAPEXII CCD diffractometer	2069 independent reflections
Radiation source: fine-focus sealed tube	1645 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
T = 296(2) K	θ_{max} = 28.3°
ω scans	θ_{min} = 1.4°
Absorption correction: multi-scan (SADABS; Bruker, 2005)	*h* = −37→37
T_{min} = 0.581, T_{max} = 0.806	*k* = −5→5
9236 measured reflections	*l* = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 5.77P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
2069 reflections	$\Delta\rho_{max} = 0.55$ e Å^{-3}
109 parameters	$\Delta\rho_{min} = -1.32$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > \sigma(F^2)$ 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_{iso}*/U_{eq}
I1	0.215538 (11)	1.09655 (7)	0.64904 (2)	0.05339 (15)
O1	0.05919 (14)	0.3811 (8)	0.2873 (2)	0.0582 (10)
N1	0.03174 (13)	0.1851 (9)	0.4262 (2)	0.0404 (8)
H1A	0.0321 (18)	0.170 (11)	0.4905 (9)	0.049*
N2	−0.00239 (14)	0.0017 (10)	0.3743 (2)	0.0408 (8)
H2A	−0.0195 (16)	0.119 (9)	0.335 (3)	0.049*
H2B	0.0127 (17)	−0.126 (9)	0.341 (3)	0.049*
C1	0.09627 (14)	0.5363 (10)	0.4445 (3)	0.0358 (8)
C2	0.09382 (15)	0.5693 (10)	0.5484 (3)	0.0403 (9)
H2	0.0695	0.4777	0.5799	0.048*
C3	0.12697 (15)	0.7356 (11)	0.6046 (3)	0.0441 (10)
H3	0.1244	0.7611	0.6733	0.053*
C4	0.16380 (15)	0.8638 (9)	0.5594 (3)	0.0397 (9)
C5	0.16696 (17)	0.8370 (11)	0.4566 (3)	0.0489 (11)
H5	0.1918	0.9256	0.4260	0.059*
C6	0.13267 (18)	0.6768 (12)	0.3998 (3)	0.0489 (11)

H6	0.1342	0.6637	0.3305	0.059*
C7	0.06129 (16)	0.3608 (9)	0.3801 (3)	0.0375 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0461 (2)	0.0498 (2)	0.0626 (2)	−0.00097 (13)	−0.00736 (14)	0.00125 (13)
O1	0.069 (2)	0.080 (3)	0.0273 (13)	−0.0221 (19)	0.0124 (14)	−0.0003 (14)
N1	0.0469 (19)	0.0465 (19)	0.0277 (14)	−0.0046 (17)	0.0010 (13)	0.0067 (14)
N2	0.050 (2)	0.0425 (19)	0.0302 (15)	0.0003 (17)	0.0039 (14)	0.0051 (15)
C1	0.0384 (19)	0.038 (2)	0.0317 (17)	0.0053 (17)	0.0080 (14)	0.0015 (16)
C2	0.039 (2)	0.051 (3)	0.0317 (17)	−0.0001 (18)	0.0109 (15)	0.0041 (17)
C3	0.043 (2)	0.054 (3)	0.0352 (18)	0.001 (2)	0.0049 (16)	0.0000 (19)
C4	0.036 (2)	0.039 (2)	0.044 (2)	0.0035 (16)	−0.0004 (16)	0.0018 (17)
C5	0.048 (2)	0.053 (3)	0.047 (2)	−0.008 (2)	0.0168 (19)	0.002 (2)
C6	0.055 (3)	0.056 (3)	0.038 (2)	−0.004 (2)	0.0156 (19)	0.001 (2)
C7	0.042 (2)	0.041 (2)	0.0309 (17)	0.0060 (17)	0.0088 (15)	0.0037 (15)

Geometric parameters (Å, °)

I1—C4	2.098 (4)	C1—C7	1.486 (6)
O1—C7	1.237 (5)	C2—C3	1.375 (6)
N1—C7	1.332 (5)	C2—H2	0.9300
N1—N2	1.410 (6)	C3—C4	1.371 (6)
N1—H1A	0.857 (10)	C3—H3	0.9300
N2—H2A	0.862 (10)	C4—C5	1.384 (6)
N2—H2B	0.860 (10)	C5—C6	1.386 (7)
C1—C6	1.382 (6)	C5—H5	0.9300
C1—C2	1.398 (5)	C6—H6	0.9300
C7—N1—N2	123.3 (3)	C2—C3—H3	120.0
C7—N1—H1A	123 (3)	C3—C4—C5	120.5 (4)
N2—N1—H1A	114 (3)	C3—C4—I1	118.8 (3)
N1—N2—H2A	106 (3)	C5—C4—I1	120.7 (3)
N1—N2—H2B	107 (4)	C4—C5—C6	119.3 (4)
H2A—N2—H2B	112 (5)	C4—C5—H5	120.4
C6—C1—C2	118.3 (4)	C6—C5—H5	120.4
C6—C1—C7	118.6 (3)	C1—C6—C5	121.1 (4)
C2—C1—C7	123.1 (4)	C1—C6—H6	119.5
C3—C2—C1	120.8 (4)	C5—C6—H6	119.5
C3—C2—H2	119.6	O1—C7—N1	121.3 (4)
C1—C2—H2	119.6	O1—C7—C1	121.3 (4)
C4—C3—C2	120.0 (4)	N1—C7—C1	117.4 (3)
C4—C3—H3	120.0		
C6—C1—C2—C3	−0.5 (7)	C7—C1—C6—C5	−178.3 (4)
C7—C1—C2—C3	−179.6 (4)	C4—C5—C6—C1	−1.9 (8)
C1—C2—C3—C4	−2.0 (7)	N2—N1—C7—O1	2.7 (7)
C2—C3—C4—C5	2.5 (7)	N2—N1—C7—C1	−178.5 (4)
C2—C3—C4—I1	−176.8 (3)	C6—C1—C7—O1	−12.8 (6)

C3—C4—C5—C6	−0.6 (7)	C2—C1—C7—O1	166.3 (4)
I1—C4—C5—C6	178.8 (4)	C6—C1—C7—N1	168.4 (4)
C2—C1—C6—C5	2.4 (7)	C2—C1—C7—N1	−12.4 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···N2^{i}	0.857 (10)	2.19 (3)	2.964 (5)	151 (5)
N2—H2A···O1^{ii}	0.862 (10)	2.240 (14)	3.094 (5)	170 (5)
C3—H3···O1^{iii}	0.93	2.56	3.257 (5)	132

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*, *y*, −*z*+1/2; (iii) *x*, −*y*+1, *z*+1/2.

Fig. 1

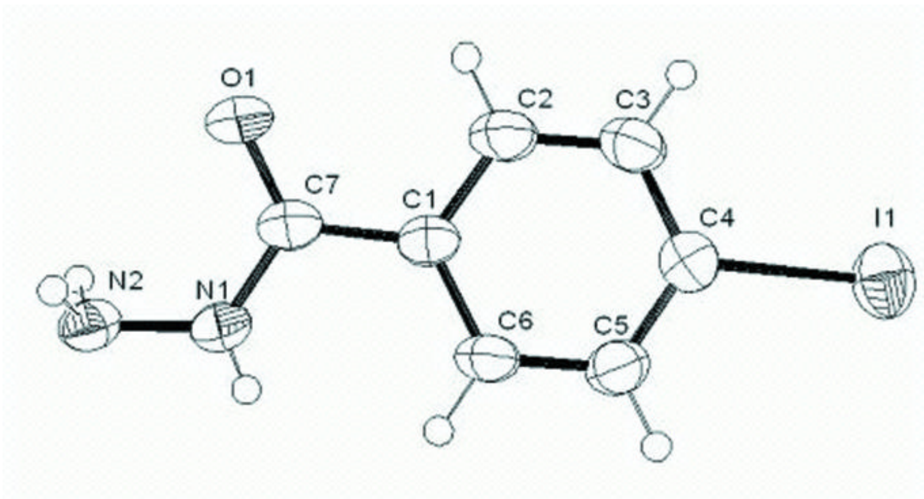

Fig. 2