Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,4,5-Trihydroxybenzohydrazide

Uzma Ashiq,[a]* Rifat Ara Jamal[a] and Sammer Yousuf[b]

[a]Department of Chemistry, University of Karachi, Karachi-75270, Pakistan, and [b]HEJ Research Institute of Chemistry, International Center for Chemical and Biological Sciences, University of Karachi, Karachi-75270, Pakistan
Correspondence e-mail: uzzmma@yahoo.com

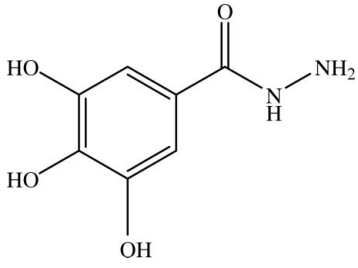
Received 12 August 2011; accepted 21 August 2011

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.092; data-to-parameter ratio = 9.0.

In the title compound, $C_7H_8N_2O_4$, the dihedral angle between the aromatic ring and the hydrazide grouping is 21.34 (7)°. In the crystal, the molecules are linked into a three-dimensional network by O—H···O, O—H···N and N—H···O hydrogen bonds.

Related literature

For the biological activity of hydrazides, see: Maqsood *et al.* (2006). For related structures, see: Jamal *et al.* (2009); Saeed *et al.* (2008); Zareef *et al.* (2006).



Experimental

Crystal data

$C_7H_8N_2O_4$
M_r = 184.15
Monoclinic, $P2_1/c$
a = 3.7307 (3) Å
b = 22.8402 (18) Å
c = 8.7064 (7) Å
β = 93.290 (2)°
V = 740.65 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 273 K
0.28 × 0.21 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.963, T_{max} = 0.973
4345 measured reflections
1352 independent reflections
1234 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.092
S = 1.09
1352 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.85 (3)	2.09 (2)	2.8254 (14)	145.0 (19)
N1—H1B···O1^{ii}	0.86 (2)	2.24 (2)	2.9960 (15)	146.2 (16)
O2—H2A···N2^{iii}	0.91 (3)	1.80 (2)	2.6877 (17)	165 (2)
N2—H2B···O3^{iv}	0.88 (2)	2.25 (2)	3.1158 (17)	167.0 (18)
N2—H2C···O4^{v}	0.92 (2)	2.454 (18)	3.2255 (18)	141.8 (15)
O3—H3A···O4^{vi}	0.89 (2)	1.77 (2)	2.6522 (15)	171 (2)

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x+2, -y+1, -z+1$; (iii) $x-1, y, z+1$; (iv) $x, y, z-1$; (v) $x+1, y, z$; (vi) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

The authors are grateful to the Higher Education Commission (HEC) Pakistan for financial support under the National Research Grants Program for Universities (grant No. 1862/R&D/10).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6363).

References

Bruker (2000). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jamal, R. A., Ashiq, U., Arshad, M. N., Maqsood, Z. T. & Khan, I. U. (2009). *Acta Cryst.* E**65**, o1764.
Maqsood, Z. T., Khan, K. M., Ashiq, U., Ara, R., Chohan, Z. H., Mahroof-Tahir, M. & Supuran, C. T. (2006). *J. Enzyme Inhib. Med. Chem.* **21**, 37–42.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Saeed, A., Mumtaz, A., Rafique, H., Gotoh, K. & Ishida, H. (2008). *Acta Cryst.* E**64**, o2336.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Zareef, M., Iqbal, R., Qadeer, G., Arfan, M. & Lu, X.-M. (2006). *Acta Cryst.* E**62**, o3259–o3261.

supporting information

Acta Cryst. (2011). E**67**, o2462 [doi:10.1107/S1600536811034374]

3,4,5-Trihydroxybenzohydrazide

Uzma Ashiq, Rifat Ara Jamal and Sammer Yousuf

S1. Comment

In order to further explore the biological significance of hydrazides, we have prepared the title compound (I). It was found to be active against DPPH radical scavenging activity and inactive against all fungal strains (Maqsood *et al.* 2006). The crystal structures of trimethoxybenzohydrazide (Saeed *et al.* 2008, Zareef *et al.* 2006) and *para* hydroxy-benzohydrazide (Jamal *et al.* 2009) analogues of (I) have already been reported.

The molecular structure of (I) is composed of a hydrazide moiety attached to the phenyl ring (Fig. 1). The phenyl ring is almost planar with a maximum deviation of 0.009 (1) Å from the least-squares plane. The bond lengths and angles all are in normal range as in other structurally related compounds (Saeed *et al.* 2008; Zareef *et al.*, 2006). In the crystal, the molecules are linked to form three-dimensional molecular network *via* O1—H1A···O2, N1—H1B···O1, O2—H2A···N2, N2—H2B···O3, N2—H2C···O4 and O3—H3A···O4 intermolecular hydrogen bonds (Tab. 1 & Fig. 2).

S2. Experimental

To a solution of methyl-3,4,5-trihydroxybenzoate (3.68 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 3,4,5-trihydroxybenzohydrazide (yield 87%) (Maqsood *et al.*, 2006). Colourless blocks of (I) were grown from a solution of methanol by slow evaporation at room temperature.

S3. Refinement

The H atoms on the N atoms (N–H= 0.92 (2)–0.86 (19) Å) O atoms (O–H= 0.91 (3)–0.85 (19) Å) and Carbon (C–H= 0.961 (18)–0.955 (17) Å) atoms were located in difference Fourier maps and refined isotropically.

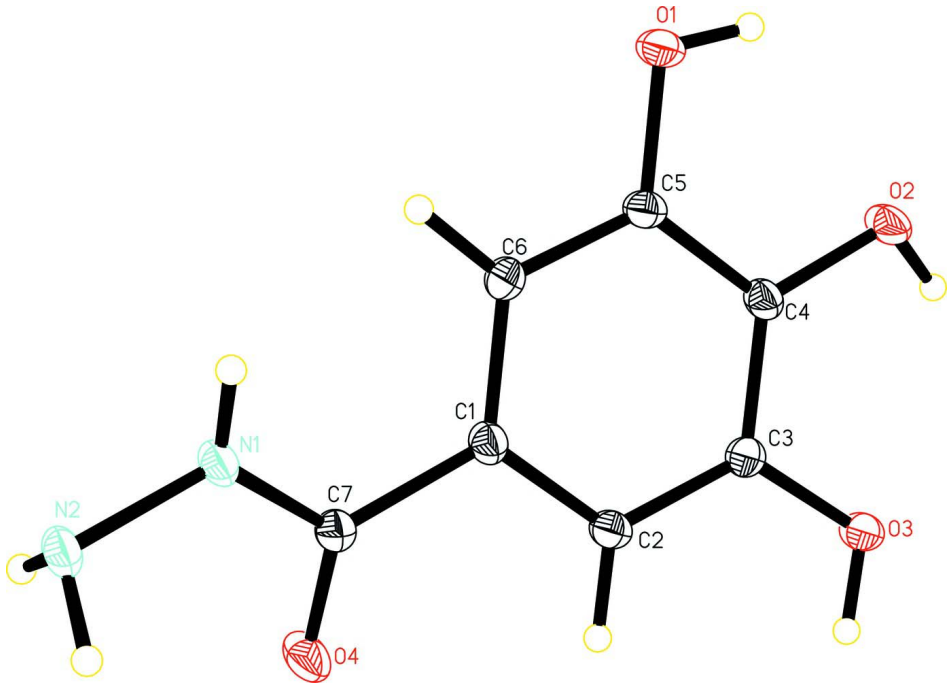


Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at 50% probability level.

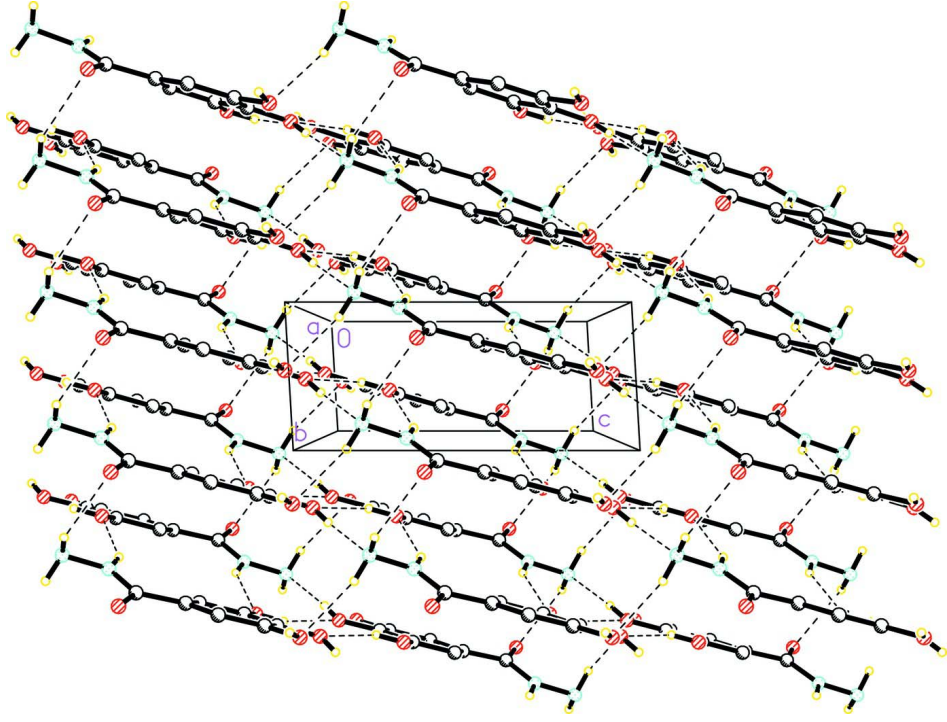


Figure 2

The crystal packing of the title compound I. Only hydrogen atoms involved in hydrogen bonding are shown.

3,4,5-Trihydroxybenzohydrazide

Crystal data

$C_7H_8N_2O_4$
$M_r = 184.15$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 3.7307$ (3) Å
$b = 22.8402$ (18) Å
$c = 8.7064$ (7) Å
$\beta = 93.290$ (2)°
$V = 740.65$ (10) Å^3
$Z = 4$
$F(000) = 384$
$D_x = 1.651$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2464 reflections
$\theta = 3.0$–$28.3°$
$\mu = 0.14$ mm^{-1}
$T = 273$ K
Block, colorles
0.28 × 0.21 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.963$, $T_{max} = 0.973$
4345 measured reflections
1352 independent reflections
1234 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.8°$
$h = -4 \rightarrow 4$
$k = -27 \rightarrow 26$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.092$
$S = 1.09$
1352 reflections
150 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.2069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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}
O1	0.6088 (3)	0.49395 (4)	0.75022 (12)	0.0388 (3)
O2	0.4790 (3)	0.57014 (4)	0.97692 (11)	0.0328 (3)
H2A	0.375 (6)	0.5994 (11)	1.029 (3)	0.069 (7)*

O3	0.5451 (3)	0.68880 (5)	0.93130 (11)	0.0334 (3)
H3A	0.602 (6)	0.7255 (10)	0.907 (2)	0.061 (6)*
O4	0.7876 (3)	0.70585 (5)	0.36218 (12)	0.0402 (3)
N1	1.0268 (3)	0.62028 (5)	0.30646 (13)	0.0270 (3)
H1B	1.100 (5)	0.5854 (9)	0.329 (2)	0.037 (5)*
N2	1.1408 (4)	0.64212 (6)	0.16535 (13)	0.0283 (3)
H2C	1.285 (5)	0.6739 (9)	0.188 (2)	0.041 (5)*
H2B	0.950 (6)	0.6553 (8)	0.112 (2)	0.044 (5)*
C1	0.7698 (4)	0.63145 (6)	0.55422 (14)	0.0224 (3)
C2	0.7029 (4)	0.67156 (6)	0.66914 (15)	0.0250 (3)
H2	0.718 (4)	0.7128 (8)	0.6488 (18)	0.031 (4)*
C3	0.6093 (4)	0.65203 (6)	0.81195 (14)	0.0235 (3)
C4	0.5743 (4)	0.59242 (6)	0.84021 (14)	0.0231 (3)
C5	0.6437 (4)	0.55257 (6)	0.72421 (15)	0.0244 (3)
C6	0.7421 (4)	0.57157 (6)	0.58183 (15)	0.0246 (3)
H6	0.780 (4)	0.5428 (7)	0.5049 (19)	0.030 (4)*
C7	0.8618 (4)	0.65546 (6)	0.40269 (14)	0.0239 (3)
H1A	0.548 (6)	0.4893 (9)	0.842 (3)	0.057 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0733 (9)	0.0179 (5)	0.0271 (6)	0.0005 (5)	0.0199 (5)	0.0016 (4)
O2	0.0557 (7)	0.0214 (5)	0.0230 (5)	0.0030 (5)	0.0173 (5)	0.0034 (4)
O3	0.0602 (8)	0.0190 (5)	0.0227 (5)	−0.0028 (5)	0.0169 (5)	−0.0024 (4)
O4	0.0707 (9)	0.0224 (5)	0.0295 (6)	0.0123 (5)	0.0199 (5)	0.0066 (4)
N1	0.0409 (8)	0.0210 (6)	0.0202 (6)	0.0058 (5)	0.0118 (5)	0.0038 (4)
N2	0.0388 (8)	0.0276 (7)	0.0194 (6)	0.0021 (6)	0.0105 (5)	0.0028 (5)
C1	0.0257 (7)	0.0229 (7)	0.0190 (6)	0.0014 (5)	0.0045 (5)	0.0011 (5)
C2	0.0332 (8)	0.0188 (7)	0.0235 (7)	−0.0002 (6)	0.0063 (5)	0.0012 (5)
C3	0.0291 (8)	0.0211 (7)	0.0209 (6)	0.0002 (5)	0.0061 (5)	−0.0016 (5)
C4	0.0280 (8)	0.0225 (7)	0.0194 (6)	0.0006 (5)	0.0062 (5)	0.0023 (5)
C5	0.0317 (8)	0.0177 (7)	0.0241 (7)	0.0009 (5)	0.0055 (5)	0.0014 (5)
C6	0.0322 (8)	0.0218 (7)	0.0205 (7)	0.0027 (6)	0.0064 (5)	−0.0023 (5)
C7	0.0302 (8)	0.0211 (7)	0.0207 (6)	0.0001 (5)	0.0047 (5)	0.0000 (5)

Geometric parameters (Å, º)

O1—C5	1.3654 (16)	N2—H2B	0.88 (2)
O1—H1A	0.85 (2)	C1—C2	1.3897 (18)
O2—C4	1.3603 (16)	C1—C6	1.3934 (19)
O2—H2A	0.91 (3)	C1—C7	1.4869 (17)
O3—C3	1.3678 (16)	C2—C3	1.3844 (18)
O3—H3A	0.89 (2)	C2—H2	0.961 (18)
O4—C7	1.2306 (17)	C3—C4	1.3912 (19)
N1—C7	1.3363 (18)	C4—C5	1.3946 (19)
N1—N2	1.4139 (15)	C5—C6	1.3828 (19)
N1—H1B	0.860 (19)	C6—H6	0.955 (17)

N2—H2C	0.92 (2)		
C5—O1—H1A	108.1 (14)	O3—C3—C2	123.28 (12)
C4—O2—H2A	107.7 (15)	O3—C3—C4	116.38 (11)
C3—O3—H3A	110.1 (14)	C2—C3—C4	120.35 (12)
C7—N1—N2	120.34 (12)	O2—C4—C3	123.55 (12)
C7—N1—H1B	124.6 (12)	O2—C4—C5	117.28 (12)
N2—N1—H1B	114.4 (12)	C3—C4—C5	119.17 (12)
N1—N2—H2C	107.3 (11)	O1—C5—C6	119.29 (12)
N1—N2—H2B	107.8 (12)	O1—C5—C4	119.76 (12)
H2C—N2—H2B	106.7 (17)	C6—C5—C4	120.95 (12)
C2—C1—C6	120.31 (12)	C5—C6—C1	119.26 (12)
C2—C1—C7	117.11 (12)	C5—C6—H6	118.0 (10)
C6—C1—C7	122.56 (12)	C1—C6—H6	122.7 (10)
C3—C2—C1	119.94 (13)	O4—C7—N1	119.12 (12)
C3—C2—H2	120.1 (9)	O4—C7—C1	122.66 (12)
C1—C2—H2	119.9 (9)	N1—C7—C1	118.21 (12)
C6—C1—C2—C3	0.2 (2)	C3—C4—C5—C6	−0.8 (2)
C7—C1—C2—C3	178.80 (13)	O1—C5—C6—C1	178.56 (13)
C1—C2—C3—O3	178.95 (13)	C4—C5—C6—C1	−0.4 (2)
C1—C2—C3—C4	−1.4 (2)	C2—C1—C6—C5	0.7 (2)
O3—C3—C4—O2	0.5 (2)	C7—C1—C6—C5	−177.85 (13)
C2—C3—C4—O2	−179.16 (13)	N2—N1—C7—O4	5.1 (2)
O3—C3—C4—C5	−178.65 (13)	N2—N1—C7—C1	−175.85 (13)
C2—C3—C4—C5	1.7 (2)	C2—C1—C7—O4	−20.3 (2)
O2—C4—C5—O1	1.0 (2)	C6—C1—C7—O4	158.29 (15)
C3—C4—C5—O1	−179.71 (13)	C2—C1—C7—N1	160.76 (13)
O2—C4—C5—C6	179.99 (13)	C6—C1—C7—N1	−20.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{i}	0.85 (3)	2.09 (2)	2.8254 (14)	145.0 (19)
N1—H1*B*···O1^{ii}	0.86 (2)	2.24 (2)	2.9960 (15)	146.2 (16)
O2—H2*A*···N2^{iii}	0.91 (3)	1.80 (2)	2.6877 (17)	165 (2)
N2—H2*B*···O3^{iv}	0.88 (2)	2.25 (2)	3.1158 (17)	167.0 (18)
N2—H2*C*···O4^{v}	0.92 (2)	2.454 (18)	3.2255 (18)	141.8 (15)
O3—H3*A*···O4^{vi}	0.89 (2)	1.77 (2)	2.6522 (15)	171 (2)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*+1; (iv) *x*, *y*, *z*−1; (v) *x*+1, *y*, *z*; (vi) *x*, −*y*+3/2, *z*+1/2.