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3-(3,5-Di-*tert*-butyl-4-hydroxyphenyl)-propionohydrazide

Wei-Ming Xu, Xiu-Rong Hu* and Jian-Ming Gu

Center of Analysis and Measurement, Zhejiang University, Hangzhou, Zhejiang 310028, People's Republic of China

Correspondence e-mail: huxiurong@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.084
Data-to-parameter ratio = 20.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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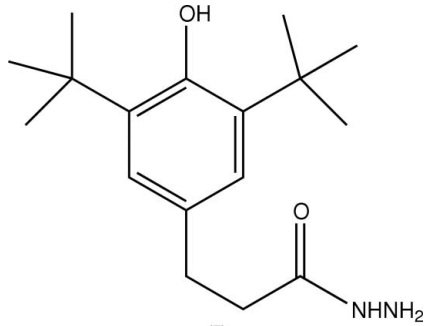
The structure of the title compound, $C_{17}H_{28}N_2O_2$, exhibits an elaborate network of N—H···N, N—H···O and O—H···O hydrogen bonds.

Comment

Substituted hydrazides are very important intermediates in organic synthesis, and are commonly used in the preparation of 1,3,4-oxadiazoles and 1,3,4-thiadiazoles (Kramer *et al.*, 1994). In the structure of the title compound, (I), the O atom of the hydroxy group is displaced slightly from the benzene ring, with a deviation of 0.057 (2) Å, probably due to an intermolecular O—H···O hydrogen bond. The benzene ring and the mean plane through atoms C7–C9/N31 are perpendicular to each other, with a dihedral angle of 89.30 (7)° (Fig. 1).



(I)

The structure of (I) exhibits an elaborate network of N—H···N, N—H···O and O—H···O hydrogen bonds. The mol-

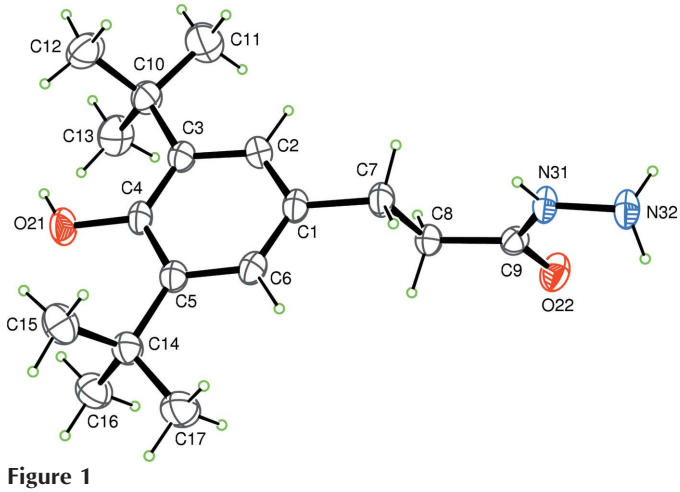


Figure 1
The molecular configuration and atom-numbering scheme of (I). Displacement ellipsoids are drawn at the 40% probability level.

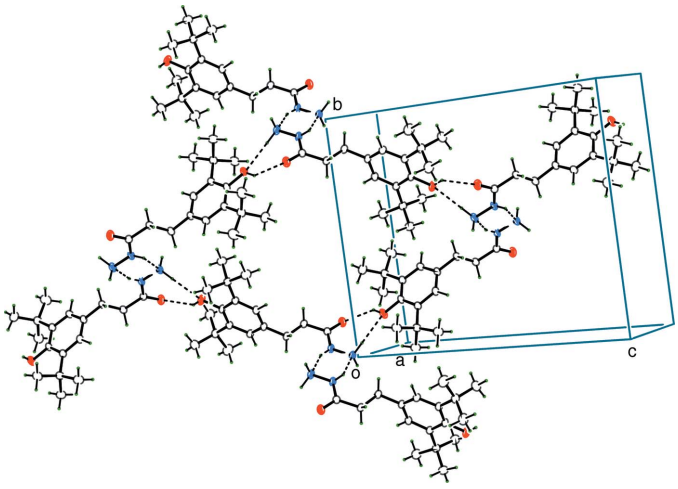


Figure 2
A partial packing diagram of (I), showing the hydrogen-bonded dimer and the macrocyclic ring. Hydrogen bonds are shown as dashed lines.

ecules form centrosymmetric hydrogen-bonded dimers of graph-set descriptor $R_2^2(6)$ (Etter, 1990) through pairs of N31—H301···N32^{ii} hydrogen bonds [symmetry code (ii) as in Table 2]. The dimers are linked by O21—H201···O22^{i} and N32—H302···O21^{iii} hydrogen bonds (Table 2), giving a macrocyclic ring (Fig. 2). In addition, a N32—H303···O2^{iv} hydrogen bond links the rings, forming a three-dimensional hydrogen-bonded network.

Experimental

The title compound was prepared according to the method described by Yin & Shou (2003). 3-(3,5-Di-*tert*-butyl-4-hydroxyphenyl)-propionyl chloride (2.96 g,10 mmol) and 85% hydrazine hydrate (1.1 ml, 12 mmol) were mixed in 50 ml methanol. The mixture was stirred overnight at room temperature. After the reaction, the mixture was evaporated and extracted with EtOAc and water, dried with anhydrous $MgSO_4$ and recrystallized from ethanol to obtain 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionohyrazide (yield 2.83 g, 97%).

Crystal data

$C_{17}H_{28}N_2O_2$
M_r = 292.42
Monoclinic, $P2_1/c$
a = 6.1848 (16) Å
b = 14.685 (5) Å
c = 19.097 (5) Å
β = 95.439 (10)°
V = 1726.7 (9) Å^3
Z = 4
D_x = 1.125 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12611 reflections
θ = 3.3–27.5°
μ = 0.07 mm^{-1}
T = 296 (1) K
Platelet, colorless
0.23 × 0.20 × 0.11 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.934, T_{max} = 0.992
16372 measured reflections
3890 independent reflections
2380 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.039
θ_{max} = 27.5°
$h = -7 \rightarrow 8$
$k = -19 \rightarrow 19$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.084
S = 1.00
3890 reflections
191 parameters
H-atom parameters constrained
$w = 1/[0.0001F_o^2 + 1.09\sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 1.9 (3) × 10^2

Table 1
Selected bond lengths (Å).

O21—C4	1.3794 (14)	N31—N32	1.4202 (14)
O22—C9	1.2365 (14)	N31—C9	1.3270 (16)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O21—H201···O22^{i}	0.84	2.19	2.8710 (10)	139
N31—H301···N32^{ii}	0.89	2.11	2.9182 (14)	151
N32—H302···O21^{iii}	0.96	2.24	3.2011 (18)	173
N32—H303···O22^{iv}	0.89	2.20	3.0756 (18)	164

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

The H atoms of the hydrazino and hydroxy groups were located in a difference Fourier map and refined as riding with their as-found O—H and N—H bond lengths; their isotropic displacement parameters were initially refined, but fixed in the final stage. All other H atoms were placed in calculated positions (C—H = 0.96–0.98 Å) and included in the refinement in the riding-model approximation [U_{iso}(H) = 1.2U_{eq}(C)].

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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