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N-Hexyl-3,4-dihydroxybenzamide

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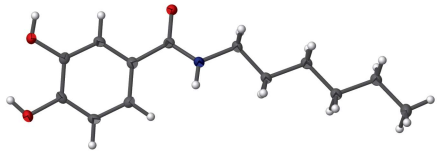
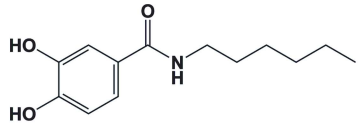
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{19}NO_3$, the hexyl chain has en extended conformation and its mean plane is inclined to the benzene ring by 3.29 (10)°. There is a short O—H···O contact in the molecule involving the adjacent hydroxy groups. In the crystal, molecules are linked *via* O—H···O and N—H···O hydrogen bonds, forming slabs parallel to (001). Within the slabs, there are also C—H···O hydrogen bonds and C—H···π interactions present.

3D view



Chemical scheme



Structure description

N-alkyl-3,4-dihydroxy benzamides are valuable in biological chemistry, having been identified as inhibitors of the trypanosome alternative oxidase in a cell-free mitochondrial preparation of *Tiypanosoma brucei brucei* (Grady *et al.*, 1993). They were also used for the identification of potent antimalarial agents against *Plasmodium falciparum* (3D7) parasites and a normal human cell line (Choomuenwai *et al.*, 2013). Some 3,4-dihydroxy benzamide derivatives have been used as inhibitors of ribonucleotide reductase with antineoplastic activity (Elford *et al.*, 1979).

The molecular structure of the title compound is illustrated in Fig. 1. The hexyl chain has en extended conformation and its mean plane [C8–C13; maximum deviation of 0.039 (2) Å for atom C12] is inclined to the benzene ring by 3.29 (10)°. The amide group (O3/C7/N1) is inclined to the benzene ring and the mean plane of the hexyl chain by 17.85 (14) and 16.33 (10)°, respectively. There is a short O—H···O contact in the molecule involving adjacent hydroxy groups (Table 1).

In the crystal, molecules are linked *via* O—H···O hydrogen bonds, forming ribbons along the *b*-axis direction which enclose $R_2^2(10)$ and $R_2^2(14)$ ring motifs (Table 1 and Fig. 2). The ribbons are linked *via* N—H···O and C—H···O hydrogen bonds, forming slabs parallel to the *ab* plane (Table 1 and Figs. 3 and 4). Within the slabs there are C—H···π interactions present (Table 1).

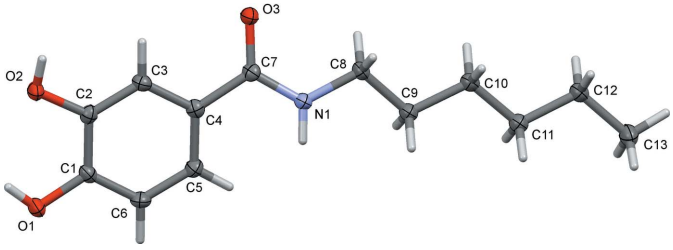


Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

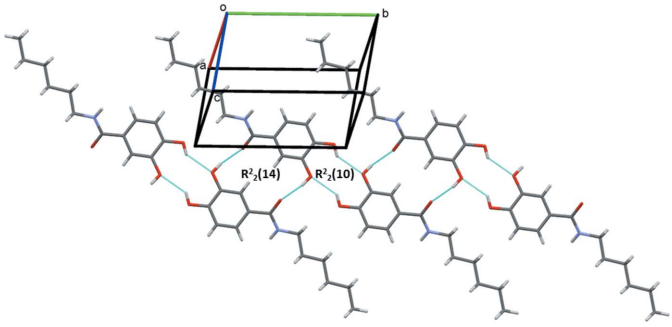


Figure 2
Partial crystal packing diagram of the title compound, illustrating the formation of the hydrogen bonded (see Table 1) ribbons extending in the *b*-axis direction.

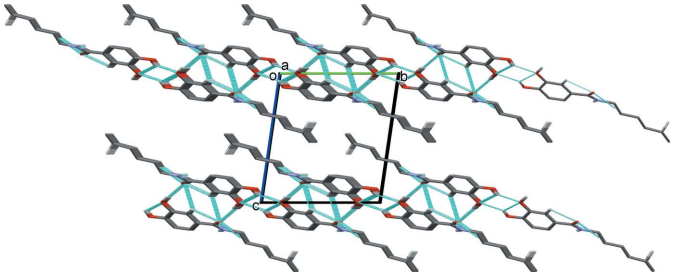


Figure 3
Crystal packing of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines (see Table 1) and H atoms not involved in these interactions have been omitted for clarity.

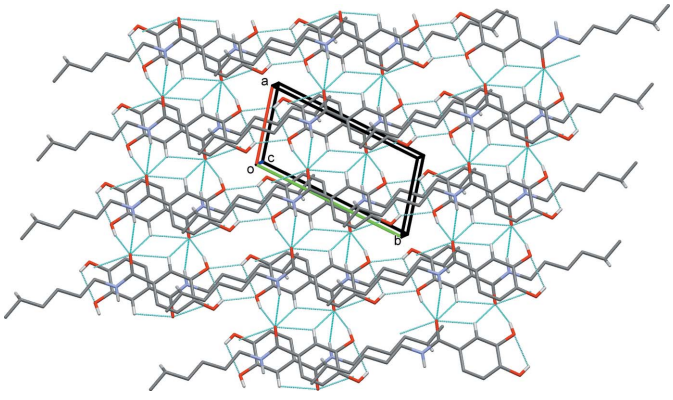


Figure 4
Crystal packing of the title compound, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines (see Table 1) and H atoms not involved in these interactions have been omitted for clarity.

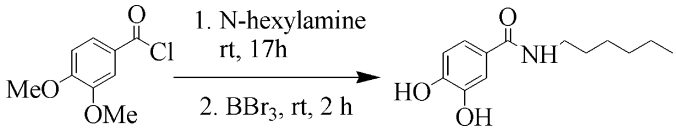


Figure 5
Reaction scheme for the synthesis of the title compound.

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	2.31	2.744 (2)	114
O1—H1···O2^{i}	0.82	2.19	2.868 (2)	140
O2—H2···O3^{ii}	0.82	1.96	2.754 (2)	162
N1—H7···O3^{iii}	0.86	2.46	3.220 (2)	147
C3—H3···O3^{ii}	0.93	2.53	3.155 (2)	125
C12—H12*A*···*Cg*1^{iv}	0.97	2.92	3.763 (3)	146

Symmetry codes: (i) $-x+3, -y+2, -z+2$; (ii) $-x+3, -y+1, -z+2$; (iii) $x-1, y, z$; (iv) $x-1, y-1, z+1$.

Synthesis and crystallization

The synthesis of the title compound is illustrated in Fig. 5. *N*-hexylamine (0.6 g, 5.5 mmol, 1.1 eq) and Et_3N (0.6 g, 5.5 mmol, 1.1 eq) were added to 20 ml of freshly distilled CH_2Cl_2 and cooled to 273 K. To this mixture 3,4-dimethoxy benzoyl chloride (1 g, 5.0 mmol, 1 eq) in 10 ml CH_2Cl_2 was

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{19}NO_3$
M_r	237.29
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	120
a, *b*, *c* (Å)	5.2542 (17), 10.374 (3), 11.341 (4)
α, β, γ (°)	94.937 (3), 102.429 (3), 102.811 (3)
V (Å^3)	582.8 (3)
Z	2
Radiation type	Mo *Kα*
μ (mm^{-1})	0.10
Crystal size (mm)	0.45 × 0.30 × 0.25
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.766, 0.976
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5584, 2055, 1735
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.036, 0.090, 1.04
No. of reflections	2055
No. of parameters	157
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.20

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009).

added dropwise. The mixture was stirred for 17 h at rt. The solvent was removed under reduced pressure. The resulting crude material was then dissolved in 40 ml of AcOEt. The organic phase was washed in 10% HCl, 10 ml of 10% Na_2CO_3, and brine solution. The organic layer was evaporated under reduced pressure and added to freshly distilled CH_2Cl_2 (10 ml). Under cooling, BBr_3 (6 mmol) was added slowly to the solution. The mixture was stirred for 2 h at rt. After adding H_2O (20 ml), the mixture was stirred for a few min, then the aqueous layer was extracted with Et_2O. The organic phase was washed with brine, dried (Na_2SO_4), and concentrated under reduced pressure. The solid obtained was recrystallized in MeOH by slow evaporation at room temperature giving colourless prismatic crystals of the title compound.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160346 [doi:10.1107/S2414314616003461]

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Crystal data

$C_{13}H_{19}NO_3$
M_r = 237.29
Triclinic, $P\overline{1}$
a = 5.2542 (17) Å
b = 10.374 (3) Å
c = 11.341 (4) Å
α = 94.937 (3)°
β = 102.429 (3)°
γ = 102.811 (3)°
V = 582.8 (3) Å^3
Z = 2
$F(000)$ = 256
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2404 reflections
θ = 2.6–25.0°
μ = 0.10 mm^{-1}
T = 120 K
Prism, colourless
0.45 × 0.30 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.6666 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.766, T_{max} = 0.976
5584 measured reflections
2055 independent reflections
1735 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −6→6
k = −12→12
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.090
S = 1.04
2055 reflections
157 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
$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2 + 0.1931P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0844 (3)	0.78967 (13)	0.87939 (12)	0.0183 (3)
C2	1.2999 (3)	0.73975 (13)	0.93220 (12)	0.0175 (3)
C3	1.2798 (3)	0.60507 (13)	0.91268 (12)	0.0179 (3)
H3	1.4264	0.5724	0.9463	0.021*
C4	1.0462 (3)	0.51669 (13)	0.84413 (11)	0.0170 (3)
C5	0.8318 (3)	0.56750 (13)	0.79226 (12)	0.0195 (3)
H5	0.6733	0.51	0.7457	0.023*
C6	0.8530 (3)	0.70264 (14)	0.80954 (13)	0.0206 (3)
H6	0.7089	0.7358	0.7735	0.025*
C7	1.0362 (3)	0.37214 (13)	0.82975 (11)	0.0169 (3)
C8	0.7592 (3)	0.14432 (13)	0.76710 (13)	0.0195 (3)
H8A	0.7809	0.1068	0.843	0.023*
H8B	0.895	0.1257	0.7269	0.023*
C9	0.4842 (3)	0.08023 (13)	0.68689 (12)	0.0191 (3)
H9A	0.4637	0.1184	0.6113	0.023*
H9B	0.3494	0.1001	0.7273	0.023*
C10	0.4357 (3)	−0.06982 (13)	0.65796 (13)	0.0196 (3)
H10A	0.5701	−0.0892	0.6172	0.023*
H10B	0.4588	−0.1075	0.7338	0.023*
C11	0.1601 (3)	−0.13682 (13)	0.57857 (13)	0.0206 (3)
H11A	0.1334	−0.0959	0.5046	0.025*
H11B	0.0259	−0.1212	0.6212	0.025*
C12	0.1159 (3)	−0.28569 (13)	0.54417 (13)	0.0221 (3)
H12A	0.1555	−0.3259	0.6179	0.027*
H12B	0.2407	−0.3013	0.496	0.027*
C13	−0.1679 (3)	−0.35323 (14)	0.47253 (14)	0.0276 (4)
H13A	−0.2919	−0.3425	0.5214	0.041*
H13B	−0.1824	−0.4466	0.4511	0.041*
H13C	−0.2092	−0.3134	0.3997	0.041*
N1	0.7959 (2)	0.28767 (11)	0.79265 (10)	0.0191 (3)
H7	0.6557	0.3191	0.7833	0.023*
O1	1.0932 (2)	0.92220 (9)	0.89274 (9)	0.0242 (3)
H1	1.2393	0.9638	0.9362	0.036*
O2	1.52394 (19)	0.82996 (9)	1.00208 (9)	0.0213 (2)
H2	1.6232	0.7902	1.042	0.032*
O3	1.24515 (18)	0.33164 (9)	0.85060 (8)	0.0201 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0223 (8)	0.0143 (7)	0.0207 (7)	0.0068 (6)	0.0079 (6)	0.0034 (5)
C2	0.0171 (7)	0.0173 (7)	0.0168 (7)	0.0022 (6)	0.0041 (6)	0.0007 (5)
C3	0.0183 (7)	0.0183 (7)	0.0186 (7)	0.0077 (6)	0.0039 (6)	0.0034 (5)
C4	0.0189 (7)	0.0169 (7)	0.0157 (7)	0.0043 (6)	0.0053 (6)	0.0021 (5)
C5	0.0167 (7)	0.0186 (7)	0.0207 (7)	0.0028 (6)	0.0015 (6)	0.0013 (5)
C6	0.0168 (7)	0.0208 (7)	0.0252 (7)	0.0083 (6)	0.0028 (6)	0.0047 (6)
C7	0.0182 (7)	0.0179 (7)	0.0137 (6)	0.0051 (6)	0.0018 (5)	0.0012 (5)
C8	0.0198 (7)	0.0137 (7)	0.0245 (7)	0.0057 (6)	0.0036 (6)	0.0008 (5)
C9	0.0178 (7)	0.0159 (7)	0.0236 (7)	0.0058 (6)	0.0037 (6)	0.0021 (6)
C10	0.0185 (8)	0.0169 (7)	0.0237 (7)	0.0061 (6)	0.0049 (6)	0.0015 (6)
C11	0.0191 (7)	0.0175 (7)	0.0249 (7)	0.0058 (6)	0.0042 (6)	0.0018 (6)
C12	0.0206 (8)	0.0177 (7)	0.0276 (8)	0.0066 (6)	0.0043 (6)	0.0003 (6)
C13	0.0262 (8)	0.0180 (7)	0.0338 (8)	0.0048 (6)	0.0001 (7)	−0.0016 (6)
N1	0.0157 (6)	0.0147 (6)	0.0257 (6)	0.0054 (5)	0.0021 (5)	0.0006 (5)
O1	0.0226 (6)	0.0140 (5)	0.0326 (6)	0.0055 (4)	−0.0001 (5)	0.0001 (4)
O2	0.0187 (5)	0.0139 (5)	0.0268 (5)	0.0028 (4)	−0.0020 (4)	0.0001 (4)
O3	0.0174 (5)	0.0164 (5)	0.0243 (5)	0.0053 (4)	0.0002 (4)	0.0004 (4)

Geometric parameters (Å, º)

C1—O1	1.3596 (16)	C9—C10	1.5152 (18)
C1—C6	1.3771 (19)	C9—H9A	0.97
C1—C2	1.389 (2)	C9—H9B	0.97
C2—O2	1.3683 (16)	C10—C11	1.5087 (19)
C2—C3	1.3724 (19)	C10—H10A	0.97
C3—C4	1.3851 (19)	C10—H10B	0.97
C3—H3	0.93	C11—C12	1.5131 (19)
C4—C5	1.3874 (19)	C11—H11A	0.97
C4—C7	1.4823 (19)	C11—H11B	0.97
C5—C6	1.375 (2)	C12—C13	1.5136 (19)
C5—H5	0.93	C12—H12A	0.97
C6—H6	0.93	C12—H12B	0.97
C7—O3	1.2438 (16)	C13—H13A	0.96
C7—N1	1.3253 (17)	C13—H13B	0.96
C8—N1	1.4517 (17)	C13—H13C	0.96
C8—C9	1.5040 (19)	N1—H7	0.86
C8—H8A	0.97	O1—H1	0.82
C8—H8B	0.97	O2—H2	0.82
O1—C1—C6	118.18 (12)	C10—C9—H9B	109.1
O1—C1—C2	122.54 (12)	H9A—C9—H9B	107.8
C6—C1—C2	119.27 (12)	C11—C10—C9	113.60 (11)
O2—C2—C3	123.39 (12)	C11—C10—H10A	108.8
O2—C2—C1	117.08 (12)	C9—C10—H10A	108.8
C3—C2—C1	119.53 (12)	C11—C10—H10B	108.8

C2—C3—C4	121.52 (12)	C9—C10—H10B	108.8
C2—C3—H3	119.2	H10A—C10—H10B	107.7
C4—C3—H3	119.2	C10—C11—C12	113.76 (11)
C3—C4—C5	118.47 (13)	C10—C11—H11A	108.8
C3—C4—C7	118.58 (12)	C12—C11—H11A	108.8
C5—C4—C7	122.95 (12)	C10—C11—H11B	108.8
C6—C5—C4	120.18 (13)	C12—C11—H11B	108.8
C6—C5—H5	119.9	H11A—C11—H11B	107.7
C4—C5—H5	119.9	C11—C12—C13	113.17 (12)
C5—C6—C1	121.00 (13)	C11—C12—H12A	108.9
C5—C6—H6	119.5	C13—C12—H12A	108.9
C1—C6—H6	119.5	C11—C12—H12B	108.9
O3—C7—N1	121.25 (12)	C13—C12—H12B	108.9
O3—C7—C4	121.37 (12)	H12A—C12—H12B	107.8
N1—C7—C4	117.38 (12)	C12—C13—H13A	109.5
N1—C8—C9	110.52 (11)	C12—C13—H13B	109.5
N1—C8—H8A	109.5	H13A—C13—H13B	109.5
C9—C8—H8A	109.5	C12—C13—H13C	109.5
N1—C8—H8B	109.5	H13A—C13—H13C	109.5
C9—C8—H8B	109.5	H13B—C13—H13C	109.5
H8A—C8—H8B	108.1	C7—N1—C8	122.83 (11)
C8—C9—C10	112.50 (11)	C7—N1—H7	118.6
C8—C9—H9A	109.1	C8—N1—H7	118.6
C10—C9—H9A	109.1	C1—O1—H1	109.5
C8—C9—H9B	109.1	C2—O2—H2	109.5

O1—C1—C2—O2	1.6 (2)	C2—C1—C6—C5	0.5 (2)
C6—C1—C2—O2	−179.16 (12)	C3—C4—C7—O3	−17.92 (19)
O1—C1—C2—C3	−178.48 (12)	C5—C4—C7—O3	162.25 (13)
C6—C1—C2—C3	0.8 (2)	C3—C4—C7—N1	162.71 (12)
O2—C2—C3—C4	178.11 (12)	C5—C4—C7—N1	−17.1 (2)
C1—C2—C3—C4	−1.9 (2)	N1—C8—C9—C10	−179.79 (11)
C2—C3—C4—C5	1.5 (2)	C8—C9—C10—C11	179.43 (12)
C2—C3—C4—C7	−178.29 (12)	C9—C10—C11—C12	177.05 (12)
C3—C4—C5—C6	−0.2 (2)	C10—C11—C12—C13	175.68 (12)
C7—C4—C5—C6	179.63 (13)	O3—C7—N1—C8	−3.6 (2)
C4—C5—C6—C1	−0.8 (2)	C4—C7—N1—C8	175.74 (11)
O1—C1—C6—C5	179.83 (12)	C9—C8—N1—C7	−159.96 (12)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	2.31	2.744 (2)	114
O1—H1···O2^{i}	0.82	2.19	2.868 (2)	140
O2—H2···O3^{ii}	0.82	1.96	2.754 (2)	162
N1—H7···O3^{iii}	0.86	2.46	3.220 (2)	147

C3—H3···O3^{ii}	0.93	2.53	3.155 (2)	125
C12—H12*A*···*Cg*1^{iv}	0.97	2.92	3.763 (3)	146

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