Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Nitro-1-*n*-octyl-1*H*-benzimidazol-2(3*H*)-one

Younes Ouzidan,[a] Y. Kandri Rodi,[a] Natalie Saffon,[b] El Mokhtar Essassi[c] and Seik Weng Ng[d]*

[a]Laboratoire de Chimie Organique Appliquée, Faculté des Sciences et Techniques, Université Sidi Mohamed Ben Abdallah, Fés, Morocco, [b]Service Commun Rayons-X FR2599, Université Paul Sabatier Bâtiment 2R1, 118 route de Narbonne, Toulouse, France, [c]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, and [d]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

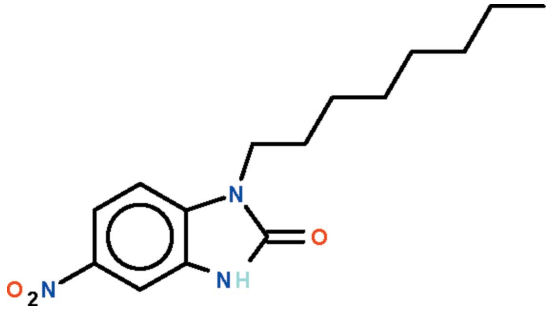
Received 20 January 2011; accepted 26 January 2011

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.055; wR factor = 0.144; data-to-parameter ratio = 15.8.

The benzimidazolone part of the molecule of the title compound, $C_{15}H_{21}N_3O_3$, is almost planar (r.m.s. deviation = 0.007 Å) with its mean plane aligned at a dihedral angle of 10.4 (3)° with respect to the mean plane of the nitro substituent. In the crystal, two molecules are disposed about a center of inversion, generating an N—H···O hydrogen-bonded cyclic dimer with $R_2^2(8)$ graph-set motif.

Related literature

For the crystal structure of 1-isopropenyl-1*H*-benzimidazol-2(3*H*)-one, see: Saber *et al.* (2010). For graph-set notation, see: Etter (1990).



Experimental

Crystal data

$C_{15}H_{21}N_3O_3$
M_r = 291.35
Triclinic, $P\overline{1}$
a = 4.9997 (3) Å
b = 11.4942 (6) Å
c = 13.8739 (7) Å
α = 74.214 (3)°
β = 79.637 (4)°
γ = 84.108 (4)°
V = 753.50 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.22 × 0.12 × 0.06 mm

Data collection

Bruker APEXII diffractometer
10215 measured reflections
3084 independent reflections
1538 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.144
S = 0.95
3084 reflections
195 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···O1^{i}	0.93 (3)	1.84 (3)	2.755 (3)	169 (3)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Université Sidi Mohamed Ben Abdallah, Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2090).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Saber, A., Zouihri, H., Essassi, E. M. & Ng, S. W. (2010). *Acta Cryst.* E**66**, o1409.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2011). E**67**, o558 [doi:10.1107/S1600536811003503]

5-Nitro-1-*n*-octyl-1*H*-benzimidazol-2(3*H*)-one

Younes Ouzidan, Y. Kandri Rodi, Natalie Saffon, El Mokhtar Essassi and Seik Weng Ng

S1. Comment

Tetraalkylammonium halides are used as phase-transfer catalyst in the synthesis of alkyl-substituted benzimidazolones. A previous study reported the 1-isopropenyl derivative; the amino –NH unit forms a hydrogen bond to the inversion-related molecule to generate a hydrogen-bonded dimer (Saber *et al.*, 2010). The present compound (Scheme I) features a long *n*-octyl chain that adopts an extended zigzag conformation (Fig. 1). The benzimidazolone part of the $C_{15}H_{21}N_3O_3$ molecule is planar (r.m.s. deviation 0.007 Å) and its mean plane is aligned at 10.4 (3) ° with respect to the mean plane of the nitro substituent. Two molecules are disposed about a center of inversion to generate a hydrogen-bonded cyclic dimer, whose hydrogen-bonding motif is described by the $R_2^2(8)$ graph set (Etter, 1990).

S2. Experimental

To 5-nitro-1*H*-benzoimidazol-2(3*H*)-one (0.2 g, 1.1 mmol), potassium carbonate (0.30 g, 2.2 mmol) and tetra-*n*-butyl-ammonium bromide (0.07 g, 0.2 mmol) in DMF (15 ml) was added 1-bromo-*n*-octane (0.38 ml, 2.2 mmol). Stirring was continued at room temperature for 6 h. The salt was removed by filtration and the filtrate concentrated under reduced pressure. The residue was separated by chromatography on a column of silica gel with ethyl acetate/hexane (1/2) as eluent. The compound was recrystallized from diethyl ether to give colorless crystals.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2–1.5U_{eq}(C).

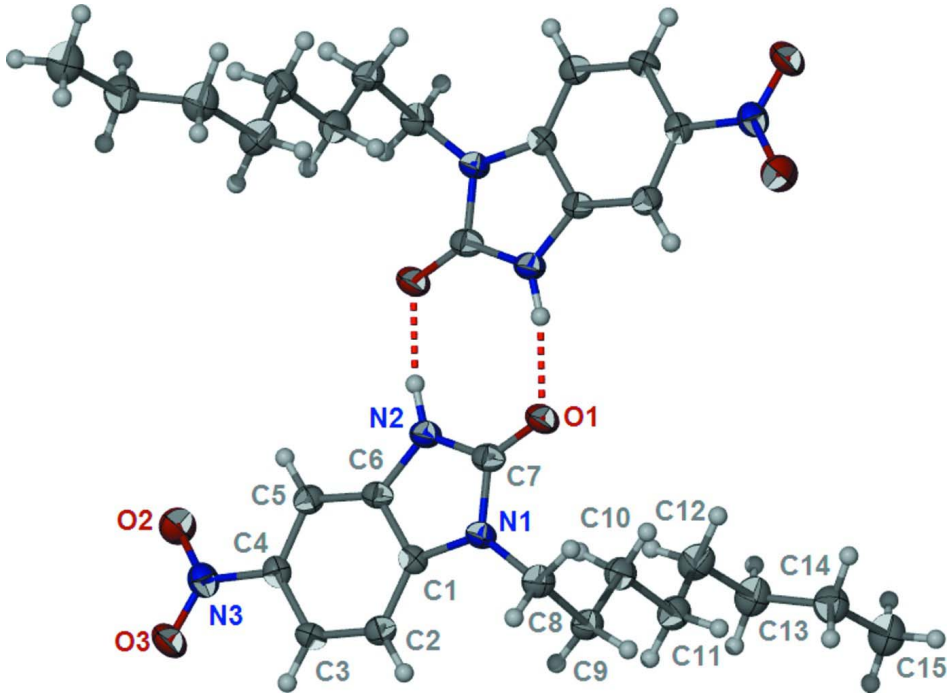


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of two molecules of $C_{15}H_{21}N_3O_3$ disposed about a center of inversion: drawn at the 50% probability level.

5-Nitro-1-*n*-octyl-1*H*-benzimidazol-2(3*H*)-one

Crystal data

$C_{15}H_{21}N_3O_3$
$M_r = 291.35$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.9997$ (3) Å
$b = 11.4942$ (6) Å
$c = 13.8739$ (7) Å
$\alpha = 74.214$ (3)°
$\beta = 79.637$ (4)°
$\gamma = 84.108$ (4)°
$V = 753.50$ (7) Å^3
$Z = 2$
$F(000) = 312$
$D_x = 1.284$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 926 reflections
$\theta = 2.1$–$26.5°$
$\mu = 0.09$ mm^{-1}
$T = 295$ K
Block, colorless
0.22 × 0.12 × 0.06 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
10215 measured reflections
3084 independent reflections
1538 reflections with $I > 2\sigma(I)$
$R_{int} = 0.080$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.1°$
$h = -5 \rightarrow 6$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.144$
$S = 0.95$
3084 reflections
195 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
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.016 (4)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.6268 (4)	0.47630 (17)	0.37325 (14)	0.0426 (5)
O2	−0.5372 (4)	0.15689 (19)	0.72705 (15)	0.0534 (6)
O3	−0.5678 (4)	0.02018 (19)	0.64945 (15)	0.0534 (6)
N1	0.3626 (4)	0.32787 (19)	0.35991 (16)	0.0325 (6)
N2	0.2757 (5)	0.3907 (2)	0.50001 (18)	0.0351 (6)
N3	−0.4666 (5)	0.1112 (2)	0.65519 (18)	0.0407 (6)
C1	0.1487 (5)	0.2628 (2)	0.42194 (19)	0.0299 (6)
C2	0.0021 (5)	0.1746 (2)	0.4085 (2)	0.0341 (7)
H2	0.0387	0.1491	0.3491	0.041*
C3	−0.2003 (5)	0.1257 (2)	0.48628 (19)	0.0336 (7)
H3	−0.3034	0.0660	0.4800	0.040*
C4	−0.2502 (5)	0.1658 (2)	0.57430 (19)	0.0324 (6)
C5	−0.1062 (5)	0.2549 (2)	0.58994 (19)	0.0339 (7)
H5	−0.1433	0.2802	0.6494	0.041*
C6	0.0937 (5)	0.3025 (2)	0.51182 (19)	0.0316 (7)
C7	0.4414 (6)	0.4067 (3)	0.4080 (2)	0.0364 (7)
C8	0.4867 (6)	0.3212 (3)	0.25805 (19)	0.0399 (7)
H8A	0.5128	0.2370	0.2563	0.048*
H8B	0.6644	0.3549	0.2417	0.048*
C9	0.3124 (6)	0.3898 (2)	0.17815 (19)	0.0381 (7)
H9A	0.4130	0.3908	0.1113	0.046*
H9B	0.1488	0.3469	0.1872	0.046*
C10	0.2313 (6)	0.5199 (2)	0.1835 (2)	0.0407 (7)
H10A	0.1183	0.5181	0.2484	0.049*
H10B	0.3947	0.5602	0.1806	0.049*
C11	0.0788 (6)	0.5937 (3)	0.0999 (2)	0.0429 (8)
H11A	−0.0721	0.5490	0.0964	0.051*
H11B	0.1997	0.6062	0.0354	0.051*
C12	−0.0298 (6)	0.7161 (3)	0.1179 (2)	0.0491 (8)
H12A	0.1215	0.7581	0.1248	0.059*
H12B	−0.1543	0.7024	0.1816	0.059*
C13	−0.1770 (6)	0.7985 (3)	0.0349 (2)	0.0507 (9)

H13A	−0.3121	0.7528	0.0213	0.061*
H13B	−0.2729	0.8647	0.0601	0.061*
C14	0.0045 (6)	0.8505 (3)	−0.0629 (2)	0.0509 (8)
H14A	0.0916	0.7847	−0.0908	0.061*
H14B	0.1463	0.8924	−0.0492	0.061*
C15	−0.1477 (7)	0.9384 (3)	−0.1419 (2)	0.0608 (10)
H15A	−0.0220	0.9683	−0.2027	0.091*
H15B	−0.2301	1.0050	−0.1156	0.091*
H15C	−0.2864	0.8972	−0.1568	0.091*
H1	0.296 (6)	0.428 (3)	0.549 (2)	0.059 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0398 (12)	0.0451 (12)	0.0468 (12)	−0.0145 (11)	−0.0018 (10)	−0.0170 (10)
O2	0.0546 (14)	0.0572 (14)	0.0491 (13)	−0.0085 (12)	0.0087 (11)	−0.0240 (11)
O3	0.0546 (14)	0.0502 (14)	0.0571 (14)	−0.0244 (12)	0.0059 (11)	−0.0184 (11)
N1	0.0301 (13)	0.0333 (13)	0.0366 (13)	−0.0044 (11)	−0.0056 (11)	−0.0123 (10)
N2	0.0323 (13)	0.0398 (14)	0.0369 (14)	−0.0067 (11)	−0.0038 (11)	−0.0153 (11)
N3	0.0350 (14)	0.0398 (15)	0.0446 (15)	−0.0036 (12)	−0.0038 (12)	−0.0074 (12)
C1	0.0253 (15)	0.0291 (15)	0.0350 (15)	−0.0002 (13)	−0.0035 (12)	−0.0089 (12)
C2	0.0343 (16)	0.0342 (16)	0.0353 (15)	0.0021 (13)	−0.0053 (13)	−0.0129 (12)
C3	0.0337 (16)	0.0302 (15)	0.0402 (16)	−0.0038 (13)	−0.0076 (13)	−0.0127 (13)
C4	0.0276 (15)	0.0311 (15)	0.0357 (15)	−0.0002 (13)	−0.0033 (12)	−0.0058 (12)
C5	0.0327 (16)	0.0371 (16)	0.0327 (15)	0.0001 (14)	−0.0044 (13)	−0.0119 (13)
C6	0.0306 (15)	0.0303 (15)	0.0371 (16)	0.0002 (13)	−0.0102 (13)	−0.0115 (12)
C7	0.0348 (17)	0.0355 (16)	0.0426 (17)	−0.0001 (14)	−0.0136 (14)	−0.0123 (14)
C8	0.0335 (16)	0.0457 (18)	0.0409 (17)	−0.0014 (14)	−0.0004 (13)	−0.0159 (14)
C9	0.0344 (16)	0.0467 (18)	0.0353 (16)	−0.0033 (14)	0.0021 (13)	−0.0186 (13)
C10	0.0399 (17)	0.0428 (17)	0.0394 (16)	−0.0041 (14)	−0.0029 (14)	−0.0121 (13)
C11	0.0412 (17)	0.0477 (18)	0.0393 (16)	−0.0004 (15)	−0.0021 (14)	−0.0142 (14)
C12	0.055 (2)	0.0479 (19)	0.0447 (18)	0.0084 (16)	−0.0101 (15)	−0.0150 (15)
C13	0.051 (2)	0.051 (2)	0.0470 (18)	0.0069 (16)	−0.0025 (16)	−0.0134 (15)
C14	0.0452 (19)	0.054 (2)	0.0514 (19)	−0.0034 (16)	−0.0071 (16)	−0.0098 (16)
C15	0.063 (2)	0.061 (2)	0.053 (2)	−0.0012 (19)	−0.0071 (18)	−0.0094 (17)

Geometric parameters (Å, º)

O1—C7	1.230 (3)	C9—C10	1.526 (4)
O2—N3	1.231 (3)	C9—H9A	0.9700
O3—N3	1.235 (3)	C9—H9B	0.9700
N1—C7	1.385 (3)	C10—C11	1.515 (4)
N1—C1	1.388 (3)	C10—H10A	0.9700
N1—C8	1.457 (3)	C10—H10B	0.9700
N2—C7	1.368 (3)	C11—C12	1.524 (4)
N2—C6	1.389 (3)	C11—H11A	0.9700
N2—H1	0.93 (3)	C11—H11B	0.9700
N3—C4	1.464 (3)	C12—C13	1.527 (4)

C1—C2	1.380 (4)	C12—H12A	0.9700
C1—C6	1.414 (3)	C12—H12B	0.9700
C2—C3	1.378 (4)	C13—C14	1.500 (4)
C2—H2	0.9300	C13—H13A	0.9700
C3—C4	1.392 (4)	C13—H13B	0.9700
C3—H3	0.9300	C14—C15	1.529 (4)
C4—C5	1.394 (4)	C14—H14A	0.9700
C5—C6	1.369 (4)	C14—H14B	0.9700
C5—H5	0.9300	C15—H15A	0.9600
C8—C9	1.530 (3)	C15—H15B	0.9600
C8—H8A	0.9700	C15—H15C	0.9600
C8—H8B	0.9700		

C7—N1—C1	109.6 (2)	C10—C9—H9B	108.9
C7—N1—C8	123.1 (2)	C8—C9—H9B	108.9
C1—N1—C8	127.3 (2)	H9A—C9—H9B	107.8
C7—N2—C6	110.3 (2)	C11—C10—C9	114.5 (2)
C7—N2—H1	123.8 (19)	C11—C10—H10A	108.6
C6—N2—H1	125.6 (19)	C9—C10—H10A	108.6
O2—N3—O3	122.7 (2)	C11—C10—H10B	108.6
O2—N3—C4	118.5 (3)	C9—C10—H10B	108.6
O3—N3—C4	118.8 (2)	H10A—C10—H10B	107.6
C2—C1—N1	131.6 (2)	C10—C11—C12	111.7 (2)
C2—C1—C6	121.5 (2)	C10—C11—H11A	109.3
N1—C1—C6	106.8 (2)	C12—C11—H11A	109.3
C3—C2—C1	117.6 (3)	C10—C11—H11B	109.3
C3—C2—H2	121.2	C12—C11—H11B	109.3
C1—C2—H2	121.2	H11A—C11—H11B	107.9
C2—C3—C4	119.8 (3)	C11—C12—C13	115.1 (2)
C2—C3—H3	120.1	C11—C12—H12A	108.5
C4—C3—H3	120.1	C13—C12—H12A	108.5
C3—C4—C5	123.8 (3)	C11—C12—H12B	108.5
C3—C4—N3	118.1 (3)	C13—C12—H12B	108.5
C5—C4—N3	118.0 (2)	H12A—C12—H12B	107.5
C6—C5—C4	115.4 (2)	C14—C13—C12	114.8 (2)
C6—C5—H5	122.3	C14—C13—H13A	108.6
C4—C5—H5	122.3	C12—C13—H13A	108.6
C5—C6—N2	131.8 (3)	C14—C13—H13B	108.6
C5—C6—C1	121.7 (3)	C12—C13—H13B	108.6
N2—C6—C1	106.5 (2)	H13A—C13—H13B	107.6
O1—C7—N2	127.7 (3)	C13—C14—C15	113.2 (2)
O1—C7—N1	125.5 (3)	C13—C14—H14A	108.9
N2—C7—N1	106.8 (3)	C15—C14—H14A	108.9
N1—C8—C9	112.1 (2)	C13—C14—H14B	108.9
N1—C8—H8A	109.2	C15—C14—H14B	108.9
C9—C8—H8A	109.2	H14A—C14—H14B	107.8
N1—C8—H8B	109.2	C14—C15—H15A	109.5
C9—C8—H8B	109.2	C14—C15—H15B	109.5

H8A—C8—H8B	107.9	H15A—C15—H15B	109.5
C10—C9—C8	113.2 (2)	C14—C15—H15C	109.5
C10—C9—H9A	108.9	H15A—C15—H15C	109.5
C8—C9—H9A	108.9	H15B—C15—H15C	109.5
C7—N1—C1—C2	−179.8 (3)	C2—C1—C6—C5	0.4 (4)
C8—N1—C1—C2	2.2 (4)	N1—C1—C6—C5	−179.3 (2)
C7—N1—C1—C6	−0.2 (3)	C2—C1—C6—N2	−179.9 (2)
C8—N1—C1—C6	−178.2 (2)	N1—C1—C6—N2	0.5 (3)
N1—C1—C2—C3	179.3 (2)	C6—N2—C7—O1	−179.2 (3)
C6—C1—C2—C3	−0.3 (3)	C6—N2—C7—N1	0.4 (3)
C1—C2—C3—C4	0.0 (4)	C1—N1—C7—O1	179.5 (2)
C2—C3—C4—C5	0.1 (4)	C8—N1—C7—O1	−2.4 (4)
C2—C3—C4—N3	−179.8 (2)	C1—N1—C7—N2	−0.1 (3)
O2—N3—C4—C3	−170.3 (2)	C8—N1—C7—N2	177.9 (2)
O3—N3—C4—C3	10.9 (3)	C7—N1—C8—C9	−100.3 (3)
O2—N3—C4—C5	9.7 (3)	C1—N1—C8—C9	77.4 (3)
O3—N3—C4—C5	−169.1 (2)	N1—C8—C9—C10	52.0 (3)
C3—C4—C5—C6	0.0 (4)	C8—C9—C10—C11	175.1 (2)
N3—C4—C5—C6	179.9 (2)	C9—C10—C11—C12	172.1 (3)
C4—C5—C6—N2	−179.9 (2)	C10—C11—C12—C13	177.7 (3)
C4—C5—C6—C1	−0.2 (3)	C11—C12—C13—C14	−71.2 (4)
C7—N2—C6—C5	179.2 (3)	C12—C13—C14—C15	−176.5 (3)
C7—N2—C6—C1	−0.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O1^{i}	0.93 (3)	1.84 (3)	2.755 (3)	169 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.