

## 1,3-Diallyl-5-chloro-1*H*-benzimidazol-2(3*H*)-one

Younes Ouzidan,<sup>a</sup> Y. Kandri Rodi,<sup>a</sup> Natalie Saffon,<sup>b</sup>  
El Mokhtar Essassi<sup>c</sup> and Seik Weng Ng<sup>d\*</sup>

<sup>a</sup>Laboratoire de Chimie Organique Appliquée, Faculté des Sciences et Techniques, Université Sidi Mohamed Ben Abdallah, Fés, Morocco, <sup>b</sup>Service Commun Rayons-X FR2599, Université Paul Sabatier Bâtiment 2R1, 118 route de Narbonne, Toulouse, France, <sup>c</sup>Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, and <sup>d</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

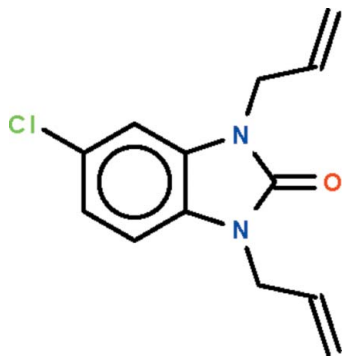
Received 19 January 2011; accepted 23 January 2011

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.049;  $wR$  factor = 0.156; data-to-parameter ratio = 18.6.

The benzimidazolone part of the title molecule,  $\text{C}_{13}\text{H}_{13}\text{ClN}_2\text{O}$ , is almost planar (r.m.s. deviation = 0.006 Å) and its mean plane is aligned at dihedral angles of 62.5 (1) and 78.0 (1)° with respect to the mean planes of the allyl substituents.

### Related literature

For the synthesis, see: Vernin *et al.* (1981).



### Experimental

#### Crystal data

$\text{C}_{13}\text{H}_{13}\text{ClN}_2\text{O}$   
 $M_r = 248.70$   
Monoclinic,  $P2_1/c$   
 $a = 7.8831$  (1) Å  
 $b = 15.2481$  (3) Å  
 $c = 10.3593$  (2) Å  
 $\beta = 93.056$  (1)°

$V = 1243.44$  (4) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.29$  mm<sup>-1</sup>  
 $T = 295$  K  
0.35 × 0.20 × 0.20 mm

#### Data collection

Bruker APEXII diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1997)  
 $T_{\min} = 0.905$ ,  $T_{\max} = 0.944$

17723 measured reflections  
2858 independent reflections  
2230 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.156$   
 $S = 1.03$   
2858 reflections

154 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.98$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.34$  e Å<sup>-3</sup>

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: *pubCIF* (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: JH2259).

### References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.  
Sheldrick, G. M. (1997). *SADABS*. University of Göttingen, Germany.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Vernin, G., Domlog, H., Siv, C., Metzger, J. & El-Shafei, A. K. (1981). *J. Heterocycl. Chem.* **18**, 85–89.  
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

**supplementary materials**

*Acta Cryst.* (2011). E67, o520 [ doi:10.1107/S1600536811003035 ]

## 1,3-Diallyl-5-chloro-1*H*-benzimidazol-2(3*H*)-one

Y. Ouzidan, Y. Kandri Rodi, N. Saffon, E. M. Essassi and S. W. Ng

### Comment

Tetraalkylammonium halides are used as phase-transfer catalyst in the synthesis of 1,3-dialkyl-1,2-benzimidazolones, butyltriethylammonium chloride being used in the synthesis of the 1,3-diallyl derivative (Vernin *et al.*, 1981). This compound as well as its derivatives possess pharmacologically important properties. The title chlorine-substituted compound (Scheme I) was synthesized for evaluation of such properties. The benzimidazolone part of the  $C_{13}H_{13}ClN_2O$  molecule (Fig. 1) is planar (r.m.s. deviation 0.006 Å); its mean plane is aligned at 62.5 (1) and 78.0 (1) with respect to the mean planes of the allyl substituents.

### Experimental

To 5-chloro-1*H*-benzo[*d*]imidazol-2(3*H*)-one (0.2 g, 1.18 mmol), potassium carbonate (0.4 g, 2.8 mmol), and tetra-*n*-butylammonium bromide (0.08 g, 0.23 mmol) in DMF (15 ml) was added allyl-bromide (0.22 ml, 2.6 mmol). Stirring was continued at room temperature for 6 h. The salts were 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. Colorless crystals were isolated when the solvent was allowed to evaporate.

### 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  $U(H)$  set to 1.2–1.5 $U(C)$ .

### Figures

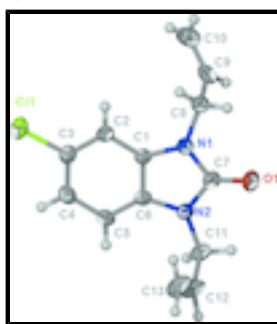


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of  $C_{13}H_{13}ClN_2O$  at the 50% probability level; hydrogen atoms are drawn as arbitrary radius.

## 1,3-Diallyl-5-chloro-1*H*-benzimidazol-2(3*H*)-one

*Crystal data*

$C_{13}H_{13}ClN_2O$

$F(000) = 520$

# supplementary materials

---

$M_r = 248.70$	$D_x = 1.329 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 4645 reflections
$a = 7.8831 (1) \text{ \AA}$	$\theta = 2.4\text{--}29.0^\circ$
$b = 15.2481 (3) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$c = 10.3593 (2) \text{ \AA}$	$T = 295 \text{ K}$
$\beta = 93.056 (1)^\circ$	Block, colourless
$V = 1243.44 (4) \text{ \AA}^3$	$0.35 \times 0.20 \times 0.20 \text{ mm}$
$Z = 4$	

## Data collection

Bruker APEXII diffractometer	2858 independent reflections
Radiation source: fine-focus sealed tube graphite	2230 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.033$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$\theta_{\text{max}} = 27.5^\circ$ , $\theta_{\text{min}} = 2.4^\circ$
$T_{\text{min}} = 0.905$ , $T_{\text{max}} = 0.944$	$h = -10 \rightarrow 10$
17723 measured reflections	$k = -19 \rightarrow 19$
	$l = -12 \rightarrow 13$

## Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.049$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.156$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0846P)^2 + 0.6164P]$
2858 reflections	where $P = (F_o^2 + 2F_c^2)/3$
154 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.98 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.25390 (9)	0.66457 (5)	0.19928 (6)	0.0609 (2)
O1	0.6243 (2)	0.62064 (10)	0.86933 (14)	0.0468 (4)
N1	0.5616 (2)	0.66732 (10)	0.65711 (15)	0.0341 (4)
N2	0.4252 (2)	0.55197 (10)	0.72844 (15)	0.0340 (4)
C1	0.4530 (2)	0.63903 (12)	0.55655 (17)	0.0304 (4)
C2	0.4221 (2)	0.67167 (12)	0.43253 (18)	0.0346 (4)
H2	0.4781	0.7208	0.4029	0.042*
C3	0.3021 (3)	0.62631 (14)	0.35525 (19)	0.0388 (5)

C4	0.2168 (3)	0.55288 (14)	0.3964 (2)	0.0407 (5)
H4	0.1391	0.5244	0.3403	0.049*
C5	0.2476 (2)	0.52151 (13)	0.5227 (2)	0.0371 (4)
H5	0.1904	0.4728	0.5526	0.045*
C6	0.3663 (2)	0.56580 (12)	0.60094 (17)	0.0307 (4)
C7	0.5461 (3)	0.61349 (12)	0.76399 (18)	0.0348 (4)
C8	0.6889 (3)	0.73629 (13)	0.6498 (2)	0.0385 (5)
H8A	0.7127	0.7607	0.7353	0.046*
H8B	0.6442	0.7829	0.5941	0.046*
C9	0.8507 (3)	0.70238 (14)	0.5983 (2)	0.0438 (5)
H9	0.9055	0.6560	0.6414	0.053*
C10	0.9186 (3)	0.73440 (16)	0.4965 (3)	0.0548 (6)
H10A	0.8664	0.7808	0.4516	0.066*
H10B	1.0192	0.7109	0.4687	0.066*
C11	0.3710 (3)	0.48204 (13)	0.8125 (2)	0.0410 (5)
H11A	0.4544	0.4757	0.8842	0.049*
H11B	0.3681	0.4274	0.7645	0.049*
C12	0.2008 (3)	0.49716 (17)	0.8649 (2)	0.0561 (7)
H12	0.1536	0.4507	0.9088	0.067*
C13	0.1120 (4)	0.5692 (2)	0.8550 (3)	0.0647 (8)
H13A	0.1540	0.6174	0.8120	0.078*
H13B	0.0066	0.5723	0.8911	0.078*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0596 (4)	0.0792 (5)	0.0427 (3)	-0.0024 (3)	-0.0096 (3)	0.0139 (3)
O1	0.0592 (10)	0.0442 (8)	0.0362 (7)	-0.0043 (7)	-0.0055 (7)	0.0002 (6)
N1	0.0399 (9)	0.0296 (8)	0.0326 (8)	-0.0039 (6)	0.0018 (7)	-0.0010 (6)
N2	0.0393 (9)	0.0291 (8)	0.0339 (8)	-0.0015 (6)	0.0054 (6)	0.0019 (6)
C1	0.0300 (9)	0.0290 (8)	0.0329 (9)	0.0024 (7)	0.0062 (7)	-0.0024 (7)
C2	0.0335 (10)	0.0357 (10)	0.0352 (9)	0.0027 (7)	0.0070 (8)	0.0034 (7)
C3	0.0355 (10)	0.0477 (11)	0.0335 (9)	0.0087 (8)	0.0041 (8)	0.0005 (8)
C4	0.0320 (10)	0.0473 (11)	0.0427 (11)	-0.0002 (8)	0.0013 (8)	-0.0081 (9)
C5	0.0332 (10)	0.0350 (9)	0.0437 (10)	-0.0031 (8)	0.0069 (8)	-0.0039 (8)
C6	0.0303 (9)	0.0288 (8)	0.0336 (9)	0.0028 (7)	0.0071 (7)	-0.0023 (7)
C7	0.0406 (10)	0.0288 (9)	0.0351 (9)	0.0020 (8)	0.0043 (8)	-0.0014 (7)
C8	0.0465 (12)	0.0300 (9)	0.0391 (10)	-0.0081 (8)	0.0024 (8)	-0.0036 (8)
C9	0.0348 (11)	0.0325 (10)	0.0627 (13)	-0.0023 (8)	-0.0097 (9)	0.0045 (9)
C10	0.0429 (13)	0.0474 (12)	0.0750 (17)	-0.0005 (10)	0.0119 (11)	-0.0051 (12)
C11	0.0500 (12)	0.0313 (10)	0.0424 (11)	0.0032 (8)	0.0094 (9)	0.0089 (8)
C12	0.0663 (16)	0.0553 (14)	0.0492 (13)	0.0046 (12)	0.0263 (12)	0.0163 (10)
C13	0.0656 (17)	0.0776 (18)	0.0535 (14)	0.0202 (14)	0.0259 (13)	0.0098 (13)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C11—C3	1.741 (2)	C5—H5	0.9300
O1—C7	1.229 (2)	C8—C9	1.501 (3)
N1—C1	1.382 (2)	C8—H8A	0.9700

## supplementary materials

---

N1—C7	1.389 (2)	C8—H8B	0.9700
N1—C8	1.459 (2)	C9—C10	1.303 (3)
N2—C7	1.373 (3)	C9—H9	0.9300
N2—C6	1.393 (2)	C10—H10A	0.9300
N2—C11	1.455 (2)	C10—H10B	0.9300
C1—C2	1.387 (3)	C11—C12	1.492 (3)
C1—C6	1.400 (3)	C11—H11A	0.9700
C2—C3	1.390 (3)	C11—H11B	0.9700
C2—H2	0.9300	C12—C13	1.303 (4)
C3—C4	1.385 (3)	C12—H12	0.9300
C4—C5	1.402 (3)	C13—H13A	0.9300
C4—H4	0.9300	C13—H13B	0.9300
C5—C6	1.381 (3)		
C1—N1—C7	109.83 (16)	N2—C7—N1	106.21 (16)
C1—N1—C8	125.89 (16)	N1—C8—C9	111.75 (16)
C7—N1—C8	123.90 (17)	N1—C8—H8A	109.3
C7—N2—C6	110.02 (15)	C9—C8—H8A	109.3
C7—N2—C11	124.17 (17)	N1—C8—H8B	109.3
C6—N2—C11	125.80 (17)	C9—C8—H8B	109.3
N1—C1—C2	131.12 (17)	H8A—C8—H8B	107.9
N1—C1—C6	107.17 (16)	C10—C9—C8	123.5 (2)
C2—C1—C6	121.71 (18)	C10—C9—H9	118.2
C3—C2—C1	115.81 (18)	C8—C9—H9	118.2
C3—C2—H2	122.1	C9—C10—H10A	120.0
C1—C2—H2	122.1	C9—C10—H10B	120.0
C4—C3—C2	123.46 (19)	H10A—C10—H10B	120.0
C4—C3—C11	118.11 (17)	N2—C11—C12	113.75 (17)
C2—C3—C11	118.43 (16)	N2—C11—H11A	108.8
C3—C4—C5	120.04 (19)	C12—C11—H11A	108.8
C3—C4—H4	120.0	N2—C11—H11B	108.8
C5—C4—H4	120.0	C12—C11—H11B	108.8
C6—C5—C4	117.31 (18)	H11A—C11—H11B	107.7
C6—C5—H5	121.3	C13—C12—C11	126.3 (2)
C4—C5—H5	121.3	C13—C12—H12	116.9
C5—C6—N2	131.58 (17)	C11—C12—H12	116.9
C5—C6—C1	121.65 (18)	C12—C13—H13A	120.0
N2—C6—C1	106.76 (16)	C12—C13—H13B	120.0
O1—C7—N2	127.35 (18)	H13A—C13—H13B	120.0
O1—C7—N1	126.44 (18)		
C7—N1—C1—C2	179.23 (19)	C2—C1—C6—C5	1.1 (3)
C8—N1—C1—C2	-7.7 (3)	N1—C1—C6—N2	0.17 (19)
C7—N1—C1—C6	0.2 (2)	C2—C1—C6—N2	-178.95 (16)
C8—N1—C1—C6	173.33 (17)	C6—N2—C7—O1	179.96 (19)
N1—C1—C2—C3	-179.86 (18)	C11—N2—C7—O1	-1.2 (3)
C6—C1—C2—C3	-1.0 (3)	C6—N2—C7—N1	0.6 (2)
C1—C2—C3—C4	-0.1 (3)	C11—N2—C7—N1	179.50 (16)
C1—C2—C3—C11	178.90 (14)	C1—N1—C7—O1	-179.86 (19)
C2—C3—C4—C5	1.1 (3)	C8—N1—C7—O1	6.9 (3)

C11—C3—C4—C5	-177.91 (15)	C1—N1—C7—N2	-0.5 (2)
C3—C4—C5—C6	-1.0 (3)	C8—N1—C7—N2	-173.81 (16)
C4—C5—C6—N2	179.97 (18)	C1—N1—C8—C9	-83.2 (2)
C4—C5—C6—C1	-0.1 (3)	C7—N1—C8—C9	88.9 (2)
C7—N2—C6—C5	179.44 (19)	N1—C8—C9—C10	122.9 (2)
C11—N2—C6—C5	0.6 (3)	C7—N2—C11—C12	104.9 (2)
C7—N2—C6—C1	-0.5 (2)	C6—N2—C11—C12	-76.4 (3)
C11—N2—C6—C1	-179.34 (17)	N2—C11—C12—C13	-9.0 (4)
N1—C1—C6—C5	-179.79 (17)		

Fig. 1

