Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,3-Bis(2-methylprop-2-enoyl)-1*H*-benzimidazol-2(3*H*)-one

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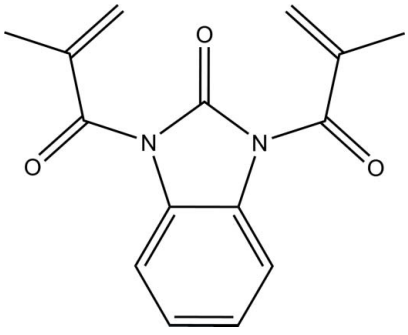
Received 2 March 2013; accepted 25 April 2013

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.057; wR factor = 0.208; data-to-parameter ratio = 12.5.

The molecules of the title compound, $C_{15}H_{14}N_2O_3$, possesses crystallographically imposed twofold rotational symmetry, so the asymmetric unit contains one half-molecule. The fused-ring system deviates significantly from planarity; the planes of the five- and six-membered rings are twisted with respect to each other by 3.0 (1)°. In the crystal, weak C—H···O hydrogen bonds link molecules related by translation in [010] into chains.

Related literature

For applications of substituted benzimidazoles, see: Gravatt *et al.* (1994); Srikanth *et al.* (2011). For the crystal structures of related compounds, see: Ouzidan *et al.* (2011); Kandri Rodi *et al.* (2011).



Experimental

Crystal data

$C_{15}H_{14}N_2O_3$ M_r = 270.28
Monoclinic, $C2/c$
a = 16.6359 (9) Å
b = 8.8629 (5) Å
c = 9.6221 (4) Å
β = 102.775 (2)°
V = 1383.59 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.22 × 0.18 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.980, T_{max} = 0.991
4086 measured reflections
1165 independent reflections
1042 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.208
S = 1.16
1165 reflections
93 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1^{i}	0.93	2.57	3.193 (3)	124

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the Department of Chemistry, IIT Madras, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5390).

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supplementary materials

Acta Cryst. (2013). E**69**, o818 [doi:10.1107/S1600536813011380]

1,3-Bis(2-methylprop-2-enoyl)-1*H*-benzimidazol-2(3*H*)-one

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Comment

Substituted benzimidazole derivatives have found wide range of therapeutic and pharmacological applications (Gravatt *et al.*, 1994; Srikanth *et al.*, 2011). Herewith we present the title compound, (I), which is a new derivative of benzimidazole.

In (I) (Fig. 1), all bond lengths and angles are normal and comparable with those reported for related compounds (Ouzidan *et al.*, 2011; Kandri Rodi *et al.*, 2011). The molecules in (I) possess a crystallographically imposed twofold rotational symmetry, with half of the molecule in the assymetric unit. The five- and six-membered rings are twisted with a dihedral angle of 3.0 (1)°.

In the crystal, weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules related by translation in [010] into chains.

Experimental

2-Hydroxy benzimidazole (1 g, 0.007 moles) and THF (100 ml) were placed in a 3-neck round bottomed flask. Methacrylic anhydride (2.29 g, 0.014 moles) was added slowly, using a syringe, with stirring. The mixture was allowed to cool to 0 °C using ice-salt mixture with stirring. The mixture was treated with sodium hydroxide (0.56 g, 0.014 moles) drop by drop and the amide formation reaction was allowed to stir for 6 h at RT. The resulting crude product was dissolved in ethyl acetate, washed with bicarbonate solution and then with water thrice followed by brine solution and dried over anhydrous sodium sulfate. The resulting solvent was removed by rotary evaporation. The product was purified by column chromatography technique using 12% ethyl acetate in hexane as the eluent to obtain a product as a bright white solid. Recrystallization of the compound from acetone gave X-ray diffraction quality crystals of (I).

Refinement

All hydrogen atoms were fixed geometrically and allowed to ride on the parent carbon atoms with aromatic C—H = 0.93 Å, methylene C—H = 0.97 Å and methyl C—H = 0.96 Å. The displacement parameters were set as U_{iso}(H) = 1.2–1.5 U_{eq}(C).

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

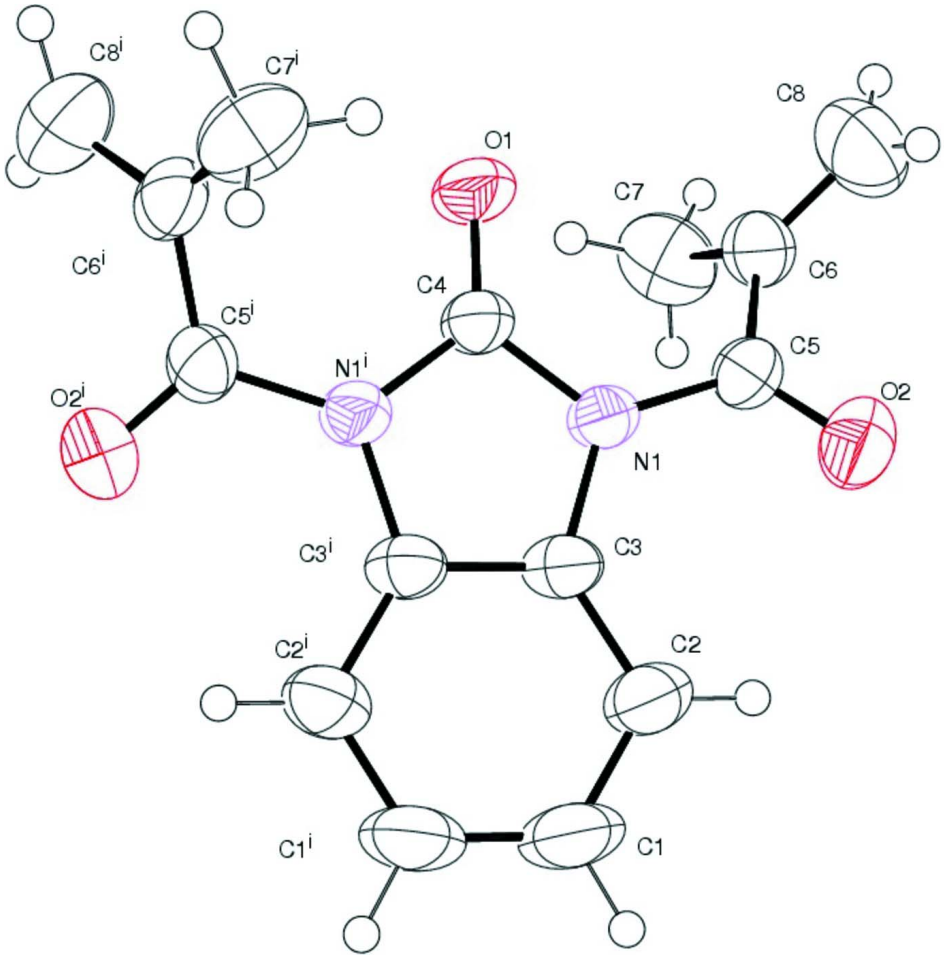

Figure 1

View of (I) showing the atomic numbering and 50% probability displacement ellipsoids [symmetry code: -*x*, *y*, 1/2 - *z*].

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Crystal data

$C_{15}H_{14}N_2O_3$
M_r = 270.28
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 16.6359 (9) Å
b = 8.8629 (5) Å
c = 9.6221 (4) Å
β = 102.775 (2)°
V = 1383.59 (12) Å^3
Z = 4
F(000) = 568
D_x = 1.298 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3023 reflections
θ = 2.6–28.4°
μ = 0.09 mm^{-1}
T = 298 K
Prism, colourless
0.22 × 0.18 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.980, T_{max} = 0.991
4086 measured reflections
1165 independent reflections

1042 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.2°$
$h = -16 \rightarrow 19$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.208$
$S = 1.16$
1165 reflections
93 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.1442P)^2 + 0.5014P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ 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	0.47013 (18)	0.8707 (3)	0.7898 (4)	0.0750 (10)
H1	0.4508	0.9622	0.8167	0.090*
C2	0.43851 (17)	0.7372 (3)	0.8301 (3)	0.0601 (8)
H2	0.3981	0.7370	0.8832	0.072*
C3	0.46936 (12)	0.6045 (2)	0.7882 (2)	0.0444 (6)
C4	0.5000	0.3574 (3)	0.7500	0.0437 (8)
C5	0.38571 (13)	0.4045 (2)	0.8743 (2)	0.0461 (7)
C6	0.34437 (13)	0.2572 (2)	0.8323 (2)	0.0529 (7)
N1	0.44820 (10)	0.45224 (19)	0.80638 (19)	0.0430 (6)
O1	0.5000	0.2222 (2)	0.7500	0.0654 (8)
O2	0.36526 (12)	0.4856 (2)	0.9606 (2)	0.0703 (7)
C7	0.31257 (19)	0.2319 (4)	0.6791 (2)	0.0781 (10)
H7A	0.3577	0.2117	0.6345	0.117*
H7B	0.2836	0.3201	0.6371	0.117*
H7C	0.2757	0.1472	0.6655	0.117*
C8	0.3311 (2)	0.1649 (4)	0.9330 (3)	0.0889 (11)
H8A	0.2999	0.0779	0.9084	0.107*
H8B	0.3529	0.1870	1.0284	0.107*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0787 (19)	0.0326 (12)	0.117 (3)	0.0049 (10)	0.0282 (17)	−0.0064 (13)

C2	0.0603 (15)	0.0416 (13)	0.0812 (19)	0.0069 (10)	0.0217 (14)	−0.0045 (11)
C3	0.0411 (11)	0.0326 (12)	0.0578 (14)	0.0014 (7)	0.0071 (10)	0.0007 (8)
C4	0.0413 (15)	0.0329 (14)	0.0592 (19)	0.000	0.0162 (13)	0.000
C5	0.0396 (11)	0.0463 (13)	0.0546 (14)	0.0070 (8)	0.0152 (10)	0.0035 (9)
C6	0.0398 (12)	0.0520 (13)	0.0710 (17)	−0.0020 (9)	0.0208 (11)	0.0046 (10)
N1	0.0389 (9)	0.0329 (10)	0.0599 (12)	0.0014 (6)	0.0166 (8)	0.0003 (7)
O1	0.0647 (16)	0.0316 (13)	0.111 (2)	0.000	0.0435 (15)	0.000
O2	0.0726 (13)	0.0674 (12)	0.0816 (14)	0.0023 (9)	0.0399 (11)	−0.0090 (9)
C7	0.0629 (17)	0.077 (2)	0.089 (2)	−0.0211 (13)	0.0050 (16)	0.0023 (15)
C8	0.100 (2)	0.081 (2)	0.095 (2)	−0.0197 (18)	0.0398 (19)	0.0097 (17)

Geometric parameters (Å, º)

C1—C1^{i}	1.382 (6)	C5—O2	1.203 (3)
C1—C2	1.385 (4)	C5—N1	1.410 (3)
C1—H1	0.9300	C5—C6	1.489 (3)
C2—C3	1.378 (3)	C6—C8	1.3242 (19)
C2—H2	0.9300	C6—C7	1.4689 (19)
C3—C3^{i}	1.383 (4)	C7—H7A	0.9600
C3—N1	1.415 (3)	C7—H7B	0.9600
C4—O1	1.198 (4)	C7—H7C	0.9600
C4—N1	1.397 (2)	C8—H8A	0.9300
C4—N1^{i}	1.397 (2)	C8—H8B	0.9300
C1^{i}—C1—C2	121.28 (17)	C8—C6—C7	124.0 (3)
C1^{i}—C1—H1	119.4	C8—C6—C5	119.0 (2)
C2—C1—H1	119.4	C7—C6—C5	116.65 (19)
C3—C2—C1	117.3 (3)	C4—N1—C5	125.50 (19)
C3—C2—H2	121.4	C4—N1—C3	109.53 (17)
C1—C2—H2	121.4	C5—N1—C3	124.95 (17)
C2—C3—C3^{i}	121.42 (15)	C6—C7—H7A	109.5
C2—C3—N1	131.2 (2)	C6—C7—H7B	109.5
C3^{i}—C3—N1	107.37 (11)	H7A—C7—H7B	109.5
O1—C4—N1	127.01 (12)	C6—C7—H7C	109.5
O1—C4—N1^{i}	127.01 (12)	H7A—C7—H7C	109.5
N1—C4—N1^{i}	106.0 (2)	H7B—C7—H7C	109.5
O2—C5—N1	119.4 (2)	C6—C8—H8A	120.0
O2—C5—C6	121.8 (2)	C6—C8—H8B	120.0
N1—C5—C6	118.77 (18)	H8A—C8—H8B	120.0
C1^{i}—C1—C2—C3	0.3 (6)	N1^{i}—C4—N1—C3	−1.51 (11)
C1—C2—C3—C3^{i}	1.4 (5)	O2—C5—N1—C4	153.7 (2)
C1—C2—C3—N1	−177.0 (2)	C6—C5—N1—C4	−28.9 (3)
O2—C5—C6—C8	−46.6 (4)	O2—C5—N1—C3	−24.6 (3)
N1—C5—C6—C8	136.0 (3)	C6—C5—N1—C3	152.8 (2)
O2—C5—C6—C7	126.7 (3)	C2—C3—N1—C4	−177.4 (2)
N1—C5—C6—C7	−50.7 (3)	C3^{i}—C3—N1—C4	4.0 (3)
O1—C4—N1—C5	0.0 (2)	C2—C3—N1—C5	1.1 (4)

N1^{i}—C4—N1—C5	180.0 (2)	C3^{i}—C3—N1—C5	−177.4 (2)
O1—C4—N1—C3	178.48 (11)		

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{ii}	0.93	2.57	3.193 (3)	124

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