



# Crystal structure and Hirshfeld surface analysis of 1-[[2-oxo-3-(prop-1-en-2-yl)-2,3-dihydro-1H-1,3-benzodiazol-1-yl]methyl]-3-(prop-1-en-2-yl)-2,3-dihydro-1H-1,3-benzodiazol-2-one

Asmaa Saber,<sup>a\*</sup> Nada Kheira Sebbar,<sup>b</sup> Tuncer Hökelek,<sup>c</sup> Brahim Hni,<sup>a</sup> Joel T. Mague<sup>d</sup> and El Mokhtar Essassi<sup>a</sup>

Received 23 October 2018

Accepted 28 October 2018

Edited by D.-J. Xu, Zhejiang University (Yuquan Campus), China

**Keywords:** crystal structure; hydrogen bond; benzodiazolone; Hirshfeld surface.

**CCDC reference:** 1875883

**Supporting information:** this article has supporting information at journals.iucr.org/e

<sup>a</sup>Laboratoire de Chimie Organique Hétérocyclique URAC 21, Pôle de Compétence Pharmacochimie, Av. Ibn Battouta, BP 1014, Faculté des Sciences, Université Mohammed V, Rabat, Morocco, <sup>b</sup>Laboratoire de Chimie Bioorganique Appliquée, Faculté des Sciences, Université Ibn Zohr, Agadir, Morocco, <sup>c</sup>Department of Physics, Hacettepe University, 06800 Beytepe, Ankara, Turkey, and <sup>d</sup>Department of Chemistry, Tulane University, New Orleans, LA 70118, USA.  
\*Correspondence e-mail: as.saber.pro@gmail.com

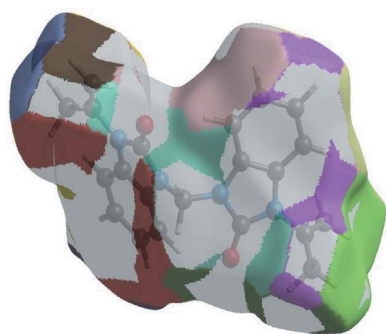
In the title compound, C<sub>21</sub>H<sub>20</sub>N<sub>4</sub>O<sub>2</sub>, the intramolecular C—H···O hydrogen-bonded benzodiazolone moieties are planar to within 0.017 (1) and 0.026 (1) Å, and are oriented at a dihedral angle of 57.35 (3)°. In the crystal, two sets of intermolecular C—H···O hydrogen bonds generate layers parallel to the *bc* plane. The Hirshfeld surface analysis of the crystal structure indicates that the most important contributions for the crystal packing are from H···H (51.8%), H···C/C···H (30.7%) and H···O/O···H (11.2%) interactions.

## 1. Chemical context

The benzimidazole unit is an important pharmacophore and a privileged structure in the functions of biological molecules. Benzimidazole derivatives have attracted considerable attention from researchers because their bioactive and pharmaceutical properties. Many members of this family are widely used as anticonvulsant, anti-fungal, analgesic, antimicrobial, anti-histaminic and hypnotic or anti-inflammatory agents (Ayhan-Kılıçgil *et al.*, 2007; Soderlind *et al.*, 1999; Luo *et al.*, 2011; Walia *et al.*, 2011; Navarrete-Vázquez *et al.*, 2001). Benzimidazolone derivatives also find commercial use as dyes for acrylic fibres. The search for new heterocyclic systems including the benzimidazolone moiety with biological activities therefore is of much current importance (Mondieig *et al.*, 2013; Lakhrissi *et al.*, 2008; Ouzidan *et al.*, 2011; Dardouri *et al.*, 2011). In this context, we are interested in the synthesis of the title compound, 1-[[2-oxo-3-(prop-1-en-2-yl)2,3-dihydro-1H-1,3-benzodiazol-1-yl]methyl]-3-(prop-1-en-2-yl)-2,3-dihydro-1H-1,3-benzodiazol-2-one, by reaction of dichloromethane with 1-(prop-1-en-2-yl)-1H-benzimidazol-2(3H)-one under phase-transfer catalysis (PTC) conditions using tetra-*n*-butylammonium bromide (TBAB) as catalyst and potassium carbonate as base. We report herein its crystal and molecular structures along with the Hirshfeld surface analysis.

## 2. Structural commentary

In the title compound (Fig. 1), the intramolecular C—H···O hydrogen-bonded (Table 1) benzodiazolone moieties are

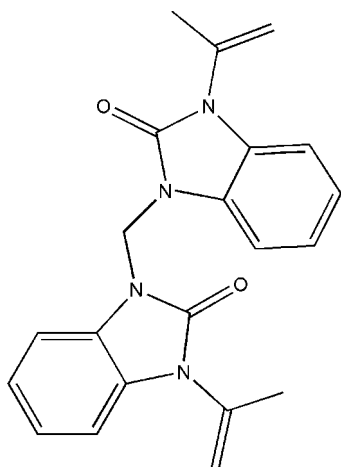


**Table 1**  
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5–H5 $\cdots$ O2	0.951 (14)	2.493 (14)	3.3598 (11)	151.4 (10)
C10–H10A $\cdots$ O1 <sup>i</sup>	0.986 (14)	2.596 (14)	3.3498 (12)	133.3 (11)
C10–H10B $\cdots$ O2 <sup>vi</sup>	0.998 (14)	2.488 (14)	3.4780 (12)	171.4 (11)
C13–H13 $\cdots$ O1	0.961 (13)	2.477 (13)	3.3381 (11)	149.1 (11)

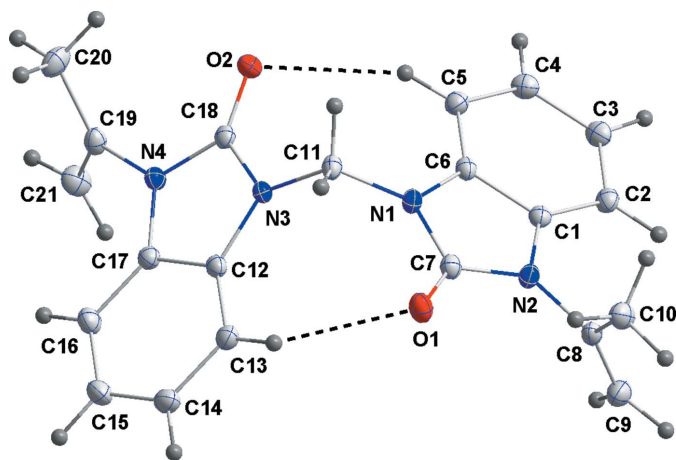
Symmetry codes: (i)  $-x, -y + 1, -z + 1$ ; (vi)  $-x, y + \frac{1}{2}, -z + \frac{1}{2}$ .

planar with the largest deviations being 0.017 (1) Å for atom C7 in the N1-containing unit (r.m.s. deviation = 0.011 Å) and 0.026 (1) Å for atom C18 in the N3-containing unit (r.m.s. deviation = 0.019 Å). The dihedral angle between the mean planes of the benzodiazolone moieties is 57.35 (3)°.

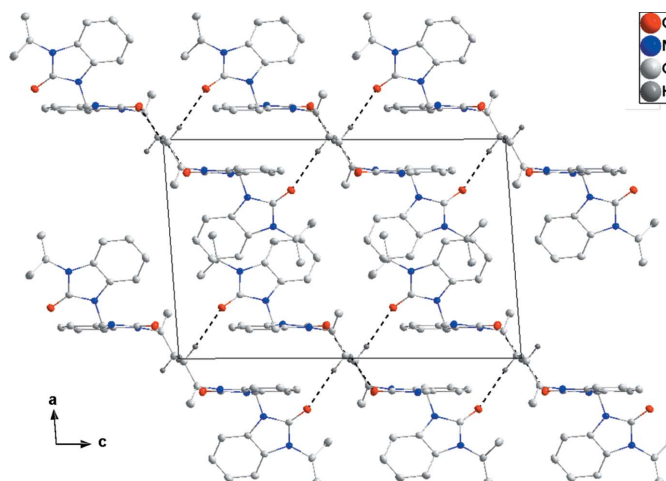


### 3. Supramolecular features

Hydrogen bonding and van der Waals contacts are the dominant interactions in the crystal packing. In the crystal, two sets of intermolecular C–H $\cdots$ O hydrogen bonds (Table 1) generate layers parallel to the  $bc$  plane. In these



**Figure 1**  
The title molecule with the labelling scheme and 50% probability ellipsoids. Intramolecular C–H $\cdots$ O hydrogen bonds are shown as dashed lines.

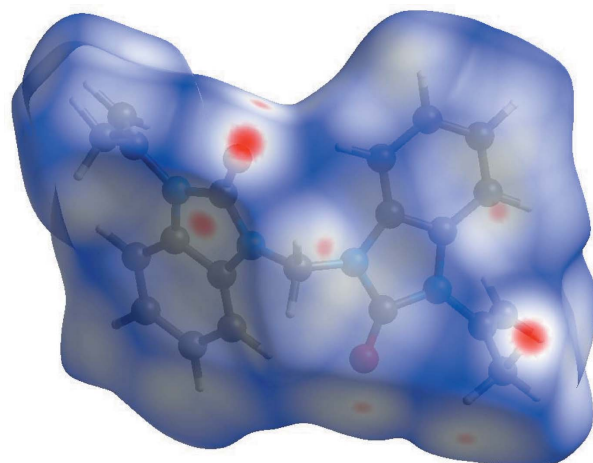


**Figure 2**  
The packing viewed along the  $b$ -axis direction giving an elevation view of two adjacent layers. Intermolecular C–H $\cdots$ O hydrogen bonds are shown as dashed lines.

layers, one of the benzodiazole units in each molecule is approximately parallel to the  $bc$  plane while the other half of the molecule protrudes from the surface (Fig. 2).

### 4. Hirshfeld surface analysis

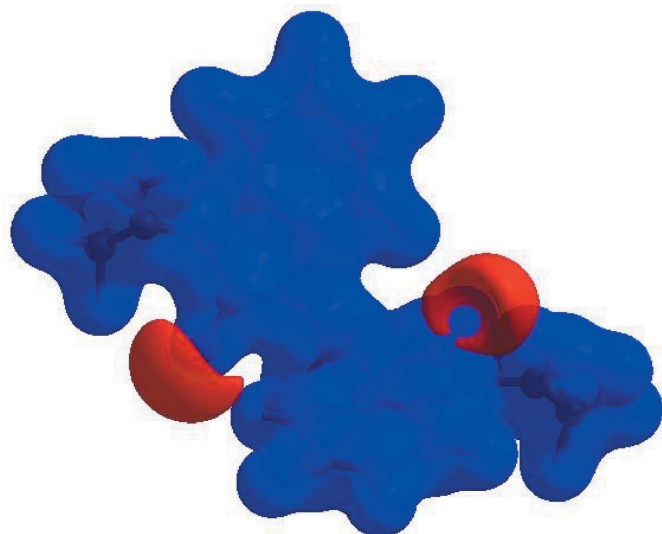
In order to visualize the intermolecular interactions in the crystal of the title compound, a Hirshfeld surface (HS) analysis (Hirshfeld, 1977; Spackman & Jayatilaka, 2009) was carried out by using *CrystalExplorer17.5* (Turner *et al.*, 2017). In the HS plotted over  $d_{\text{norm}}$  (Fig. 3), the white surface indicates contacts with distances equal to the sum of van der Waals radii, and the red and blue colours indicate distances shorter (in close contact) or longer (distinct contact) than the van der Waals radii (Venkatesan *et al.*, 2016). The bright-red spots appearing near O1, O2 and hydrogen atoms H5, H10A and H10B indicate their roles as the respective donors and



**Figure 3**  
View of the three-dimensional Hirshfeld surface of the title compound plotted over  $d_{\text{norm}}$  in the range  $-0.1476$  to  $1.2686$  a.u.

acceptors in the dominant C—H...O hydrogen bonds; they also appear as blue and red regions corresponding to positive and negative potentials on the HS mapped over electrostatic potential (Spackman *et al.*, 2008; Jayatilaka *et al.*, 2005) as shown in Fig. 4. The blue regions indicate positive electrostatic potential (hydrogen-bond donors), while the red regions indicate negative electrostatic potential (hydrogen-bond acceptors). The shape-index of the HS is a tool to visualize the  $\pi$ - $\pi$  stacking by the presence of adjacent red and blue triangles; if there are no adjacent red and/or blue triangles, then there are no  $\pi$ - $\pi$  interactions. Fig. 5 clearly indicates that no  $\pi$ - $\pi$  interactions are present in the title structure.

The overall two-dimensional fingerprint plot, Fig. 6a, and those delineated into H...H, H...C/C...H, H...O/O...H, H...N/N...H, C...C and N...C/C...N contacts (McKinnon *et al.*, 2007) are illustrated in Fig. 6b–g, respectively, together with their relative contributions to the Hirshfeld surface. The most important contribution to the overall crystal packing (51.8%) is from H...H interactions, which are shown in Fig. 6b as widely scattered points of high density due to the large hydrogen content of the molecule. The spike with the tip at  $d_e = d_i = 1.08$  Å in Fig. 6b is due to the short interatomic H...H contacts (Table 2). The fingerprint plot, Fig. 6c, delineated into H...C/C...H contacts, which make a 30.7% contribution to the HS, shows a pair of characteristic wings and a pair of spikes with the tips at  $d_e + d_i \sim 2.65$  Å. The H...O/O...H contacts in the structure with a 11.2% contribution to the HS have a symmetrical distribution of points, Fig. 6d, with the tips at  $d_e + d_i = 2.40$  Å arising from the short intra- and/or interatomic C—H...O hydrogen bonding (Table 1) as well as from the H...O/O...H contacts (Table 2). Finally, the H...N/N...H (Fig. 6e) contacts in the structure with a 5.1% con-



**Figure 4**  
View of the three-dimensional Hirshfeld surface of the title compound plotted over electrostatic potential energy in the range  $-0.0500$  to  $0.0500$  a.u. using the STO-3 G basis set at the Hartree–Fock level of theory. Hydrogen-bond donors and acceptors are shown as blue and red regions, respectively, around the atoms corresponding to positive and negative potentials.

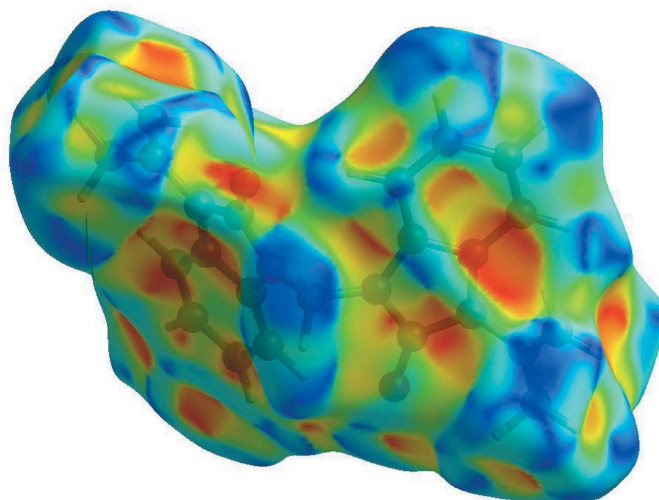
**Table 2**  
Selected interatomic distances (Å).

O1...C10	3.2233 (12)	C3...H11A <sup>vi</sup>	3.017 (12)
O1...C13	3.3381 (12)	C4...H20B <sup>vii</sup>	3.013 (14)
O1...C10 <sup>i</sup>	3.3499 (12)	C5...H11A	2.980 (12)
O2...C20	3.1500 (13)	C7...H13	3.084 (13)
O2...C5	3.3598 (12)	C7...H21B <sup>v</sup>	2.962 (14)
O1...H11B	2.510 (12)	C7...H10A	2.891 (14)
O1...H13	2.477 (13)	C8...H2	2.956 (13)
O1...H10A <sup>i</sup>	2.595 (14)	C9...H2	2.981 (13)
O1...H20C <sup>ii</sup>	2.917 (15)	C9...H15 <sup>ix</sup>	2.898 (13)
O1...H10A	2.667 (14)	C10...H9B <sup>x</sup>	3.051 (14)
O2...H3 <sup>iii</sup>	2.772 (13)	C11...H2 <sup>iii</sup>	3.077 (13)
O2...H5	2.493 (14)	C11...H13	3.009 (13)
O2...H11A	2.505 (12)	C11...H5	2.972 (13)
O2...H20C	2.581 (15)	C13...H11B	2.965 (12)
O2...H10B <sup>iv</sup>	2.487 (14)	C13...H9A <sup>iii</sup>	3.051 (14)
O2...H15 <sup>v</sup>	2.781 (14)	C14...H9A <sup>iii</sup>	2.989 (14)
N1...C3 <sup>iv</sup>	3.3814 (12)	C16...H21A	3.080 (14)
N2...C21 <sup>v</sup>	3.4318 (13)	C17...H21A	2.979 (14)
N3...H3 <sup>iii</sup>	2.938 (13)	C18...H21 <sup>v</sup>	2.858 (14)
N3...H16 <sup>v</sup>	2.920 (14)	C18...H5	3.093 (14)
C1...C21 <sup>v</sup>	3.5839 (13)	C18...H20C	2.852 (15)
C2...C11 <sup>vi</sup>	3.5159 (12)	C18...H3 <sup>iii</sup>	2.701 (13)
C2...C9	3.4314 (13)	C19...H16	2.977 (14)
C3...C18 <sup>vii</sup>	3.3908 (13)	C21...H16	2.874 (14)
C3...C11 <sup>vi</sup>	3.3994 (12)	H2...H11B <sup>vii</sup>	2.428 (18)
C7...C21 <sup>v</sup>	3.5253 (13)	H5...H11A	2.583 (18)
C16...C21	3.3315 (14)	H9B...H10C	2.495 (19)
C16...C18 <sup>viii</sup>	3.4599 (13)	H9B...H10B <sup>x</sup>	2.44 (2)
C1...H21A <sup>v</sup>	2.941 (14)	H9B...H15 <sup>ix</sup>	2.578 (19)
C1...H9A	3.064 (13)	H10C...H9B	2.495 (19)
C1...H11A <sup>vi</sup>	2.946 (12)	H20A...H21B	2.45 (2)
C2...H11A <sup>vi</sup>	2.786 (12)	H20A...H20A <sup>xi</sup>	2.34 (2)

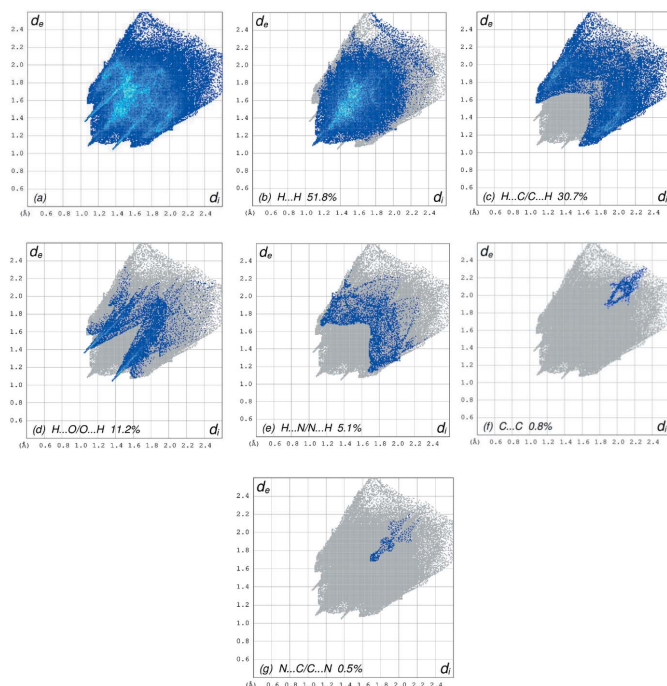
Symmetry codes: (i)  $-x, -y + 1, -z + 1$ ; (ii)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ ; (iii)  $x, y - 1, z$ ; (iv)  $-x, y - \frac{1}{2}, -z + \frac{1}{2}$ ; (v)  $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (vi)  $-x, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (vii)  $x, y + 1, z$ ; (viii)  $-x + 1,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

tribution to the HS also have a symmetrical distribution of points, with the pair of wings appearing at  $d_e + d_i = 2.80$  Å.

The Hirshfeld surface representations for the function  $d_{\text{norm}}$  are shown for the H...H, H...C/C...H, H...O/O...H and H...N/N...H interactions in Fig. 7a–d, respectively.

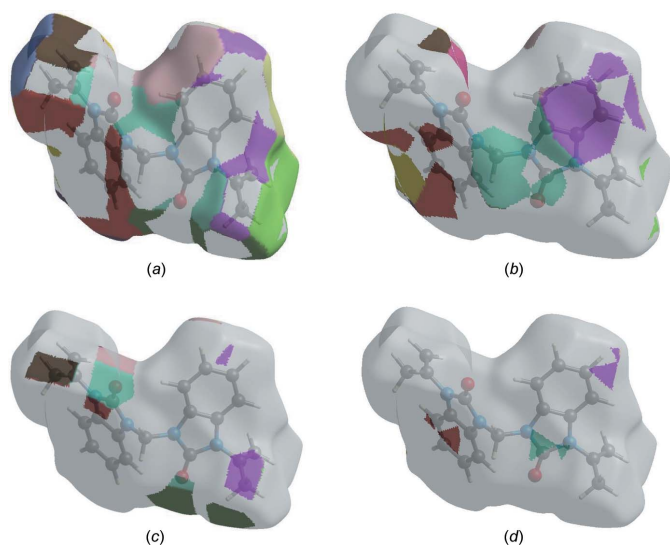


**Figure 5**  
Hirshfeld surface of the title compound plotted over shape-index.

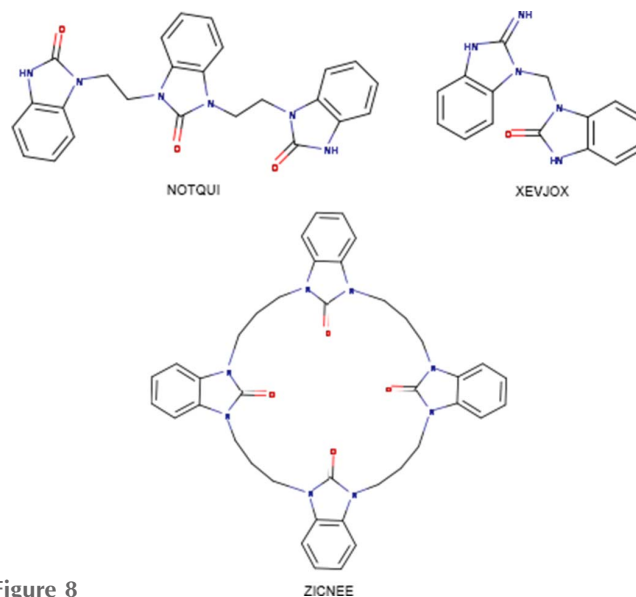

**Figure 6**

The full two-dimensional fingerprint plots for the title compound, showing (a) all interactions, and delineated into (b) H...H, (c) H...C/C...H, (d) H...O/O...H, (e) H...N/N...H, (f) C...C and (g) N...C/C...N interactions. The  $d_i$  and  $d_e$  values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

The Hirshfeld surface analysis confirms the importance of H-atom contacts in establishing the packing. The large number of H...H, H...C/C...H and H...O/O...H interactions suggest that van der Waals interactions and hydrogen bonding play the major roles in the crystal packing (Hathwar *et al.*, 2015).


**Figure 7**

The Hirshfeld surface representations with the function  $d_{\text{norm}}$  plotted onto the surface for (a) H...H, (b) H...C/C...H, (c) H...O/O...H and (d) H...N/N...H interactions.


**Figure 8**  
 NOTQUI, XEVJOX and ZICNEE.

## 5. Database survey

A search of the Cambridge Structural Database (CSD, version 5.39, update of August 2018; Groom *et al.*, 2016) for benzimidazolin-2-one derivatives in which both nitrogen atoms form exocyclic C—N bonds gave 61 hits. In these structures, the bicyclic ring system is either planar, has a slight twist end-to-end or, in the cases where the exocyclic substituents form a ring, has a very shallow bowl shape. The closest examples to the title compound are NOTQUI (Díez-Barra *et al.*, 1997) and XEVJOX (Huang *et al.*, 2001) with ZICNEE (Shi & Thummel, 1995) as a more distant relative (see Fig. 8). In XEVJOX, the N—C—N angle connecting the two bicyclic units [114.19 (12)°] is essentially the same as in the title compound [114.04 (7)°]. In both of these, the bicyclic units are in an *anti* arrangement and this is basically the same for ZICNEE. Interestingly, the three bicyclic units in NOTQUI are close to all being *syn* to one another.

## 6. Synthesis and crystallization

To a solution of 1-(prop-1-en-2-yl)-1*H*-benzimidazol-2(3*H*)-one (2.87 mmol) in dichloromethane (30 ml) as reagent and solvent were added potassium carbonate (5.71 mmol) and a catalytic amount of tetra-*n*-butylammonium bromide (0.37 mmol). The mixture was heated for 24 h. The solid material was removed by filtration and the solvent evaporated under vacuum. The solid product was purified by recrystallization from ethanol solution to afford colourless crystals in 67% yield.

## 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were located in a difference-Fourier map and were freely refined.

Table 3

Experimental details.

Crystal data	
Chemical formula	C <sub>21</sub> H <sub>20</sub> N <sub>4</sub> O <sub>2</sub>
<i>M<sub>r</sub></i>	360.41
Crystal system, space group	Monoclinic, <i>P</i> <sub>2</sub> <sub>1</sub> / <i>c</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.5244 (5), 8.6312 (4), 17.9845 (8)
$\beta$ (°)	94.134 (1)
<i>V</i> (Å <sup>3</sup> )	1784.25 (14)
<i>Z</i>	4
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.09
Crystal size (mm)	0.40 × 0.39 × 0.22
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.89, 0.98
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	33699, 4912, 4269
<i>R<sub>int</sub></i>	0.027
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.696
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.039, 0.113, 1.09
No. of reflections	4912
No. of parameters	324
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.47, -0.18

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

### Funding information

JTM thanks Tulane University for support of the Tulane Crystallography Laboratory. TH is grateful to Hacettepe University Scientific Research Project Unit (grant No. 013 D04 602 004).

### References

Ayhan-Kılıçgil, G., Kus, G., Özdamar, E. D., Can-Eke, B. & Iscan, M. (2007). *Arch. Pharm. Chem. Life Sci.* **340**, 607–611.  
 Brandenburg, K. & Putz, H. (2012). *DIAMOND*, Crystal Impact GbR, Bonn, Germany.  
 Bruker (2016). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Dardouri, R., Rodi, Y. K., Saffon, N., Essassi, E. M. & Ng, S. W. (2011). *Acta Cryst.* **E67**, o1853.  
 Díez-Barra, E., Dotor, J., de la Hoz, A., Foces-Foces, C., Enjalbal, C., Aubagnac, J. L., Claramunt, R. M. & Elguero, J. (1997). *Tetrahedron*, **53**, 7689–7704.  
 Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.  
 Hathwar, V. R., Sist, M., Jørgensen, M. R. V., Mamakhel, A. H., Wang, X., Hoffmann, C. M., Sugimoto, K., Overgaard, J. & Iversen, B. B. (2015). *IUCrJ*, **2**, 563–574.  
 Hirshfeld, H. L. (1977). *Theor. Chim. Acta*, **44**, 129–138.  
 Huang, K. S., Haddadin, M. J., Olmstead, M. M. & Kurth, M. J. (2001). *J. Org. Chem.* **66**, 1310–1315.  
 Jayatilaka, D., Grimwood, D. J., Lee, A., Lemay, A., Russel, A. J., Taylor, C., Wolff, S. K., Cassam-Chenai, P. & Whitton, A. (2005). *TONTO - A System for Computational Chemistry*. Available at: <http://hirshfeldsurface.net/>  
 Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.  
 Lakhri, B., Benksim, A., Massoui, M., Essassi, E. M., Lequart, V., Joly, N., Beaupère, D., Wadouachi, A. & Martin, P. (2008). *Carbohydr. Res.* **343**, 421–433.  
 Luo, Y., Yao, J. P., Yang, L., Feng, C. L., Tang, W., Wang, G. F., Zuo, J. P. & Lu, W. (2011). *Arch. Pharm. Pharm. Med. Chem.* **344**, 78–83.  
 McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.* pp. 3814–3816.  
 Mondieig, D., Lakhri, L., El Assry, A., Lakhri, B., Negrier, P., Essassi, E. M., Massoui, M., Michel Leger, J. & Benali, B. (2013). *J. Mar. Chim. Heterocycl.* **12**, 51–61.  
 Navarrete-Vázquez, G., Cedillo, R., Hernández-Campos, A., Yépez, L., Hernández-Luis, F., Valdez, J., Morales, R., Corés, R., Hernandez, M. & Castillo, R. (2001). *Bioorg. Med. Chem. Lett.* **11**, 187–190.  
 Ouzidan, Y., Kandri Rodi, Y., Fronczek, F. R., Venkatraman, R., El Ammari, L. & Essassi, E. M. (2011). *Acta Cryst.* **E67**, o362–o363.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.  
 Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.  
 Shi, Z. & Thummel, R. P. (1995). *J. Org. Chem.* **60**, 5935–5945.  
 Soderlind, K. J., Gorodetsky, B., Singh, A. K., Bachur, N., Miller, G. G. & Lown, J. W. (1999). *Anticancer Drug. Des.* **14**, 19–36.  
 Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm*, **11**, 19–32.  
 Spackman, M. A., McKinnon, J. J. & Jayatilaka, D. (2008). *CrystEngComm*, **10**, 377–388.  
 Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *CrystalExplorer17*. The University of Western Australia.  
 Venkatesan, P., Thamocharan, S., Ilangovan, A., Liang, H. & Sundius, T. (2016). *Spectrochim. Acta Part A*, **153**, 625–636.  
 Walia, R., Hedaitullah, M., Naaz, S. F., Iqbal, K. & Lamba, H. S. (2011). *Int. J. Res. Pharm. Chem.* **1**, 565–574.

## supporting information

*Acta Cryst.* (2018). E74, 1746-1750 [https://doi.org/10.1107/S2056989018015219]

## Crystal structure and Hirshfeld surface analysis of 1-[[2-oxo-3-(prop-1-en-2-yl)-2,3-dihydro-1H-1,3-benzodiazol-1-yl]methyl]-3-(prop-1-en-2-yl)-2,3-dihydro-1H-1,3-benzodiazol-2-one

Asmaa Saber, Nada Kheira Sebbar, Tuncer Hökelek, Brahim Hni, Joel T. Mague and El Mokhtar Essassi

### Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

### 1-[[2-Oxo-3-(prop-1-en-2-yl)-2,3-dihydro-1H-1,3-benzodiazol-1-yl]methyl]-3-(prop-1-en-2-yl)-2,3-dihydro-1H-1,3-benzodiazol-2-one

#### Crystal data

$C_{21}H_{20}N_4O_2$

$M_r = 360.41$

Monoclinic,  $P2_1/c$

$a = 11.5244$  (5) Å

$b = 8.6312$  (4) Å

$c = 17.9845$  (8) Å

$\beta = 94.134$  (1)°

$V = 1784.25$  (14) Å<sup>3</sup>

$Z = 4$

$F(000) = 760$

$D_x = 1.342$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9896 reflections

$\theta = 2.3$ – $29.6$ °

$\mu = 0.09$  mm<sup>-1</sup>

$T = 100$  K

Block, colourless

$0.40 \times 0.39 \times 0.22$  mm

#### Data collection

Bruker SMART APEX CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Krause *et al.*, 2015)

$T_{\min} = 0.89$ ,  $T_{\max} = 0.98$

33699 measured reflections

4912 independent reflections

4269 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 29.7$ °,  $\theta_{\min} = 1.8$ °

$h = -15 \rightarrow 15$

$k = -11 \rightarrow 11$

$l = -25 \rightarrow 25$

Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.113$

$S = 1.09$

4912 reflections

324 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.248P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$

Special details

**Experimental.** The diffraction data were obtained from 3 sets of 400 frames, each of width  $0.5^\circ$  in  $\omega$ , collected at  $\varphi = 0.00, 90.00$  and  $180.00^\circ$  and 2 sets of 800 frames, each of width  $0.45^\circ$  in  $\varphi$ , collected at  $\omega = -30.00$  and  $210.00^\circ$ . The scan time was 15 sec/frame.

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.14989 (6)	0.48310 (8)	0.43717 (4)	0.01960 (15)
O2	0.23203 (6)	0.33229 (8)	0.13640 (4)	0.01728 (15)
N1	0.15509 (6)	0.53341 (9)	0.30979 (4)	0.01421 (16)
N2	0.14171 (6)	0.73261 (9)	0.38646 (4)	0.01478 (16)
N3	0.26416 (6)	0.31586 (9)	0.26564 (4)	0.01347 (16)
N4	0.39789 (6)	0.21797 (9)	0.19636 (4)	0.01465 (16)
C1	0.14167 (7)	0.79263 (10)	0.31433 (5)	0.01364 (17)
C2	0.13453 (8)	0.94353 (11)	0.28808 (5)	0.01628 (18)
H2	0.1277 (11)	1.0298 (15)	0.3234 (7)	0.020 (3)*
C3	0.13427 (8)	0.96453 (11)	0.21101 (5)	0.01748 (18)
H3	0.1274 (10)	1.0686 (15)	0.1888 (7)	0.021 (3)*
C4	0.14246 (8)	0.83917 (11)	0.16280 (5)	0.01769 (18)
H4	0.1443 (11)	0.8573 (15)	0.1091 (7)	0.021 (3)*
C5	0.15041 (8)	0.68681 (11)	0.18947 (5)	0.01611 (18)
H5	0.1568 (11)	0.5978 (16)	0.1588 (7)	0.024 (3)*
C6	0.14948 (7)	0.66652 (10)	0.26575 (5)	0.01342 (17)
C7	0.14860 (7)	0.57243 (10)	0.38459 (5)	0.01462 (17)
C8	0.12521 (8)	0.81967 (11)	0.45309 (5)	0.01582 (18)
C9	0.20574 (8)	0.92203 (12)	0.47637 (5)	0.02068 (19)
H9A	0.2757 (12)	0.9366 (16)	0.4506 (7)	0.027 (3)*
H9B	0.1940 (12)	0.9911 (16)	0.5186 (7)	0.027 (3)*
C10	0.01276 (8)	0.78748 (12)	0.48653 (5)	0.01963 (19)

H10A	0.0075 (12)	0.6777 (17)	0.5012 (8)	0.029 (3)*
H10B	-0.0541 (12)	0.8114 (16)	0.4499 (8)	0.026 (3)*
H10C	0.0073 (11)	0.8515 (16)	0.5326 (8)	0.027 (3)*
C11	0.15234 (7)	0.37476 (10)	0.28445 (5)	0.01449 (17)
H11A	0.0994 (10)	0.3665 (14)	0.2396 (7)	0.016 (3)*
H11B	0.1243 (11)	0.3110 (14)	0.3247 (7)	0.018 (3)*
C12	0.35153 (7)	0.25387 (10)	0.31484 (5)	0.01369 (17)
C13	0.36158 (8)	0.24451 (11)	0.39187 (5)	0.01675 (18)
H13	0.3032 (11)	0.2870 (16)	0.4215 (7)	0.022 (3)*
C14	0.46041 (9)	0.16942 (11)	0.42436 (5)	0.01995 (19)
H14	0.4683 (12)	0.1616 (16)	0.4794 (8)	0.027 (3)*
C15	0.54473 (8)	0.10818 (12)	0.38065 (5)	0.0208 (2)
H15	0.6129 (12)	0.0591 (17)	0.4037 (7)	0.031 (3)*
C16	0.53439 (8)	0.11942 (11)	0.30305 (5)	0.01861 (19)
H16	0.5928 (11)	0.0792 (17)	0.2729 (8)	0.028 (3)*
C17	0.43617 (7)	0.19291 (10)	0.27099 (5)	0.01441 (17)
C18	0.29128 (7)	0.29272 (10)	0.19241 (5)	0.01344 (17)
C19	0.45369 (8)	0.16277 (10)	0.13270 (5)	0.01655 (18)
C20	0.38248 (9)	0.05439 (13)	0.08298 (6)	0.0238 (2)
H20A	0.4249 (13)	0.0262 (18)	0.0389 (8)	0.040 (4)*
H20B	0.3693 (12)	-0.0429 (18)	0.1112 (8)	0.034 (4)*
H20C	0.3068 (13)	0.0959 (18)	0.0661 (8)	0.033 (3)*
C21	0.56303 (9)	0.20343 (13)	0.12450 (6)	0.0232 (2)
H21A	0.6039 (12)	0.2754 (17)	0.1589 (8)	0.028 (3)*
H21B	0.6045 (12)	0.1602 (16)	0.0827 (8)	0.030 (3)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0239 (3)	0.0166 (3)	0.0187 (3)	0.0040 (3)	0.0045 (3)	0.0038 (2)
O2	0.0170 (3)	0.0173 (3)	0.0171 (3)	0.0021 (2)	-0.0019 (2)	0.0004 (2)
N1	0.0163 (3)	0.0111 (4)	0.0153 (3)	0.0012 (3)	0.0022 (3)	-0.0005 (3)
N2	0.0180 (3)	0.0121 (4)	0.0143 (3)	0.0011 (3)	0.0021 (3)	-0.0008 (3)
N3	0.0124 (3)	0.0133 (3)	0.0146 (3)	0.0022 (3)	0.0004 (3)	-0.0010 (3)
N4	0.0134 (3)	0.0157 (4)	0.0149 (3)	0.0026 (3)	0.0016 (3)	-0.0004 (3)
C1	0.0114 (4)	0.0139 (4)	0.0156 (4)	-0.0004 (3)	0.0006 (3)	-0.0006 (3)
C2	0.0157 (4)	0.0132 (4)	0.0198 (4)	-0.0015 (3)	0.0004 (3)	-0.0014 (3)
C3	0.0171 (4)	0.0145 (4)	0.0206 (4)	-0.0030 (3)	-0.0002 (3)	0.0024 (3)
C4	0.0173 (4)	0.0187 (5)	0.0169 (4)	-0.0025 (3)	0.0001 (3)	0.0022 (3)
C5	0.0158 (4)	0.0163 (4)	0.0162 (4)	-0.0006 (3)	0.0008 (3)	-0.0016 (3)
C6	0.0111 (4)	0.0116 (4)	0.0175 (4)	0.0000 (3)	0.0007 (3)	-0.0003 (3)
C7	0.0129 (4)	0.0142 (4)	0.0169 (4)	0.0016 (3)	0.0024 (3)	-0.0005 (3)
C8	0.0178 (4)	0.0159 (4)	0.0138 (4)	0.0052 (3)	0.0008 (3)	-0.0013 (3)
C9	0.0204 (4)	0.0202 (5)	0.0213 (4)	0.0016 (4)	0.0007 (3)	-0.0049 (4)
C10	0.0173 (4)	0.0237 (5)	0.0181 (4)	0.0029 (3)	0.0031 (3)	-0.0017 (4)
C11	0.0114 (4)	0.0116 (4)	0.0206 (4)	0.0004 (3)	0.0018 (3)	-0.0020 (3)
C12	0.0134 (4)	0.0107 (4)	0.0168 (4)	0.0002 (3)	-0.0003 (3)	0.0000 (3)
C13	0.0190 (4)	0.0146 (4)	0.0167 (4)	0.0006 (3)	0.0011 (3)	-0.0017 (3)



C14	0.0237 (5)	0.0180 (4)	0.0175 (4)	0.0007 (3)	-0.0030 (3)	0.0004 (3)
C15	0.0192 (4)	0.0198 (5)	0.0226 (4)	0.0039 (3)	-0.0039 (3)	0.0015 (4)
C16	0.0153 (4)	0.0186 (4)	0.0218 (4)	0.0035 (3)	0.0010 (3)	0.0002 (3)
C17	0.0142 (4)	0.0129 (4)	0.0160 (4)	-0.0002 (3)	0.0003 (3)	-0.0003 (3)
C18	0.0133 (4)	0.0108 (4)	0.0162 (4)	-0.0008 (3)	0.0012 (3)	-0.0011 (3)
C19	0.0181 (4)	0.0157 (4)	0.0163 (4)	0.0027 (3)	0.0044 (3)	0.0008 (3)
C20	0.0235 (5)	0.0256 (5)	0.0231 (4)	-0.0021 (4)	0.0070 (4)	-0.0083 (4)
C21	0.0180 (4)	0.0292 (5)	0.0230 (5)	0.0011 (4)	0.0053 (4)	0.0011 (4)

*Geometric parameters (Å, °)*

O1—C7	1.2194 (11)	C8—C10	1.4936 (13)
O2—C18	1.2241 (11)	C9—H9A	0.967 (13)
N1—C7	1.3938 (11)	C9—H9B	0.982 (14)
N1—C6	1.3943 (11)	C10—H10A	0.986 (14)
N1—C11	1.4428 (11)	C10—H10B	0.998 (14)
N2—C7	1.3853 (12)	C10—H10C	1.001 (14)
N2—C1	1.3969 (11)	C11—H11A	0.978 (12)
N2—C8	1.4386 (11)	C11—H11B	0.982 (12)
N3—C18	1.3899 (11)	C12—C13	1.3843 (12)
N3—C12	1.3980 (11)	C12—C17	1.4003 (12)
N3—C11	1.4478 (11)	C13—C14	1.4010 (13)
N4—C18	1.3850 (11)	C13—H13	0.961 (13)
N4—C17	1.3991 (11)	C14—C15	1.3970 (14)
N4—C19	1.4343 (11)	C14—H14	0.991 (14)
C1—C2	1.3858 (12)	C15—C16	1.3956 (13)
C1—C6	1.4026 (12)	C15—H15	0.960 (14)
C2—C3	1.3976 (13)	C16—C17	1.3863 (12)
C2—H2	0.986 (12)	C16—H16	0.959 (14)
C3—C4	1.3940 (13)	C19—C21	1.3265 (13)
C3—H3	0.984 (13)	C19—C20	1.4975 (14)
C4—C5	1.4005 (13)	C20—H20A	0.992 (15)
C4—H4	0.980 (13)	C20—H20B	0.998 (15)
C5—C6	1.3839 (12)	C20—H20C	0.971 (15)
C5—H5	0.951 (14)	C21—H21A	0.973 (14)
C8—C9	1.3271 (14)	C21—H21B	0.993 (15)
O1...C10	3.2233 (12)	C3...H11A <sup>vi</sup>	3.017 (12)
O1...C13	3.3381 (12)	C4...H20B <sup>vii</sup>	3.013 (14)
O1...C10 <sup>i</sup>	3.3499 (12)	C5...H11A	2.980 (12)
O2...C20	3.1500 (13)	C7...H13	3.084 (13)
O2...C5	3.3598 (12)	C7...H21B <sup>v</sup>	2.962 (14)
O1...H11B	2.510 (12)	C7...H10A	2.891 (14)
O1...H13	2.477 (13)	C8...H2	2.956 (13)
O1...H10A <sup>i</sup>	2.595 (14)	C9...H2	2.981 (13)
O1...H20C <sup>ii</sup>	2.917 (15)	C9...H15 <sup>ix</sup>	2.898 (13)
O1...H10A	2.667 (14)	C10...H9B <sup>x</sup>	3.051 (14)
O2...H3 <sup>iii</sup>	2.772 (13)	C11...H2 <sup>iii</sup>	3.077 (13)

O2...H5	2.493 (14)	C11...H13	3.009 (13)
O2...H11A	2.505 (12)	C11...H5	2.972 (13)
O2...H20C	2.581 (15)	C13...H11B	2.965 (12)
O2...H10B <sup>iv</sup>	2.487 (14)	C13...H9A <sup>iii</sup>	3.051 (14)
O2...H15 <sup>v</sup>	2.781 (14)	C14...H9A <sup>iii</sup>	2.989 (14)
N1...C3 <sup>iv</sup>	3.3814 (12)	C16...H21A	3.080 (14)
N2...C21 <sup>v</sup>	3.4318 (13)	C17...H21A	2.979 (14)
N3...H3 <sup>iii</sup>	2.938 (13)	C18...H16 <sup>v</sup>	2.858 (14)
N3...H16 <sup>v</sup>	2.920 (14)	C18...H5	3.093 (14)
C1...C21 <sup>v</sup>	3.5839 (13)	C18...H20C	2.852 (15)
C2...C11 <sup>vi</sup>	3.5159 (12)	C18...H3 <sup>iii</sup>	2.701 (13)
C2...C9	3.4314 (13)	C19...H16	2.977 (14)
C3...C18 <sup>vii</sup>	3.3908 (13)	C21...H16	2.874 (14)
C3...C11 <sup>vi</sup>	3.3994 (12)	H2...H11B <sup>vii</sup>	2.428 (18)
C7...C21 <sup>v</sup>	3.5253 (13)	H5...H11A	2.583 (18)
C16...C21	3.3315 (14)	H9B...H10C	2.495 (19)
C16...C18 <sup>viii</sup>	3.4599 (13)	H9B...H10B <sup>x</sup>	2.44 (2)
C1...H21A <sup>v</sup>	2.941 (14)	H9B...H15 <sup>ix</sup>	2.578 (19)
C1...H9A	3.064 (13)	H10C...H9B	2.495 (19)
C1...H11A <sup>vi</sup>	2.946 (12)	H20A...H21B	2.45 (2)
C2...H11A <sup>vi</sup>	2.786 (12)	H20A...H20A <sup>xi</sup>	2.34 (2)
C7—N1—C6	110.21 (7)	C8—C10—H10C	109.8 (8)
C7—N1—C11	122.19 (7)	H10A—C10—H10C	107.5 (11)
C6—N1—C11	127.12 (7)	H10B—C10—H10C	109.9 (11)
C7—N2—C1	110.10 (7)	N1—C11—N3	114.04 (7)
C7—N2—C8	123.56 (7)	N1—C11—H11A	109.1 (7)
C1—N2—C8	126.06 (8)	N3—C11—H11A	107.2 (7)
C18—N3—C12	110.11 (7)	N1—C11—H11B	107.4 (7)
C18—N3—C11	122.45 (7)	N3—C11—H11B	108.7 (7)
C12—N3—C11	126.81 (7)	H11A—C11—H11B	110.4 (10)
C18—N4—C17	109.78 (7)	C13—C12—N3	131.31 (8)
C18—N4—C19	124.12 (7)	C13—C12—C17	122.02 (8)
C17—N4—C19	125.86 (7)	N3—C12—C17	106.66 (7)
C2—C1—N2	131.38 (8)	C12—C13—C14	116.83 (8)
C2—C1—C6	121.47 (8)	C12—C13—H13	121.4 (8)
N2—C1—C6	107.15 (8)	C14—C13—H13	121.7 (8)
C1—C2—C3	117.10 (8)	C15—C14—C13	121.19 (9)
C1—C2—H2	119.7 (7)	C15—C14—H14	121.3 (8)
C3—C2—H2	123.2 (7)	C13—C14—H14	117.5 (8)
C4—C3—C2	121.38 (8)	C16—C15—C14	121.58 (9)
C4—C3—H3	117.6 (7)	C16—C15—H15	118.0 (8)
C2—C3—H3	121.0 (7)	C14—C15—H15	120.4 (8)
C3—C4—C5	121.44 (8)	C17—C16—C15	117.13 (8)
C3—C4—H4	119.7 (8)	C17—C16—H16	121.1 (8)
C5—C4—H4	118.8 (8)	C15—C16—H16	121.8 (8)
C6—C5—C4	116.95 (8)	C16—C17—N4	131.39 (8)
C6—C5—H5	118.6 (8)	C16—C17—C12	121.25 (8)

C4—C5—H5	124.4 (8)	N4—C17—C12	107.33 (7)
C5—C6—N1	131.61 (8)	O2—C18—N4	127.76 (8)
C5—C6—C1	121.65 (8)	O2—C18—N3	126.13 (8)
N1—C6—C1	106.75 (7)	N4—C18—N3	106.11 (7)
O1—C7—N2	127.56 (8)	C21—C19—N4	119.01 (9)
O1—C7—N1	126.66 (8)	C21—C19—C20	125.66 (9)
N2—C7—N1	105.77 (7)	N4—C19—C20	115.23 (8)
C9—C8—N2	118.63 (8)	C19—C20—H20A	110.5 (9)
C9—C8—C10	127.17 (8)	C19—C20—H20B	108.6 (8)
N2—C8—C10	114.13 (8)	H20A—C20—H20B	107.6 (12)
C8—C9—H9A	121.6 (8)	C19—C20—H20C	113.4 (9)
C8—C9—H9B	121.1 (8)	H20A—C20—H20C	108.9 (12)
H9A—C9—H9B	117.2 (12)	H20B—C20—H20C	107.6 (12)
C8—C10—H10A	110.9 (8)	C19—C21—H21A	121.2 (8)
C8—C10—H10B	110.3 (8)	C19—C21—H21B	119.9 (8)
H10A—C10—H10B	108.4 (11)	H21A—C21—H21B	118.9 (12)
C7—N2—C1—C2	178.97 (9)	C18—N3—C11—N1	-106.27 (9)
C8—N2—C1—C2	4.89 (14)	C12—N3—C11—N1	83.78 (10)
C7—N2—C1—C6	-0.36 (9)	C18—N3—C12—C13	-177.76 (9)
C8—N2—C1—C6	-174.44 (8)	C11—N3—C12—C13	-6.78 (15)
N2—C1—C2—C3	-178.63 (8)	C18—N3—C12—C17	1.01 (9)
C6—C1—C2—C3	0.62 (13)	C11—N3—C12—C17	171.99 (8)
C1—C2—C3—C4	-0.86 (13)	N3—C12—C13—C14	178.01 (9)
C2—C3—C4—C5	0.49 (14)	C17—C12—C13—C14	-0.60 (13)
C3—C4—C5—C6	0.15 (13)	C12—C13—C14—C15	0.41 (14)
C4—C5—C6—N1	179.51 (9)	C13—C14—C15—C16	0.17 (15)
C4—C5—C6—C1	-0.39 (13)	C14—C15—C16—C17	-0.55 (15)
C7—N1—C6—C5	-178.70 (9)	C15—C16—C17—N4	-177.31 (9)
C11—N1—C6—C5	-6.53 (15)	C15—C16—C17—C12	0.36 (14)
C7—N1—C6—C1	1.21 (9)	C18—N4—C17—C16	177.76 (9)
C11—N1—C6—C1	173.38 (8)	C19—N4—C17—C16	3.22 (16)
C2—C1—C6—C5	0.00 (13)	C18—N4—C17—C12	-0.16 (10)
N2—C1—C6—C5	179.41 (8)	C19—N4—C17—C12	-174.69 (8)
C2—C1—C6—N1	-179.92 (8)	C13—C12—C17—C16	0.22 (14)
N2—C1—C6—N1	-0.51 (9)	N3—C12—C17—C16	-178.69 (8)
C1—N2—C7—O1	-179.53 (8)	C13—C12—C17—N4	178.40 (8)
C8—N2—C7—O1	-5.27 (14)	N3—C12—C17—N4	-0.51 (9)
C1—N2—C7—N1	1.08 (9)	C17—N4—C18—O2	-179.70 (9)
C8—N2—C7—N1	175.34 (7)	C19—N4—C18—O2	-5.05 (15)
C6—N1—C7—O1	179.19 (8)	C17—N4—C18—N3	0.76 (10)
C11—N1—C7—O1	6.57 (13)	C19—N4—C18—N3	175.42 (8)
C6—N1—C7—N2	-1.42 (9)	C12—N3—C18—O2	179.36 (9)
C11—N1—C7—N2	-174.04 (7)	C11—N3—C18—O2	7.92 (14)
C7—N2—C8—C9	120.14 (10)	C12—N3—C18—N4	-1.10 (9)
C1—N2—C8—C9	-66.54 (12)	C11—N3—C18—N4	-172.54 (7)
C7—N2—C8—C10	-62.69 (11)	C18—N4—C19—C21	127.96 (10)
C1—N2—C8—C10	110.64 (10)	C17—N4—C19—C21	-58.25 (13)

C7—N1—C11—N3	-105.28 (9)	C18—N4—C19—C20	-55.46 (12)
C6—N1—C11—N3	83.41 (10)	C17—N4—C19—C20	118.33 (10)

Symmetry codes: (i)  $-x, -y+1, -z+1$ ; (ii)  $x, -y+1/2, z+1/2$ ; (iii)  $x, y-1, z$ ; (iv)  $-x, y-1/2, -z+1/2$ ; (v)  $-x+1, y+1/2, -z+1/2$ ; (vi)  $-x, y+1/2, -z+1/2$ ; (vii)  $x, y+1, z$ ; (viii)  $-x+1, y-1/2, -z+1/2$ ; (ix)  $-x+1, -y+1, -z+1$ ; (x)  $-x, -y+2, -z+1$ ; (xi)  $-x+1, -y, -z$ .

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C5—H5 $\cdots$ O2	0.951 (14)	2.493 (14)	3.3598 (11)	151.4 (10)
C10—H10 <i>A</i> $\cdots$ O1 <sup>i</sup>	0.986 (14)	2.596 (14)	3.3498 (12)	133.3 (11)
C10—H10 <i>B</i> $\cdots$ O2 <sup>vi</sup>	0.998 (14)	2.488 (14)	3.4780 (12)	171.4 (11)
C13—H13 $\cdots$ O1	0.961 (13)	2.477 (13)	3.3381 (11)	149.1 (11)

Symmetry codes: (i)  $-x, -y+1, -z+1$ ; (vi)  $-x, y+1/2, -z+1/2$ .