

Polymorphic structures of 3-phenyl-1*H*-1,3-benzodiazol-2(3*H*)-one

Dabeen Hong and Kyounghoon Lee*

Department of Chemical Education and Research Institute of Natural Sciences, Gyeongsang National University, Gyeongsangnam-do 52828, Republic of Korea. *Correspondence e-mail: klee1@gnu.ac.kr

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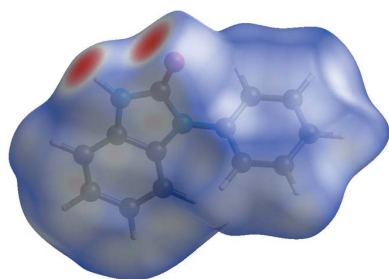
The polymorphic structures (**I** and **II**) of 3-phenyl-1*H*-1,3-benzodiazol-2(3*H*)-one, C₁₃H₁₀N₂O, acquired from pentane diffusion into the solution in THF, are reported. The structures show negligible differences in bond distances and angles, but the C–N–C–C torsion angles between the backbone and the phenyl substituent, 123.02 (15)° for **I** and 137.18 (11)° for **II**, are different. Compound **I** features a stronger C=O···H–N hydrogen bond than that in **II**, while the structure of **II** exhibits a stronger π–π interaction than in **I**, as confirmed by the shorter intercentroid distance [3.3257 (8) Å in **II** in comparison to 3.6862 (7) Å in **I**]. Overall, the supramolecular interactions of **I** and **II** are distinct, presumably originating from the variation in the dihedral angle.

1. Chemical context

Benzimidazolones are widely found in functional organic and biologically active molecules (Palin *et al.*, 2008; Monforte *et al.*, 2010; Pribut *et al.*, 2019; Bellenie *et al.*, 2020). For example, substituted benzimidazolones have been used as pigments due to their high fastness and resistance to light and weathering (Metz & Morgenroth, 2009). In addition, the biological activities of benzimidazolone derivatives have been tested for anticancer, HIV, pain regulation, *etc.* (Henning *et al.*, 1987; Elsinga *et al.*, 1997; Tapia *et al.*, 1999; Kawamoto *et al.*, 2001; Poulain *et al.*, 2001; Roger *et al.*, 2003; Dombroski *et al.*, 2004; Gustin *et al.*, 2005; Li *et al.*, 2005; Hammach *et al.*, 2006; Monforte *et al.*, 2009).

Singly N-substituted benzimidazolones exhibit interesting properties partially due to the hydrogen-bonding interactions between N–H···O=C moieties. *N*-phenyl-substituted benzimidazolone can be prepared by the intramolecular *N*-arylation of urea (Beyer *et al.*, 2011), carbonylation of 2-nitroaniline (Qi *et al.*, 2019), carbonylation of *o*-phenylenediamine with CO₂ (Yu *et al.*, 2013), carbonylation of iminophosphorane with CO₂ (Łukasik & Wróbel, 2016), iodophenyl-induced intramolecular Hofmann rearrangement of 2-(phenylamino)benzamide (Liu *et al.*, 2012), and carbonylation of N1-phenylbenzene-1,2-diamine with 1,1'-carbonyldiimidazole (Zhang *et al.*, 2008). Preparations of phenyl-substituted benzimidazolone have been reported using various reagents and catalysts, but the structure is unknown.

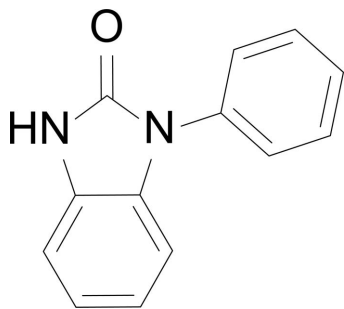
Here we report two polymorphic structures of 3-phenyl-1*H*-1,3-benzodiazol-2(3*H*)-one. The compound was prepared following the reported procedure using 1,1'-carbonyldiimidazole and N1-phenylbenzene-1,2-diamine in CH₂Cl₂ (Zhang *et al.*, 2008). Single crystals grown by pentane vapor



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diffusion into a THF solution formed colorless needles (**I**) and blocks (**II**).



2. Structural commentary

The title compounds crystallized as colorless needles (**I**) and blocks (**II**) in space groups $C2/c$ and $Pbca$, respectively. The two polymorphic structures exhibit identical bond distances and angles, except for the dihedral angle of the phenyl substituent (Fig. 1). Both structures retain the planarity of benzimidazolone moiety, as demonstrated by the low r.m.s. deviations of 0.009 and 0.023 Å for **I** and **II**, respectively. The C2–N1–C8–C9/C13 torsion angle is 123.03 (14) and -137.18 (12) $^\circ$ for **I** and **II**, respectively. No additional differences are observed from an analysis of bond distances and angles.

3. Supramolecular features

Initial investigations of supramolecular features for **I** and **II** were carried out using Hirshfeld surface analysis with *CrystalExplorer 21.5* (Spackman *et al.*, 2021). The Hirshfeld surface was mapped over d_{norm} in the ranges -0.6415 to 1.2040 a.u. and -0.5612 to 1.1830 a.u. for **I** and **II**, respectively (Figs. 2 and 3). The most intense red spots on the surface for **I** and **II** indicate the N3–H3 \cdots O1 hydrogen-bonding interactions (Tables 1 and 2), which have $R_2^2(8)$ graph-set motifs (Bernstein *et al.*, 1995). The shorter $D\cdots A$ and $H\cdots A$ distances, and more linear $D\text{--}H\cdots A$ angle reveal that the hydrogen-bonding interaction in **I** is stronger than that in **II**. In contrast, the structure of **II** contains a stronger $\pi\text{--}\pi$ interaction between the adjacent benzimidazolone moieties, as defined by the centroid \cdots centroid distance of 3.3257 (8) Å, while the corresponding distance in **I** is more elongated at 3.6862 (7) Å.

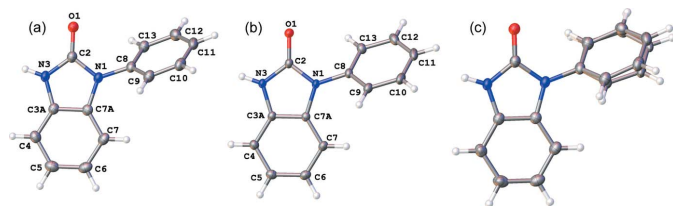


Figure 1

Molecular structures of (a) **I**, (b) **II**, and (c) overlay of **I** and **II** with displacement ellipsoids drawn at the 50% probability level.

Table 1
Hydrogen-bond geometry (Å, $^\circ$) for **I**.

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N3–H3 \cdots O1 ⁱ	0.88	1.91	2.7786 (14)	177

Symmetry code: (i) $-x + 1, -y + 2, -z + 1$.

Table 2
Hydrogen-bond geometry (Å, $^\circ$) for **II**.

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N3–H3 \cdots O1 ⁱ	0.88	2.00	2.8453 (13)	174

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

Minor intermolecular interactions are observed as faint red spots on the surface. The spots in **I** indicate the intermolecular interactions of C4 \cdots C2/C2 \cdots C4, C3A \cdots C3A and C7–H7/H7–C7, whereas those in **II** correspond to C2 \cdots C5/C5 \cdots C2, C4–H4 \cdots C12/ C12 \cdots H4–C4, C7A \cdots H6–C6/C6–H6 \cdots C7A, C3A \cdots H6–C6/C6–H6 \cdots C3A and C3A \cdots C6/C6 \cdots C3A contacts. The largest contributions to the Hirshfeld surface of **I** arises from H \cdots H (44.4%), C \cdots H/H \cdots C (31.9%), and O \cdots H/H \cdots O (13.5%) contacts, whereas the contributions for **II** are H \cdots H (45.8%), C \cdots H/H \cdots C (27.5%) and O \cdots H/H \cdots O (15.5%). Minor contributions include N \cdots H/H \cdots N (3.6%), C \cdots C (3.2%), C \cdots N/N \cdots C (2.1%), C \cdots O/O \cdots C (1.4%) for **I** and C \cdots C (5.4%), C \cdots N/N \cdots C (3.4%), N \cdots H/H \cdots N (3.2%), C \cdots O/O \cdots C (0.2%) for **II**.

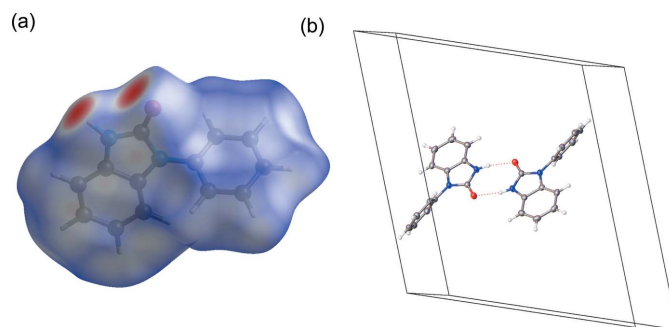


Figure 2

(a) Hirshfeld surface of **I** mapped over d_{norm} . (b) Partial packing plot of **I**.

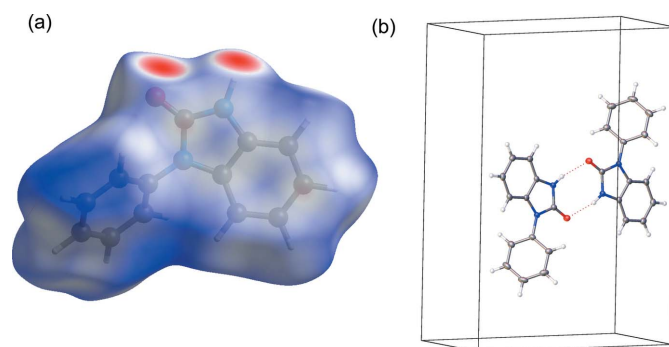


Figure 3

(a) Hirshfeld surface of **II** mapped over d_{norm} . (b) Partial packing plot of **II**.

Table 3
Experimental details.

	I	II
Crystal data		
Chemical formula	C ₁₃ H ₁₀ N ₂ O	C ₁₃ H ₁₀ N ₂ O
<i>M_r</i>	210.23	210.23
Crystal system, space group	Monoclinic, <i>C2/c</i>	Orthorhombic, <i>Pbca</i>
Temperature (K)	193	193
<i>a</i> , <i>b</i> , <i>c</i> (Å)	18.0187 (9), 6.4455 (3), 18.7315 (10)	13.7925 (3), 7.2652 (1), 19.7956 (4)
α , β , γ (°)	90, 111.181 (3), 90	90, 90, 90
<i>V</i> (Å ³)	2028.50 (18)	1983.62 (6)
<i>Z</i>	8	8
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.09	0.09
Crystal size (mm)	0.51 × 0.23 × 0.14	0.37 × 0.33 × 0.19
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.699, 0.746	0.712, 0.746
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	9328, 2350, 1956	34068, 2479, 2203
<i>R_{int}</i>	0.031	0.036
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.651	0.668
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.041, 0.104, 1.07	0.039, 0.099, 1.02
No. of reflections	2350	2479
No. of parameters	145	145
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.18, -0.23	0.25, -0.38

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

4. Database survey

A search for the title compound in the Cambridge Structural Database (CSD, Version 5.43, update of November 2022; Groom *et al.*, 2016) did not match any reported structures, including aryl-derivative searches. However, a survey for mono-N-substituted benzimidazolone compounds revealed 75 results, which included structures with simple substituents such as methyl (WIKPAJ; Rong *et al.*, 2013), *tert*-butyl (WIKNOV; Rong *et al.*, 2013), octyl (ZANXET; Belaziz, Kandri Rodi, Essassi *et al.*, 2012), nonyl (IJUGIE; Ouzidan, Kandri Rodi *et al.*, 2011), decyl (ESANAQ; Ait Elmachkouri *et al.*, 2021), dodecyl (SECBUZ; Belaziz, Kandri Rodi, Ouazzani Chahdi *et al.*, 2012), benzyl (EVEYIO; Ouzidan, Essassi *et al.*, 2011), 4-methylbenzyl (NEQBIW; Belaziz *et al.*, 2013), acetyl (VADYIM; Sebhaoui *et al.*, 2021) and a trifluoromethyl group (ZEDJAX; Bouayad-Gervais *et al.*, 2022). Most structures feature bimolecular hydrogen-bonding interactions between N–H ... O=C moieties with an *R*₂²(8) graph-set motif, but in ZEDJAX N–H ... O=C hydrogen bonds link the molecules into *C*(4) chains. The distances between a nitrogen donor and an oxygen acceptor range from 2.79–2.84 Å, comparable to the values for **I** and **II** of 2.7786 (14) and 2.8453 (14) Å, respectively.

5. Synthesis and crystallization

3-Phenyl-1*H*-1,3-benzodiazol-2(3*H*)-one was prepared following a reported procedure (Fig. 4; Zhang *et al.*, 2008; Mark *et al.*, 2013). A solution of 1,1'-carbonyldiimidazole

(0.50 g, 3.1 mmol) and 2-aminodiphenylamine (0.57 g, 3.1 mmol) in CH₂Cl₂ (15 mL) was stirred at room temperature overnight. The resulting white precipitate was filtered. An additional white precipitate was acquired by adding Et₂O (10 mL) into the filtrate. Combined yield: 0.30 g (46%). ¹H NMR (CDCl₃, 300 MHz): δ 10.75 (*br s*, NH, 1H), 7.58 (*m*, Ar, 4H), 7.45 (*m*, Ar, 1H), 7.17 (*m*, Ar, 1H), 7.10 (*m*, Ar, 1H), 7.06 (*m*, Ar, 2H). Pentane vapor diffusion into a solution of the compound in THF formed colorless needles and blocks.

6. Refinement

Crystal data, data collection, and refinement statistics are summarized in Table 3. No appreciable disorder was observed for both structures. The hydrogen atoms were optimized using riding models.

Acknowledgements

Dr Ji-Eun Lee (Gyeongsang National University) is gratefully acknowledged for collecting the single-crystal XRD data.

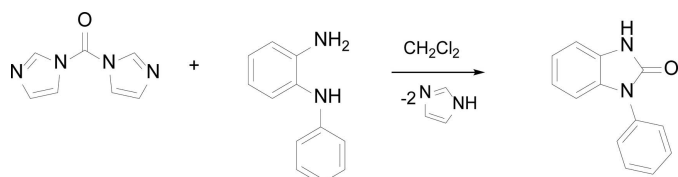


Figure 4
Synthesis of 3-phenyl-1*H*-1,3-benzodiazol-2(3*H*)-one.

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supporting information

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Polymorphic structures of 3-phenyl-1*H*-1,3-benzodiazol-2(3*H*)-one

Dabeen Hong and Kyoungsoon Lee

Computing details

For both structures, data collection: *APEX2* (Bruker, 2012); cell refinement: *S SAINT* (Bruker, 2012); data reduction: *S SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: Olex2 1.3 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 1.3 (Dolomanov *et al.*, 2009).

3-Phenyl-1*H*-1,3-benzodiazol-2(3*H*)-one (I)

Crystal data

$C_{13}H_{10}N_2O$

$M_r = 210.23$

Monoclinic, *C2/c*

$a = 18.0187$ (9) Å

$b = 6.4455$ (3) Å

$c = 18.7315$ (10) Å

$\beta = 111.181$ (3)°

$V = 2028.50$ (18) Å³

$Z = 8$

$F(000) = 880$

$D_x = 1.377$ Mg m⁻³

Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 2753 reflections

$\theta = 2.3$ – 27.5 °

$\mu = 0.09$ mm⁻¹

$T = 193$ K

NEEDLE, colourless

$0.51 \times 0.23 \times 0.14$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

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

$T_{\min} = 0.699$, $T_{\max} = 0.746$

9328 measured reflections

2350 independent reflections

1956 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 27.6$ °, $\theta_{\min} = 2.3$ °

$h = -23$ → 23

$k = -8$ → 8

$l = -24$ → 24

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.104$

$S = 1.07$

2350 reflections

145 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 1.4081P]$

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

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

$\Delta\rho_{\max} = 0.18$ e Å⁻³

$\Delta\rho_{\min} = -0.23$ e Å⁻³

Special details

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.60223 (5)	0.96614 (15)	0.57745 (5)	0.0239 (2)
N3	0.53005 (6)	0.77512 (18)	0.46886 (6)	0.0210 (3)
H3	0.488955	0.857863	0.448435	0.025*
N1	0.64477 (6)	0.64976 (18)	0.54578 (6)	0.0199 (3)
C8	0.71782 (7)	0.6314 (2)	0.60949 (7)	0.0200 (3)
C7A	0.61292 (7)	0.5048 (2)	0.48703 (7)	0.0198 (3)
C3A	0.54019 (7)	0.5865 (2)	0.43844 (7)	0.0202 (3)
C2	0.59267 (7)	0.8152 (2)	0.53505 (7)	0.0201 (3)
C9	0.73216 (8)	0.4585 (2)	0.65660 (7)	0.0242 (3)
H9	0.694564	0.348538	0.645522	0.029*
C13	0.77316 (7)	0.7904 (2)	0.62403 (7)	0.0230 (3)
H13	0.763240	0.907155	0.590917	0.028*
C7	0.64074 (8)	0.3170 (2)	0.47242 (8)	0.0236 (3)
H7	0.689738	0.261391	0.506014	0.028*
C4	0.49365 (8)	0.4809 (2)	0.37362 (7)	0.0238 (3)
H4	0.443989	0.534926	0.340804	0.029*
C5	0.52220 (8)	0.2924 (2)	0.35821 (8)	0.0274 (3)
H5	0.491700	0.217059	0.313667	0.033*
C10	0.80220 (8)	0.4481 (2)	0.72020 (8)	0.0275 (3)
H10	0.812386	0.330940	0.753147	0.033*
C6	0.59439 (8)	0.2116 (2)	0.40654 (8)	0.0266 (3)
H6	0.612378	0.082452	0.394429	0.032*
C12	0.84327 (8)	0.7775 (2)	0.68750 (8)	0.0274 (3)
H12	0.881670	0.885313	0.697783	0.033*
C11	0.85719 (8)	0.6075 (2)	0.73576 (7)	0.0283 (3)
H11	0.904654	0.600345	0.779703	0.034*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0232 (5)	0.0213 (5)	0.0244 (5)	0.0026 (4)	0.0052 (4)	-0.0008 (4)
N3	0.0185 (5)	0.0202 (6)	0.0217 (5)	0.0040 (4)	0.0041 (4)	0.0023 (4)
N1	0.0177 (5)	0.0193 (6)	0.0223 (5)	0.0027 (4)	0.0068 (4)	0.0014 (4)
C8	0.0177 (6)	0.0243 (8)	0.0193 (6)	0.0038 (5)	0.0083 (5)	0.0019 (5)
C7A	0.0191 (6)	0.0213 (7)	0.0216 (6)	-0.0015 (5)	0.0105 (5)	0.0018 (5)
C3A	0.0205 (6)	0.0207 (7)	0.0218 (6)	0.0006 (5)	0.0106 (5)	0.0032 (5)
C2	0.0195 (6)	0.0207 (7)	0.0209 (6)	0.0012 (5)	0.0081 (5)	0.0031 (5)
C9	0.0234 (6)	0.0248 (8)	0.0269 (7)	0.0031 (6)	0.0122 (5)	0.0037 (6)
C13	0.0221 (6)	0.0232 (8)	0.0244 (6)	0.0019 (5)	0.0095 (5)	0.0030 (5)

C7	0.0216 (6)	0.0236 (8)	0.0300 (7)	0.0022 (5)	0.0145 (5)	0.0029 (6)
C4	0.0229 (6)	0.0272 (8)	0.0210 (6)	-0.0022 (6)	0.0076 (5)	0.0025 (5)
C5	0.0333 (7)	0.0278 (9)	0.0251 (7)	-0.0077 (6)	0.0152 (6)	-0.0037 (6)
C10	0.0300 (7)	0.0295 (9)	0.0239 (6)	0.0100 (6)	0.0109 (5)	0.0071 (6)
C6	0.0322 (7)	0.0221 (8)	0.0327 (7)	-0.0013 (6)	0.0203 (6)	-0.0021 (6)
C12	0.0220 (6)	0.0306 (9)	0.0288 (7)	-0.0010 (6)	0.0083 (5)	-0.0044 (6)
C11	0.0232 (6)	0.0369 (9)	0.0211 (6)	0.0088 (6)	0.0037 (5)	-0.0022 (6)

Geometric parameters (Å, °)

O1—C2	1.2282 (16)	C13—H13	0.9500
N3—H3	0.8800	C13—C12	1.3896 (18)
N3—C3A	1.3824 (18)	C7—H7	0.9500
N3—C2	1.3660 (16)	C7—C6	1.3920 (19)
N1—C8	1.4266 (15)	C4—H4	0.9500
N1—C7A	1.3988 (17)	C4—C5	1.390 (2)
N1—C2	1.3864 (17)	C5—H5	0.9500
C8—C9	1.3868 (19)	C5—C6	1.390 (2)
C8—C13	1.3867 (19)	C10—H10	0.9500
C7A—C3A	1.4004 (17)	C10—C11	1.384 (2)
C7A—C7	1.375 (2)	C6—H6	0.9500
C3A—C4	1.3803 (18)	C12—H12	0.9500
C9—H9	0.9500	C12—C11	1.384 (2)
C9—C10	1.3893 (18)	C11—H11	0.9500
C3A—N3—H3	124.7	C12—C13—H13	120.3
C2—N3—H3	124.7	C7A—C7—H7	121.3
C2—N3—C3A	110.58 (11)	C7A—C7—C6	117.46 (12)
C7A—N1—C8	126.52 (11)	C6—C7—H7	121.3
C2—N1—C8	123.83 (11)	C3A—C4—H4	121.3
C2—N1—C7A	109.60 (10)	C3A—C4—C5	117.46 (12)
C9—C8—N1	120.15 (12)	C5—C4—H4	121.3
C13—C8—N1	118.93 (12)	C4—C5—H5	119.3
C13—C8—C9	120.91 (12)	C4—C5—C6	121.45 (13)
N1—C7A—C3A	106.37 (12)	C6—C5—H5	119.3
C7—C7A—N1	131.98 (12)	C9—C10—H10	119.9
C7—C7A—C3A	121.64 (12)	C11—C10—C9	120.28 (14)
N3—C3A—C7A	107.14 (11)	C11—C10—H10	119.9
C4—C3A—N3	131.89 (12)	C7—C6—H6	119.5
C4—C3A—C7A	120.97 (13)	C5—C6—C7	121.00 (14)
O1—C2—N3	127.84 (12)	C5—C6—H6	119.5
O1—C2—N1	125.88 (11)	C13—C12—H12	120.0
N3—C2—N1	106.28 (11)	C11—C12—C13	120.03 (13)
C8—C9—H9	120.4	C11—C12—H12	120.0
C8—C9—C10	119.16 (14)	C10—C11—C12	120.23 (12)
C10—C9—H9	120.4	C10—C11—H11	119.9
C8—C13—H13	120.3	C12—C11—H11	119.9
C8—C13—C12	119.38 (13)		

N3—C3A—C4—C5	178.80 (13)	C3A—N3—C2—O1	-178.55 (13)
N1—C8—C9—C10	-177.25 (12)	C3A—N3—C2—N1	1.45 (14)
N1—C8—C13—C12	177.74 (12)	C3A—C7A—C7—C6	0.98 (19)
N1—C7A—C3A—N3	-0.29 (13)	C3A—C4—C5—C6	0.7 (2)
N1—C7A—C3A—C4	179.34 (11)	C2—N3—C3A—C7A	-0.73 (14)
N1—C7A—C7—C6	-178.35 (12)	C2—N3—C3A—C4	179.69 (13)
C8—N1—C7A—C3A	178.81 (11)	C2—N1—C8—C9	123.03 (14)
C8—N1—C7A—C7	-1.8 (2)	C2—N1—C8—C13	-55.86 (17)
C8—N1—C2—O1	0.7 (2)	C2—N1—C7A—C3A	1.19 (14)
C8—N1—C2—N3	-179.32 (11)	C2—N1—C7A—C7	-179.40 (13)
C8—C9—C10—C11	-0.6 (2)	C9—C8—C13—C12	-1.1 (2)
C8—C13—C12—C11	-0.3 (2)	C9—C10—C11—C12	-0.8 (2)
C7A—N1—C8—C9	-54.27 (17)	C13—C8—C9—C10	1.62 (19)
C7A—N1—C8—C13	126.85 (14)	C13—C12—C11—C10	1.3 (2)
C7A—N1—C2—O1	178.38 (12)	C7—C7A—C3A—N3	-179.77 (11)
C7A—N1—C2—N3	-1.62 (14)	C7—C7A—C3A—C4	-0.14 (19)
C7A—C3A—C4—C5	-0.72 (19)	C4—C5—C6—C7	0.1 (2)
C7A—C7—C6—C5	-0.96 (19)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N3—H3...O1 ⁱ	0.88	1.91	2.7786 (14)	177

Symmetry code: (i) $-x+1, -y+2, -z+1$.

(II)

*Crystal data*C₁₃H₁₀N₂O $M_r = 210.23$ Orthorhombic, *Pbca* $a = 13.7925$ (3) Å $b = 7.2652$ (1) Å $c = 19.7956$ (4) Å $V = 1983.62$ (6) Å³ $Z = 8$ $F(000) = 880$ $D_x = 1.408$ Mg m⁻³Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 9912 reflections

 $\theta = 2.5$ – 28.3° $\mu = 0.09$ mm⁻¹ $T = 193$ K

BLOCK, colourless

 $0.37 \times 0.33 \times 0.19$ mm*Data collection*Bruker APEXII CCD
diffractometer φ and ω scansAbsorption correction: multi-scan
(SADABS; Krause *et al.*, 2015) $T_{\min} = 0.712$, $T_{\max} = 0.746$

34068 measured reflections

2479 independent reflections

2203 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.036$ $\theta_{\text{max}} = 28.4^\circ$, $\theta_{\text{min}} = 2.1^\circ$ $h = -18 \rightarrow 18$ $k = -8 \rightarrow 9$ $l = -26 \rightarrow 26$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$ $wR(F^2) = 0.099$ $S = 1.02$

2479 reflections

145 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 1.3866P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$ *Special details*

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.45568 (7)	0.03382 (12)	0.59030 (4)	0.0202 (2)
N1	0.38806 (7)	0.32974 (13)	0.58960 (5)	0.0156 (2)
N3	0.43199 (7)	0.20514 (13)	0.49242 (5)	0.0162 (2)
H3	0.452728	0.125653	0.462151	0.019*
C7A	0.37019 (8)	0.46038 (16)	0.53914 (6)	0.0150 (2)
C2	0.42885 (8)	0.17358 (16)	0.56054 (6)	0.0162 (2)
C8	0.37484 (8)	0.35772 (16)	0.66031 (6)	0.0164 (2)
C3A	0.39783 (8)	0.38031 (16)	0.47791 (6)	0.0152 (2)
C7	0.33606 (8)	0.63906 (16)	0.54215 (6)	0.0176 (2)
H7	0.319418	0.694844	0.583974	0.021*
C4	0.38813 (8)	0.47304 (17)	0.41748 (6)	0.0178 (2)
H4	0.405896	0.417587	0.375818	0.021*
C5	0.35117 (8)	0.65146 (17)	0.42007 (6)	0.0195 (2)
H5	0.342230	0.718091	0.379252	0.023*
C9	0.44915 (9)	0.31474 (17)	0.70512 (6)	0.0198 (2)
H9	0.507674	0.261337	0.689104	0.024*
C13	0.28837 (9)	0.43311 (16)	0.68360 (6)	0.0203 (3)
H13	0.237403	0.460514	0.652926	0.024*
C6	0.32707 (8)	0.73401 (17)	0.48132 (6)	0.0192 (2)
H6	0.304090	0.857272	0.481620	0.023*
C12	0.27728 (10)	0.46798 (17)	0.75227 (7)	0.0257 (3)
H12	0.218579	0.520089	0.768528	0.031*
C11	0.35128 (11)	0.42719 (17)	0.79701 (6)	0.0268 (3)
H11	0.343401	0.451607	0.843835	0.032*
C10	0.43697 (10)	0.35064 (18)	0.77346 (6)	0.0245 (3)
H10	0.487637	0.322592	0.804296	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0276 (5)	0.0164 (4)	0.0168 (4)	0.0051 (3)	0.0006 (3)	-0.0002 (3)
N1	0.0173 (4)	0.0150 (4)	0.0145 (4)	0.0021 (4)	0.0005 (3)	-0.0022 (4)
N3	0.0188 (5)	0.0157 (5)	0.0142 (4)	0.0028 (4)	0.0004 (4)	-0.0022 (4)
C7A	0.0122 (5)	0.0172 (5)	0.0156 (5)	-0.0013 (4)	-0.0007 (4)	-0.0008 (4)
C2	0.0157 (5)	0.0168 (5)	0.0161 (5)	0.0004 (4)	0.0004 (4)	-0.0027 (4)
C8	0.0215 (5)	0.0137 (5)	0.0140 (5)	-0.0014 (4)	0.0023 (4)	-0.0020 (4)
C3A	0.0119 (5)	0.0160 (5)	0.0178 (5)	0.0001 (4)	-0.0001 (4)	-0.0025 (4)
C7	0.0148 (5)	0.0177 (5)	0.0203 (5)	0.0006 (4)	0.0012 (4)	-0.0035 (4)
C4	0.0166 (5)	0.0208 (6)	0.0161 (5)	0.0002 (4)	0.0004 (4)	-0.0011 (4)
C5	0.0167 (5)	0.0215 (6)	0.0202 (6)	-0.0001 (5)	-0.0006 (4)	0.0039 (5)
C9	0.0219 (6)	0.0199 (5)	0.0176 (5)	-0.0022 (5)	0.0005 (4)	0.0000 (4)
C13	0.0241 (6)	0.0162 (5)	0.0205 (6)	0.0009 (5)	0.0031 (5)	-0.0014 (4)
C6	0.0151 (5)	0.0166 (5)	0.0258 (6)	0.0011 (4)	0.0004 (4)	0.0006 (5)
C12	0.0353 (7)	0.0180 (6)	0.0237 (6)	0.0025 (5)	0.0117 (5)	-0.0024 (5)
C11	0.0460 (8)	0.0188 (6)	0.0156 (5)	-0.0047 (6)	0.0058 (5)	-0.0031 (5)
C10	0.0340 (7)	0.0230 (6)	0.0166 (6)	-0.0066 (5)	-0.0028 (5)	0.0013 (5)

Geometric parameters (Å, °)

O1—C2	1.2309 (14)	C4—H4	0.9500
N1—C7A	1.3997 (15)	C4—C5	1.3939 (17)
N1—C2	1.3908 (14)	C5—H5	0.9500
N1—C8	1.4262 (14)	C5—C6	1.3928 (17)
N3—H3	0.8800	C9—H9	0.9500
N3—C2	1.3685 (15)	C9—C10	1.3880 (17)
N3—C3A	1.3872 (15)	C13—H13	0.9500
C7A—C3A	1.3976 (15)	C13—C12	1.3912 (17)
C7A—C7	1.3821 (16)	C6—H6	0.9500
C8—C9	1.3910 (17)	C12—H12	0.9500
C8—C13	1.3911 (16)	C12—C11	1.383 (2)
C3A—C4	1.3793 (16)	C11—H11	0.9500
C7—H7	0.9500	C11—C10	1.387 (2)
C7—C6	1.3933 (17)	C10—H10	0.9500
C7A—N1—C8	125.54 (10)	C5—C4—H4	121.4
C2—N1—C7A	109.23 (9)	C4—C5—H5	119.3
C2—N1—C8	125.02 (10)	C6—C5—C4	121.32 (11)
C2—N3—H3	124.8	C6—C5—H5	119.3
C2—N3—C3A	110.31 (9)	C8—C9—H9	120.3
C3A—N3—H3	124.8	C10—C9—C8	119.35 (12)
C3A—C7A—N1	106.78 (10)	C10—C9—H9	120.3
C7—C7A—N1	131.77 (11)	C8—C13—H13	120.3
C7—C7A—C3A	121.41 (11)	C8—C13—C12	119.33 (12)
O1—C2—N1	126.63 (11)	C12—C13—H13	120.3
O1—C2—N3	126.89 (11)	C7—C6—H6	119.4

N3—C2—N1	106.48 (10)	C5—C6—C7	121.19 (11)
C9—C8—N1	119.98 (10)	C5—C6—H6	119.4
C9—C8—C13	120.59 (11)	C13—C12—H12	119.8
C13—C8—N1	119.41 (10)	C11—C12—C13	120.36 (12)
N3—C3A—C7A	107.15 (10)	C11—C12—H12	119.8
C4—C3A—N3	131.35 (11)	C12—C11—H11	120.0
C4—C3A—C7A	121.50 (11)	C12—C11—C10	119.96 (12)
C7A—C7—H7	121.4	C10—C11—H11	120.0
C7A—C7—C6	117.27 (11)	C9—C10—H10	119.8
C6—C7—H7	121.4	C11—C10—C9	120.41 (12)
C3A—C4—H4	121.4	C11—C10—H10	119.8
C3A—C4—C5	117.24 (11)		
N1—C7A—C3A—N3	-0.32 (12)	C8—N1—C7A—C3A	176.62 (10)
N1—C7A—C3A—C4	179.08 (10)	C8—N1—C7A—C7	-1.01 (19)
N1—C7A—C7—C6	179.56 (11)	C8—N1—C2—O1	3.36 (19)
N1—C8—C9—C10	177.03 (11)	C8—N1—C2—N3	-177.43 (10)
N1—C8—C13—C12	-177.13 (11)	C8—C9—C10—C11	0.53 (19)
N3—C3A—C4—C5	-179.65 (11)	C8—C13—C12—C11	-0.35 (19)
C7A—N1—C2—O1	178.21 (11)	C3A—N3—C2—O1	-178.39 (11)
C7A—N1—C2—N3	-2.59 (12)	C3A—N3—C2—N1	2.40 (12)
C7A—N1—C8—C9	-129.31 (12)	C3A—C7A—C7—C6	2.22 (16)
C7A—N1—C8—C13	48.81 (16)	C3A—C4—C5—C6	1.38 (17)
C7A—C3A—C4—C5	1.11 (17)	C7—C7A—C3A—N3	177.61 (10)
C7A—C7—C6—C5	0.27 (17)	C7—C7A—C3A—C4	-2.99 (17)
C2—N1—C7A—C3A	1.81 (12)	C4—C5—C6—C7	-2.11 (18)
C2—N1—C7A—C7	-175.82 (12)	C9—C8—C13—C12	0.98 (18)
C2—N1—C8—C9	44.70 (17)	C13—C8—C9—C10	-1.07 (18)
C2—N1—C8—C13	-137.18 (12)	C13—C12—C11—C10	-0.2 (2)
C2—N3—C3A—C7A	-1.31 (13)	C12—C11—C10—C9	0.1 (2)
C2—N3—C3A—C4	179.37 (12)		

Hydrogen-bond geometry (\AA , $^\circ$)

<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>
N3—H3 \cdots O1 ⁱ	0.88	2.00	2.8453 (13)	174

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