



Synthesis, characterization, and crystal structure of 2-(2-azidophenyl)-3-oxo-3*H*-indole 1-oxide

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An attempt to explore the reactivity of the nitro group in the presence of gold catalysis in comparison to the azide group yielded intriguing results. Surprisingly, only the nitro group exhibited reactivity, ultimately giving rise to the formation of the title isatogen, C₁₄H₈N₄O₂. In the crystal structure, weak C—H···O hydrogen bonds and π – π stacking interactions link the molecules. The structure exhibits disorder of the molecule.

1. Chemical context

2,2-Disubstituted indolin-3-ones play a crucial role as fundamental structural motifs in various natural alkaloids and bioactive molecules (Dhote, Patel *et al.*, 2021; Ji *et al.*, 2019; Gu *et al.*, 2014). Thus, substantial research efforts have been dedicated to the synthesis of these essential compounds (Dhote, Patel *et al.*, 2021; Wang *et al.*, 2021; Liu & McWhorter, 2003; Wetzel & Gagosz, 2011; Liu *et al.*, 2003). These synthetic methods can be broadly sorted into three main strategies, *viz.* oxidative dearomatization of indoles (Wang *et al.*, 2021; Liu *et al.*, 2020), cyclization reactions (Dhote, Pund & Ramana, 2021; Xie *et al.*, 2021; Fu & Song, 2018) and nucleophilic additions to 3*H*-indol-3-ones or indolone-*N*-oxides (Zhang *et al.*, 2017; Liu *et al.*, 2003; Berti *et al.*, 1975). Notably, indolone-*N*-oxides, also known as isatogens, hold substantial importance in medicinal chemistry and serve as intermediates in the synthesis of natural alkaloids and bioactive compounds (Nepveu *et al.*, 2010). The literature contains a wide array of techniques for synthesizing isatogens, encompassing both metal-free and metal-catalyzed routes (Dhote & Ramana, 2021; Dhote, Halnor *et al.*, 2021). These methods have been rigorously explored and well documented, underscoring the adaptability and importance of isatogens in the realms of organic synthesis and medicinal chemistry. Over the past few years, our research group has been deeply involved in this field, particularly focusing on their synthesis through nitroalkyne cycloisomerization (Dhote, Pund & Ramana, 2021; Dhote & Ramana, 2019; Kumar & Ramana, 2014, 2015; Patel *et al.*, 2010) and we have demonstrated their utility in total synthesis endeavors (Patel *et al.*, 2014; Reddy & Ramana, 2013; Kumar *et al.*, 2012; Patel & Ramana, 2012).

As part of our efforts to demonstrate the reactivity of the nitro group compared to the azide group (Dhote & Ramana, 2022; Dhote, Halner *et al.*, 2021), we designed a substrate that incorporates both a nitro group and an azide group positioned *ortho* to an alkyne functionality. Interestingly, when we subjected this substrate to treatment with either Au^{III} or Au^I,

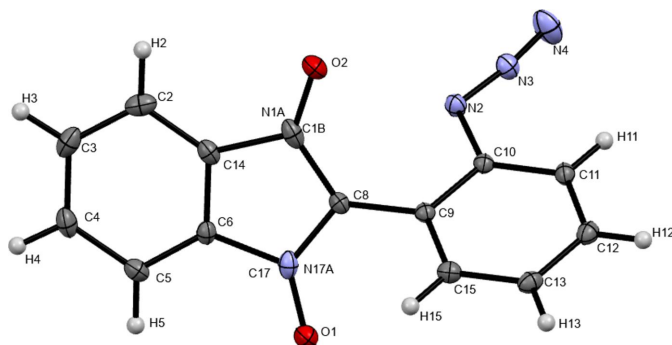
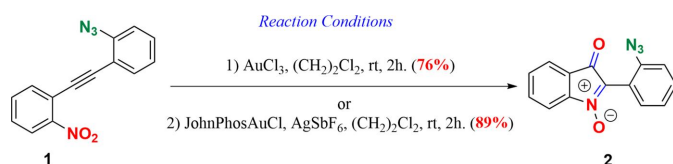


Figure 1
ORTEP diagram of **2** with 50% probability displacement ellipsoids. Only one molecule is present in the asymmetric unit.

we obtained isotogen **2** with the azide moiety intact in relatively good yield (see reaction scheme below). The structural characterization of 2-(2-azidophenyl)-3-oxo-3H-indole 1-oxide, **2**, was achieved through spectral and analytical data analysis. In the ^1H NMR spectra of **2**, the protons were observed to be overlapping, posing challenges in confirming the precise structure. Subsequently, the ^{13}C NMR spectrum indicated the absence of the alkyne carbon signal, suggesting a modification at the alkyne functionality (see Figs. S1 and S2 in the supporting information). Additionally, signals corresponding to the carbonyl and the newly formed quaternary center were observed at $\delta = 185.2$ and 140.2 ppm, respectively. The molecular composition of compound **2** was further verified as $\text{C}_{14}\text{H}_9\text{N}_4\text{O}_2$ through high-resolution mass spectrometry ($[\text{M} + \text{H}]^+$ found as 265.0771). Moreover, the structure of **2** was conclusively confirmed through single-crystal X-ray diffraction analysis.



2. Structural commentary and supramolecular features

The title compound crystallizes in space group $P-1$ with one molecule in the asymmetric unit (Fig. 1). Molecules are further

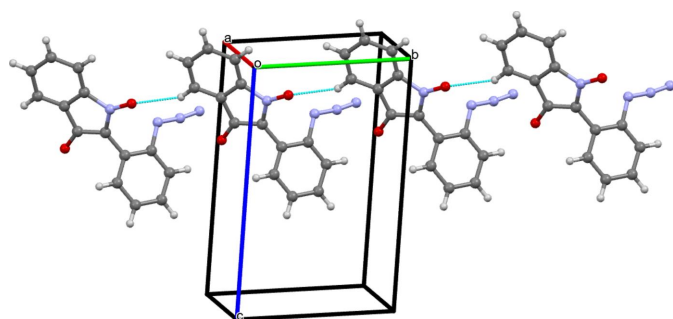


Figure 2
The packing of molecules *via* C–H...O hydrogen bonds along the *b*-axis direction. The blue lines depict the intermolecular interactions.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C5-H5}\cdots\text{O2}^i$	0.95	2.43	3.1077 (16)	128
$\text{C11-H11}\cdots\text{O1}^{ii}$	0.95	2.57	3.2327 (17)	127

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

Table 2
Covalent $\text{C=O/N}^+-\text{O}^-$ bond lengths (\AA) in **2** and related structures.

Compound	$\text{C=O/N}^+-\text{O}^-$	$\text{N}^+-\text{O}^-/\text{C=O}$
SAWYAR	1.240 (4)	1.241 (4)
SAZQIU	1.253 (2)	1.243 (4)
2	1.252 (1)	1.248 (1)

connected *via* C–H...O (Table 1) weak hydrogen bonds (Desiraju *et al.*, 1999) along the *b*-axis direction (Fig. 2). Moreover, π - π stacking [3.354 (2) \AA ; Chipot *et al.*, 1996; Chen *et al.*, 2018) is observed along the *a*-axis direction (Fig. 3). However, there are no classical hydrogen bonds present in the crystal packing.

In general, C=O bond lengths (1.22 \AA) are always shorter than N^+-O^- (1.26 \AA). However, in the current scenario, the C=O and N^+-O^- bond lengths are almost equal (Table 2). We therefore analyzed the bond lengths in similar structures found in the Cambridge Structural Database (Conquest

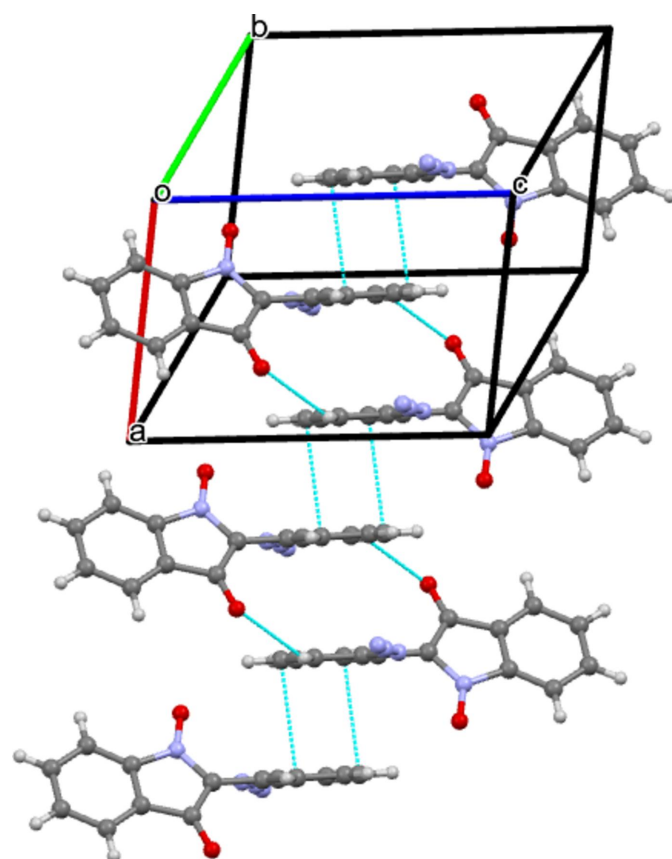
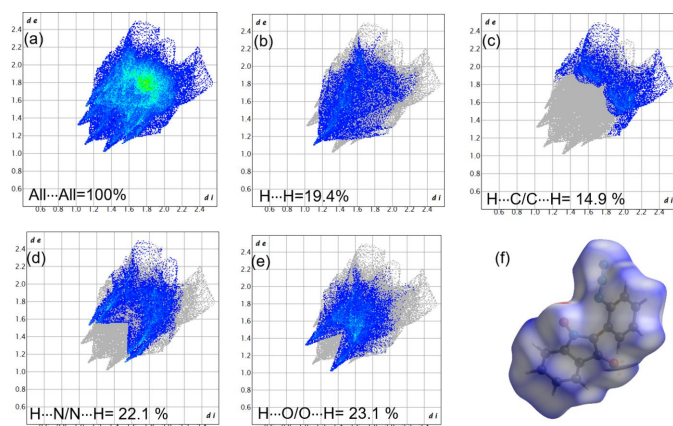


Figure 3
The single-crystal structure of **2**. π - π stacking [centroid-centroid distance = 3.354 (2) \AA] can be seen along the *a*-axis direction.


Figure 4

The two-dimensional fingerprint plots for **2**, showing interactions: (a) all interactions and delineated into (b) H \cdots H contacts; (c) H \cdots C/C \cdots H contacts; (d) H \cdots N/N \cdots H contacts and (e) H \cdots O/O \cdots H contacts; (f) Hirshfeld surface mapped with d_{norm} .

2023.3.0; CSD version 5.45, update of November 2023; Groom *et al.*, 2016), among which seven show N/C disorder and two have similar bond lengths to those in the title compound. We analyzed the bond lengths of similar molecules in the CSD database and further modeled the disorder in the current molecule. After modeling the disorder, the R factor reduced to 4.22%. Predominantly, if the atoms of molecules are disordered, the bond distance are averaged out and shows the mean distance of bonds. The N (N1A and N17A) and C (C1B and C17) atoms of isotogen are disordered over two positions with equal (0.5) site occupancy. As the molecule exhibits disorder, the bond distances were averaged out, giving N17A—O1/C17=O1 = 1.252 (1) Å and C1B=O2/N1A—O2 = 1.248 (1) Å.

3. Hirshfeld surface analysis

A Hirshfeld surface analysis of compound **2** was undertaken with *CrystalExplorer 21.5* (Spackman *et al.*, 2021) to investigate the intermolecular interactions. The overall 2D fingerprint plot is shown in Fig. 4a and those delineated into H \cdots H (19.4%), H \cdots C/C \cdots H (14.9%), H \cdots N/N \cdots H (22.1%), and H \cdots O/O \cdots H (23.1%) interactions are shown in Fig. 4b–e. Interactions such as N \cdots O/O \cdots N O \cdots C/C \cdots O and N \cdots C/C \cdots N contribute very little to the overall surface and hence those contacts are not shown. The Hirshfeld surface mapped with d_{norm} is shown in Fig. 4f (d_{norm} is the normalized sum of d_e and d_i where d_e is the distance from the Hirshfeld surface to the nearest atom i internal to the surface and d_i is distance from Hirshfeld surface to the nearest atom e external to the surface).

4. Database survey

There are twenty structures of isotogen present in the Cambridge Structural Database (CSD; Conquest 2023.3.0; CSD version 5.45, update of November 2023; Groom *et al.*,

Table 3

Experimental details.

Crystal data	
Chemical formula	C ₁₄ H ₈ N ₄ O ₂
M_r	264.24
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
a, b, c (Å)	7.166 (2), 7.686 (3), 12.172 (4)
α, β, γ (°)	95.473 (16), 105.226 (12), 113.116 (13)
V (Å ³)	579.9 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.11
Crystal size (mm)	0.12 × 0.06 × 0.05
Data collection	
Diffractometer	Bruker D8 VENTURE Kappa Duo PHOTON II CPAD
Absorption correction	Multi-scan (SADABS; Krause <i>et al.</i> , 2015)
$T_{\text{min}}, T_{\text{max}}$	0.626, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	45548, 4818, 3946
R_{int}	0.049
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.811
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.042, 0.122, 1.05
No. of reflections	4818
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.46, -0.29

Computer programs: *APEX2* and *SAINT* (Bruker, 2015), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

2016), among which ten show N/C disorder. Bond lengths associated with these atoms are unusual. In most crystal structures, C=O is always less than N⁺—O⁻. In contrast, the C=O and N⁺—O⁻ bond lengths in SAWYAR (Clegg, 2005) and SAZQIU (Clegg & Elsegood, 2005) are almost equal and are similar to those the title compound (Table 2).

5. Synthesis and crystallization

The reaction was carried out at room temperature and under an argon atmosphere. To a solution of the active gold complex prepared from JohnPhosAuCl (5 mol%) and AgSbF₆ (10 mol%) or AuCl₃ (5 mol%) in 1,2-DCE (1 ml) was added a solution of 1-azido-2-[(2 nitrophenyl)ethynyl]benzene, **1**, in 1,2-DCE (0.5 ml) dropwise over 5 minutes. The resulting solution was stirred for a period of 2 h and then concentrated under reduced pressure. The resulting crude product was purified by column chromatography to afford 2-(2-azidophenyl)-3-oxo-3H-indole 1-oxide, **2**, as a yellow solid. Next, single crystals were grown by slow evaporation of a solution of compounds (10 mg) in acetonitrile (1 ml) [placed in a long glass vial of 2 ml volume and closed with a cotton plug] at room temperature in a dark place.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were positioned geome-

trically ($C-H = 0.95 \text{ \AA}$) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Acknowledgements

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References

- Berti, C., Colonna, M., Greci, L. & Marchetti, L. (1975). *Tetrahedron*, **31**, 1745–1753.
- Bruker (2015). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chen, T., Li, M. & Liu, J. (2018). *Cryst. Growth Des.* **18**, 2765–2783.
- Chipot, C., Jaffe, R., Maigret, B., Pearlman, D. A. & Kollman, P. A. (1996). *J. Am. Chem. Soc.* **118**, 11217–11224.
- Clegg, W. (2005). *CSD Communication* (refcode SAWYAR). CCDC, Cambridge, England.
- Clegg, W. & Elsegood, M. R. J. (2005). *CSD Communication* (refcode SAZQIU). CCDC, Cambridge, England.
- Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*. New York: Oxford University Press Inc.
- Dhote, P. S., Hahn, S. V. & Ramana, C. V. (2021). *Chem. Rec.* **21**, 3546–3558.
- Dhote, P. S., Patel, P., Vanka, K. & Ramana, C. V. (2021). *Org. Biomol. Chem.* **19**, 7970–7994.
- Dhote, P. S., Pund, K. A. & Ramana, C. V. (2021). *J. Org. Chem.* **86**, 10874–10882.
- Dhote, P. S. & Ramana, C. V. (2019). *Org. Lett.* **21**, 6221–6224.
- Dhote, P. S. & Ramana, C. V. (2021). *Org. Lett.* **23**, 2632–2637.
- Dhote, P. S. & Ramana, C. V. (2022). *Adv. Synth. Catal.* **364**, 1122–1133.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Fu, W. & Song, Q. (2018). *Org. Lett.* **20**, 393–396.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Gu, W., Zhang, Y., Hao, X.-J., Yang, F.-M., Sun, Q.-Y., Morris-Natschke, S. L., Lee, K.-H., Wang, Y.-H. & Long, C.-L. (2014). *J. Nat. Prod.* **77**, 2590–2594.
- Ji, Y., He, X., Peng, C. & Huang, W. (2019). *Org. Biomol. Chem.* **17**, 2850–2864.
- Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
- Kumar, C. V. S., Puranik, V. G. & Ramana, C. V. (2012). *Chem. A Eur. J.* **18**, 9601–9611.
- Kumar, C. V. S. & Ramana, C. V. (2014). *Org. Lett.* **16**, 4766–4769.
- Kumar, C. V. S. & Ramana, C. V. (2015). *Org. Lett.* **17**, 2870–2873.
- Liu, S., Zhao, F., Chen, X., Deng, G. & Huang, H. (2020). *Adv. Synth. Catal.* **362**, 3795–3823.
- Liu, Y. & McWhorter, W. W. (2003). *J. Org. Chem.* **68**, 2618–2622.
- Nepveu, F., Kim, S., Boyer, J., Chatriant, O., Ibrahim, H., Reybier, K., Monje, M.-C., Chevalley, S., Perio, P., Lajoie, B., Bouajila, J., Deharo, E., Sauvain, M., Tahar, R., Basco, L., Pantaleo, A., Turini, F., Arese, P., Valentin, A., Thompson, E., Vivas, L., Petit, S. & Nallet, J.-P. (2010). *J. Med. Chem.* **53**, 699–714.
- Patel, P., Reddy, B. N. & Ramana, C. V. (2014). *Tetrahedron*, **70**, 510–516.
- Patel, P. & Ramana, C. V. (2012). *J. Org. Chem.* **77**, 10509–10515.
- Ramana, C. V., Patel, P., Vanka, K., Miao, B. & Degterev, A. (2010). *Eur. J. Org. Chem.* pp. 5955–5966.
- Reddy, B. N. & Ramana, C. V. (2013). *Chem. Commun.* **49**, 9767–9769.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
- Wang, Z., Xu, S., Wang, K., Kong, N. & Liu, X. (2021). *Asia. J. Org. Chem.* **10**, 1580–1594.
- Wetzel, A. & Gagosz, F. (2011). *Angew. Chem. Int. Ed.* **50**, 7354–7358.
- Xie, L. H., Li, Y., Dong, S. X., Feng, X. M. & Liu, X. H. (2021). *Chem. Commun.* **57**, 239–242.
- Zhang, X., Li, P., Lyu, C., Yong, W., Li, J., Pan, X., Zhu, X. & Rao, W. (2017). *Adv. Synth. Catal.* **359**, 4147–4152.

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Computing details

2-(2-Azidophenyl)-3-oxo-3*H*-indole 1-oxide

Crystal data

C₁₄H₈N₄O₂

M_r = 264.24

Triclinic, *P* $\bar{1}$

a = 7.166 (2) Å

b = 7.686 (3) Å

c = 12.172 (4) Å

α = 95.473 (16)°

β = 105.226 (12)°

γ = 113.116 (13)°

V = 579.9 (3) Å³

Z = 2

F(000) = 272

D_x = 1.513 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 9975 reflections

θ = 3.1–34.6°

μ = 0.11 mm⁻¹

T = 100 K

Block, colourless

0.12 × 0.06 × 0.05 mm

Data collection

Bruker D8 VENTURE Kappa Duo PHOTON II

CPAD

diffractometer

φ and ω scans

Absorption correction: multi-scan

(SADABS; Krause *et al.*, 2015)

T_{min} = 0.626, *T_{max}* = 0.745

45548 measured reflections

4818 independent reflections

3946 reflections with *I* > 2 σ (*I*)

R_{int} = 0.049

θ_{\max} = 35.2°, θ_{\min} = 3.0°

h = -11 → 11

k = -12 → 12

l = -19 → 19

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2 σ (*F*²)] = 0.042

wR(*F*²) = 0.122

S = 1.05

4818 reflections

181 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[$\sigma^2(F_o^2) + (0.066P)^2 + 0.1514P$]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

$\Delta\rho_{\max}$ = 0.46 e Å⁻³

$\Delta\rho_{\min}$ = -0.29 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>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.67828 (11)	−0.03978 (9)	0.35537 (6)	0.01840 (14)	
O2	0.33615 (11)	0.29764 (9)	0.15423 (6)	0.01970 (14)	
N4	1.05131 (16)	0.87935 (13)	0.27516 (9)	0.02787 (19)	
N2	0.81071 (12)	0.56952 (10)	0.28504 (6)	0.01489 (14)	
N3	0.93852 (12)	0.73575 (11)	0.28689 (7)	0.01669 (14)	
C10	0.77636 (12)	0.53396 (11)	0.39246 (7)	0.01172 (14)	
C9	0.64747 (12)	0.34085 (11)	0.39218 (7)	0.01194 (14)	
C8	0.54989 (12)	0.18621 (11)	0.28650 (7)	0.01240 (14)	
N1A	0.40687 (13)	0.17681 (11)	0.17988 (8)	0.01765 (16)	0.5
C14	0.34033 (13)	−0.00926 (12)	0.09961 (7)	0.01469 (15)	
C2	0.20365 (14)	−0.08622 (14)	−0.01379 (8)	0.01915 (17)	
H2	0.134768	−0.017372	−0.055149	0.023*	
C3	0.17122 (15)	−0.27158 (15)	−0.06522 (8)	0.02109 (18)	
H3	0.077902	−0.330058	−0.143374	0.025*	
C11	0.86772 (13)	0.67870 (12)	0.49428 (7)	0.01393 (15)	
H11	0.955317	0.808707	0.493893	0.017*	
C12	0.83019 (14)	0.63213 (13)	0.59624 (7)	0.01570 (15)	
H12	0.891796	0.730900	0.665580	0.019*	
C13	0.70271 (14)	0.44142 (13)	0.59774 (7)	0.01594 (15)	
H13	0.677285	0.410403	0.667753	0.019*	
C15	0.61299 (13)	0.29688 (12)	0.49620 (7)	0.01451 (15)	
H15	0.527337	0.166823	0.497471	0.017*	
C17	0.57040 (12)	0.01171 (11)	0.27811 (7)	0.01585 (15)	0.5
C6	0.44015 (13)	−0.11001 (12)	0.16023 (7)	0.01356 (14)	
C5	0.41071 (14)	−0.29159 (12)	0.11154 (8)	0.01623 (15)	
H5	0.479954	−0.359268	0.153933	0.019*	
C4	0.27263 (15)	−0.37139 (13)	−0.00430 (8)	0.01966 (17)	
H4	0.247984	−0.496292	−0.041850	0.024*	
C1B	0.40687 (13)	0.17681 (11)	0.17988 (8)	0.01765 (16)	0.5
N17A	0.57040 (12)	0.01171 (11)	0.27811 (7)	0.01585 (15)	0.5

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0195 (3)	0.0149 (3)	0.0197 (3)	0.0079 (2)	0.0042 (2)	0.0036 (2)
O2	0.0234 (3)	0.0138 (3)	0.0220 (3)	0.0095 (2)	0.0055 (2)	0.0039 (2)
N4	0.0328 (5)	0.0178 (4)	0.0343 (5)	0.0059 (3)	0.0205 (4)	0.0074 (3)
N2	0.0166 (3)	0.0121 (3)	0.0142 (3)	0.0040 (2)	0.0059 (2)	0.0031 (2)
N3	0.0184 (3)	0.0153 (3)	0.0178 (3)	0.0072 (3)	0.0084 (3)	0.0037 (2)

C10	0.0113 (3)	0.0120 (3)	0.0122 (3)	0.0057 (2)	0.0036 (2)	0.0023 (2)
C9	0.0112 (3)	0.0116 (3)	0.0128 (3)	0.0053 (3)	0.0033 (2)	0.0023 (2)
C8	0.0124 (3)	0.0099 (3)	0.0145 (3)	0.0043 (2)	0.0048 (3)	0.0024 (2)
N1A	0.0196 (4)	0.0103 (3)	0.0231 (4)	0.0038 (3)	0.0121 (3)	0.0015 (3)
C14	0.0162 (3)	0.0110 (3)	0.0183 (4)	0.0053 (3)	0.0088 (3)	0.0031 (3)
C2	0.0170 (4)	0.0261 (4)	0.0194 (4)	0.0118 (3)	0.0078 (3)	0.0116 (3)
C3	0.0172 (4)	0.0269 (4)	0.0129 (3)	0.0052 (3)	0.0033 (3)	0.0012 (3)
C11	0.0134 (3)	0.0120 (3)	0.0145 (3)	0.0053 (3)	0.0028 (3)	0.0004 (3)
C12	0.0165 (3)	0.0178 (4)	0.0130 (3)	0.0096 (3)	0.0028 (3)	0.0008 (3)
C13	0.0175 (4)	0.0202 (4)	0.0130 (3)	0.0104 (3)	0.0056 (3)	0.0044 (3)
C15	0.0148 (3)	0.0152 (3)	0.0153 (3)	0.0074 (3)	0.0057 (3)	0.0050 (3)
C17	0.0135 (3)	0.0138 (3)	0.0155 (3)	0.0015 (3)	0.0058 (3)	-0.0005 (2)
C6	0.0134 (3)	0.0117 (3)	0.0128 (3)	0.0029 (3)	0.0046 (3)	0.0010 (2)
C5	0.0187 (4)	0.0132 (3)	0.0191 (4)	0.0078 (3)	0.0077 (3)	0.0056 (3)
C4	0.0223 (4)	0.0133 (3)	0.0198 (4)	0.0041 (3)	0.0087 (3)	-0.0014 (3)
C1B	0.0196 (4)	0.0103 (3)	0.0231 (4)	0.0038 (3)	0.0121 (3)	0.0015 (3)
N17A	0.0135 (3)	0.0138 (3)	0.0155 (3)	0.0015 (3)	0.0058 (3)	-0.0005 (2)

Geometric parameters (Å, °)

O1—C17	1.2523 (11)	C14—C1B	1.4767 (12)
O1—N17A	1.2523 (11)	C2—H2	0.9500
O2—N1A	1.2485 (11)	C2—C3	1.4033 (15)
O2—C1B	1.2485 (11)	C3—H3	0.9500
N4—N3	1.1323 (12)	C3—C4	1.3892 (14)
N2—N3	1.2396 (11)	C11—H11	0.9500
N2—C10	1.4245 (11)	C11—C12	1.3876 (12)
C10—C9	1.4061 (12)	C12—H12	0.9500
C10—C11	1.3933 (12)	C12—C13	1.3950 (13)
C9—C8	1.4628 (12)	C13—H13	0.9500
C9—C15	1.3996 (12)	C13—C15	1.3911 (12)
C8—N1A	1.4019 (13)	C15—H15	0.9500
C8—C17	1.4028 (12)	C17—C6	1.4697 (12)
C8—C1B	1.4019 (13)	C6—C5	1.3744 (12)
C8—N17A	1.4028 (12)	C6—N17A	1.4697 (12)
N1A—C14	1.4767 (12)	C5—H5	0.9500
C14—C2	1.3762 (13)	C5—C4	1.4033 (13)
C14—C6	1.3865 (12)	C4—H4	0.9500
N3—N2—C10	116.77 (7)	C12—C11—H11	120.1
N4—N3—N2	171.89 (9)	C11—C12—H12	119.7
C9—C10—N2	116.31 (7)	C11—C12—C13	120.52 (8)
C11—C10—N2	123.03 (8)	C13—C12—H12	119.7
C11—C10—C9	120.64 (8)	C12—C13—H13	120.2
C10—C9—C8	121.71 (7)	C15—C13—C12	119.67 (8)
C15—C9—C10	118.70 (7)	C15—C13—H13	120.2
C15—C9—C8	119.59 (7)	C9—C15—H15	119.6
N1A—C8—C9	126.97 (7)	C13—C15—C9	120.76 (8)

N1A—C8—C17	107.79 (7)	C13—C15—H15	119.6
C17—C8—C9	125.04 (7)	O1—C17—C8	127.91 (8)
C1B—C8—C9	126.97 (7)	O1—C17—C6	123.29 (8)
C1B—C8—N17A	107.79 (7)	C8—C17—C6	108.80 (7)
N17A—C8—C9	125.04 (7)	C14—C6—C17	107.50 (7)
O2—N1A—C8	127.81 (8)	C14—C6—N17A	107.50 (7)
O2—N1A—C14	123.50 (8)	C5—C6—C14	122.61 (8)
C8—N1A—C14	108.64 (7)	C5—C6—C17	129.89 (8)
C2—C14—N1A	131.01 (8)	C5—C6—N17A	129.89 (8)
C2—C14—C6	121.74 (8)	C6—C5—H5	121.9
C2—C14—C1B	131.01 (8)	C6—C5—C4	116.25 (8)
C6—C14—N1A	107.23 (8)	C4—C5—H5	121.9
C6—C14—C1B	107.23 (8)	C3—C4—C5	121.38 (8)
C14—C2—H2	121.7	C3—C4—H4	119.3
C14—C2—C3	116.59 (8)	C5—C4—H4	119.3
C3—C2—H2	121.7	O2—C1B—C8	127.81 (8)
C2—C3—H3	119.3	O2—C1B—C14	123.50 (8)
C4—C3—C2	121.42 (8)	C8—C1B—C14	108.64 (7)
C4—C3—H3	119.3	O1—N17A—C8	127.91 (8)
C10—C11—H11	120.1	O1—N17A—C6	123.29 (8)
C12—C11—C10	119.71 (8)	C8—N17A—C6	108.80 (7)
O1—C17—C6—C14	178.78 (8)	C14—C6—C5—C4	0.01 (12)
O1—C17—C6—C5	-0.58 (14)	C14—C6—N17A—O1	178.78 (8)
O2—N1A—C14—C2	2.51 (14)	C14—C6—N17A—C8	-0.88 (9)
O2—N1A—C14—C6	-176.07 (8)	C2—C14—C6—C17	-179.07 (7)
N2—C10—C9—C8	-1.48 (11)	C2—C14—C6—C5	0.35 (13)
N2—C10—C9—C15	178.39 (7)	C2—C14—C6—N17A	-179.07 (7)
N2—C10—C11—C12	-178.73 (7)	C2—C14—C1B—O2	2.51 (14)
N3—N2—C10—C9	-174.08 (7)	C2—C14—C1B—C8	-179.99 (9)
N3—N2—C10—C11	4.34 (12)	C2—C3—C4—C5	0.43 (14)
C10—C9—C8—N1A	-59.12 (12)	C11—C10—C9—C8	-179.94 (7)
C10—C9—C8—C17	126.65 (9)	C11—C10—C9—C15	-0.08 (11)
C10—C9—C8—C1B	-59.12 (12)	C11—C12—C13—C15	0.14 (13)
C10—C9—C8—N17A	126.65 (9)	C12—C13—C15—C9	-0.61 (12)
C10—C9—C15—C13	0.57 (12)	C15—C9—C8—N1A	121.02 (9)
C10—C11—C12—C13	0.35 (12)	C15—C9—C8—C17	-53.22 (11)
C9—C10—C11—C12	-0.38 (12)	C15—C9—C8—C1B	121.02 (9)
C9—C8—N1A—O2	0.35 (14)	C15—C9—C8—N17A	-53.22 (11)
C9—C8—N1A—C14	-177.02 (7)	C17—C8—N1A—O2	175.39 (8)
C9—C8—C17—O1	-2.70 (13)	C17—C8—N1A—C14	-1.97 (9)
C9—C8—C17—C6	176.94 (7)	C17—C6—C5—C4	179.29 (8)
C9—C8—C1B—O2	0.35 (14)	C6—C14—C2—C3	-0.31 (13)
C9—C8—C1B—C14	-177.02 (7)	C6—C14—C1B—O2	-176.07 (8)
C9—C8—N17A—O1	-2.70 (13)	C6—C14—C1B—C8	1.43 (9)
C9—C8—N17A—C6	176.94 (7)	C6—C5—C4—C3	-0.39 (13)
C8—C9—C15—C13	-179.56 (7)	C5—C6—N17A—O1	-0.58 (14)
C8—N1A—C14—C2	-179.99 (9)	C5—C6—N17A—C8	179.76 (8)

C8—N1A—C14—C6	1.43 (9)	C1B—C8—N17A—O1	-177.87 (8)
C8—C17—C6—C14	-0.88 (9)	C1B—C8—N17A—C6	1.78 (9)
C8—C17—C6—C5	179.76 (8)	C1B—C14—C2—C3	-178.72 (8)
N1A—C8—C17—O1	-177.87 (8)	C1B—C14—C6—C5	179.09 (7)
N1A—C8—C17—C6	1.78 (9)	C1B—C14—C6—N17A	-0.32 (9)
N1A—C14—C2—C3	-178.72 (8)	N17A—C8—C1B—O2	175.39 (8)
N1A—C14—C6—C17	-0.32 (9)	N17A—C8—C1B—C14	-1.97 (9)
N1A—C14—C6—C5	179.09 (7)	N17A—C6—C5—C4	179.29 (8)
C14—C2—C3—C4	-0.07 (13)		

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 ⁱ	0.95	2.43	3.1077 (16)	128
C11—H11 \cdots O1 ⁱⁱ	0.95	2.57	3.2327 (17)	127

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

Table 2. Weak hydrogen bond table of compound 2

No classical hydrogen bonds found. For C—H \cdots acceptor interactions, see: Steiner, Th., (1996), *Cryst. Rev.*, 6, 1-57 H-Bond classification [Jeffrey, G. A., Maluszynska, H. & Mitra, J., (1985), *Int J. Biol. Macromol.* 7, 336-348]

S.No	D-H \cdots A	H \cdots A (Å)	D \cdots A (Å)	\langle D-H \cdots A \rangle (°)
1	C5-H5 \cdots O2 ^(a)	2.43	3.1077 (16)	128
2	C11-H11 \cdots O1 ^(b)	2.57	3.2327 (17)	127

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