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# 2-(5-Methoxy-2-methyl-1*H*-indol-3-yl)-*N*′-[(1*E*,2*E*)-3-phenylprop-2-en-1-ylidene]acetohydrazide

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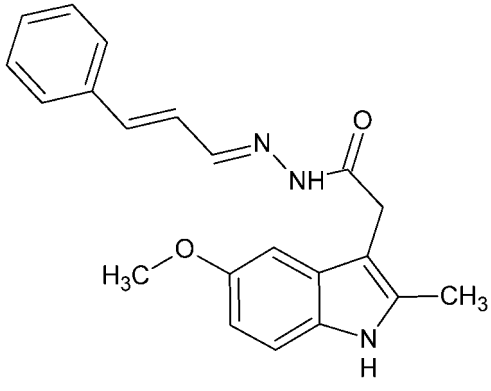
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.045; $wR$ factor = 0.126; data-to-parameter ratio = 18.7.

The title compound, $C_{21}H_{21}N_3O_2$, adopts a J-shaped conformation which appears to be at least partially directed by a weak intramolecular C—H···N hydrogen bond. In the crystal, molecules are linked by N—H···O hydrogen bonds into $R_2^2(8)$ and $R_2^2(14)$ cyclic dimers, which form a chain running parallel to the $b$ axis.

## Related literature

For general background to side-effect toxicity of non-steroidal anti-inflammatory drugs (NSAIDs), see: Agrawal *et al.* (2010); Champion *et al.* (1997); Allan & Fletcher (1990). For reduction of GI toxicity attributed to NSAIDs, see: Halen *et al.* (2009); Schoen & Vender (1989); Mitchell & Warner (1999). For hydrogen-bond motifs, see: Etter *et al.* (1990).




## Experimental

### Crystal data

$C_{21}H_{21}N_3O_2$
$M_r$ = 347.41
Triclinic, $P\overline{1}$
$a$ = 8.2786 (9) Å
$b$ = 10.1194 (11) Å
$c$ = 11.7739 (13) Å
$\alpha$ = 93.001 (2)°
$\beta$ = 108.993 (2)°
$\gamma$ = 105.578 (2)°
$V$ = 887.76 (17) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 150 K
0.26 × 0.13 × 0.08 mm

### Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min}$ = 0.78, $T_{max}$ = 0.99
16303 measured reflections
4574 independent reflections
3675 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.126
$S$ = 1.08
4574 reflections
245 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.891 (18) | 2.093 (18) | 2.9841 (14) | 178.1 (9) |
| N2—H2···O2$^{ii}$ | 0.903 (16) | 2.012 (16) | 2.9055 (13) | 170.0 (14) |
| C7—H7···N3 | 0.95 | 2.54 | 3.3609 (15) | 145 |

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; 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) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON*.

Manchester Metropolitan University, Tulane University and Erciyes University are gratefully acknowledged for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QM2100).

## References

Agrawal, N., Chandrasekar, M. J. N., Sara, U. V. S. & Rohini, A. (2010). *Int. J. Drug Deliv. Tech.* **2**, 12–17.
Allan, H. P. & Fletcher, M. (1990). *Drugs*, **40**, 1–11.
Bruker (2013). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Champion, G. D., Feng, P. H., Azuma, T., Caughey, D. E., Chan, K. H., Kashiwazaki, S., Liu, H.-C., Nasution, A. R., Hobunaga, M., Prichanond, S., Torralba, T. P., Udom, V. & Yoo, M. C. (1997). *Drugs*, **53**, 61–69.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Halen, P. K., Murumkar, P. R., Giridhar, R. & Yadav, M. R. (2009). *Mini Rev. Med. Chem.* **9**, 124–139.
Mitchell, J. A. & Warner, T. D. (1999). *Br. J. Pharmacol.* **128**, 1121–1132.
Schoen, R. T. & Vender, R. J. (1989). *Am. J. Med.* **86**, 449–458.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

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# 2-(5-Methoxy-2-methyl-1*H*-indol-3-yl)-*N*′-[(1*E*,2*E*)-3-phenylprop-2-en-1-ylidene]acetohydrazide

**Mehmet Akkurt, Joel T. Mague, Shaaban K. Mohamed, Mustafa R. Albayati and Mahmoud A. A. El-Remaily**

## S1. Comment

Indomethacin as other common anti-inflammatory drugs (NSAIDs) which are widely employed in the treatment of pain and inflammation has been reported to be associated with a number of undesirable effects, in particular gastrointestinal (GI) toxicity and ulceration (Agrawal *et al.*, 2010; Champion *et al.*, 1997; Allan & Fletcher, 1990) which represent a still unsolved therapeutic problem. Topical irritation by the free carboxylic group of Indomethacin is considered an important factor in establishing superficial stomach erosion (Schoen & Vender, 1989; Mitchell & Warner, 1999). Considerable attention has been focused on the development of bio-reversible derivatives of such pro-drugs to temporarily mask the acidic group as a promising means of reducing or abolishing the GI toxicity due to the local action mechanism (Halen *et al.*, 2009). Based on such facts and continue to our on-going study in functionalization of NSAIDs we herein report the synthesis and crystal structure of the title compound.

The molecular conformation adopted by I in the crystal is "J" shaped (Fig. 1) and appears to be at least partially directed by a weak, intramolecular C7—H7···N3 hydrogen bond. The indole ring system is almostly planar [maximum deviations = -0.046 (1) Å for N1, -0.036 (1) Å for C2 and 0.035 (1) Å for C4] and the dihedral angle between it and the terminal phenyl ring is 79.10 (5)°.

In the crystal structure, the N—H···O hydrogen bonding consists of $R^2_2(8)$ rings (Etter *et al.*, 1990) with 2 N2—H2···O2 contacts and $R^2_2(14)$ rings with 2 N1—H1···O2 contacts which form a chain running parallel to the *b* axis (Table 1, Figs. 2 & 3).

## S2. Experimental

A mixture of 233 mg (1 mmol) 2-(5-methoxy-2-methyl-1*H*-indole-3-yl)acetohydrazide and 132 mg (1 mmol) of (2E)-3-phenylprop-2-enal in 50 ml of ethanol containing a few drops of glacial acetic acid was refluxed for 6 hrs. The mixture was cooled to room temperature and the excess solvent was evaporated under *vacuum*. The resulting solid was collected, washed with ethanol and recrystalized from dioxan to give colourless tablets (*M*.p. 410–413 K) suitable for X-ray analysis.

IR (KBr $cm^{-1}$): (C=O amide 1661), (NH 3301), (C=N 1606) (C—H, Ar 3021–3072), (C—H aliphatic 2836–2957). $^1$H-NMR: (DMSO-$d_6$) $\delta$ at 3.6(s, 3H, –$OCH_3$), 2.3(s, 3H, $CH_3$), 3.4(s, 2H, –$CH_2$), 6.5(d,1*H*, –CH= alkene), 6.6(d,1*H*, –CH= alkene), 8.2(d, 1H, –CH=N), 11.8(s, 1H, –NH amide), the aromatic protons of indole nuclei and benzene ring were appeared in the range of 7.0–7.9. $^{13}$C-NMR: 161(C=O amide), 149(–CH=N), 55(–$OCH_3$), 10(1 C, $CH_3$), 136(–C=C alkene), 125(–C=C alkene).

### S3. Refinement

C-bound H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H = 0.95–0.99 Å, with $U_{iso}$(H) = 1.5 $U_{iso}$(C) for methyl H atoms and $U_{iso}$(H) = 1.2 $U_{iso}$(C) for other H atoms. H atoms bonded to N atoms were located in difference Fourier maps [N1—H1 = 0.891 (18) Å and N2—H2 = 0.903 (16) Å] and refined isotropically.

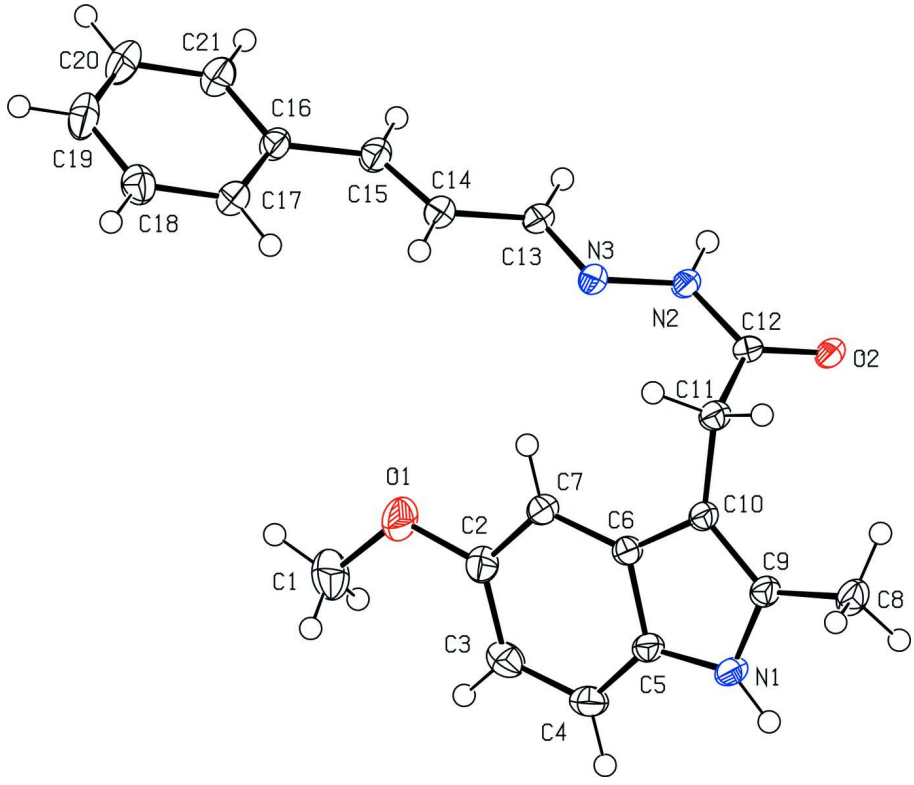


**Figure 1**

Perspective view of the title molecule with 50% probability displacement ellipsoids.

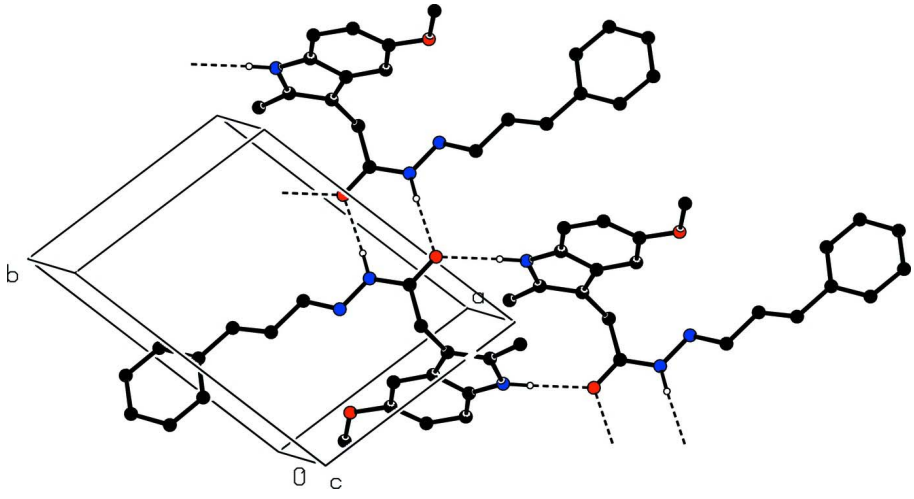

**Figure 2**

Partial view of the $R_2^2(8)$ and $R_2^2(14)$ cyclic dimers, down the *a* axis.

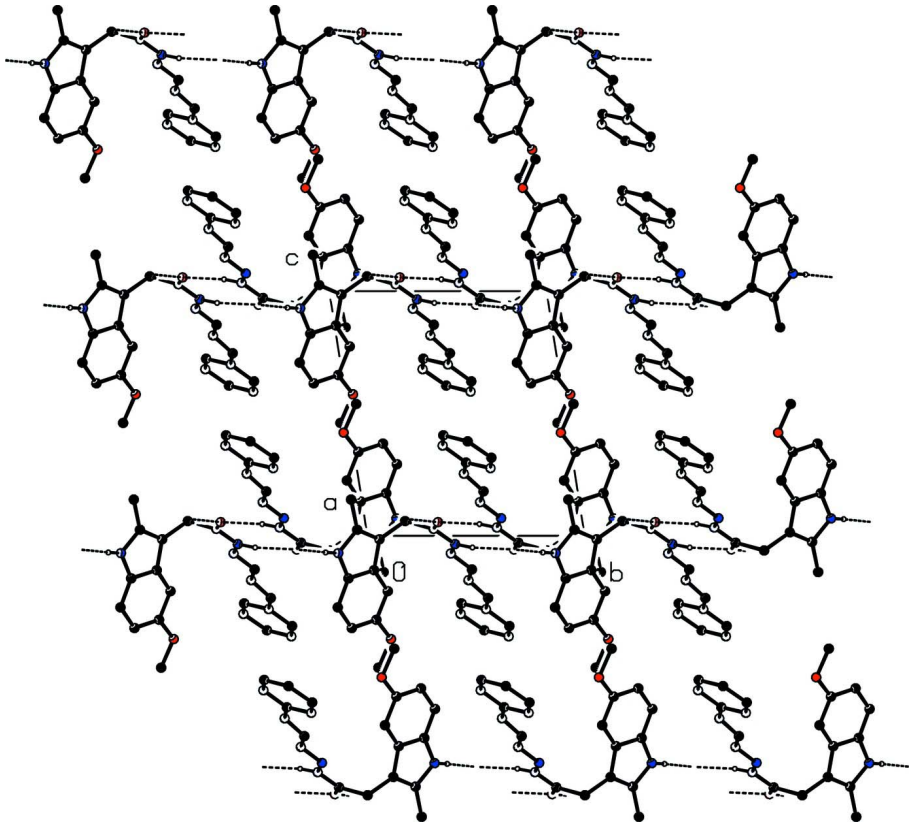


**Figure 3**

Packing of the title molecule viewed down *a* with the hydrogen bonds shown by dotted lines.

**2-(5-Methoxy-2-methyl-1*H*-indol-3-yl)-*N*′-[(1*E*,2*E*)-3-phenylprop-2-en-1-ylidene]acetohydrazide**

*Crystal data*

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$M_r$ = 347.41
Triclinic, $P\overline{1}$
Hall symbol: -P 1
*a* = 8.2786 (9) Å
*b* = 10.1194 (11) Å
*c* = 11.7739 (13) Å
*α* = 93.001 (2)°
*β* = 108.993 (2)°
*γ* = 105.578 (2)°
*V* = 887.76 (17) Å$^3$
*Z* = 2
*F*(000) = 368
$D_x$ = 1.300 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 8428 reflections
*θ* = 2.6–29.1°
*μ* = 0.09 mm$^{-1}$
*T* = 150 K
Tablet, clear colourless
0.26 × 0.13 × 0.08 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3660 pixels mm$^{-1}$
*φ* and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min}$ = 0.78, $T_{max}$ = 0.99
16303 measured reflections

4574 independent reflections
3675 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 29.1°$, $\theta_{min} = 1.9°$
$h = -11 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.126$
$S = 1.08$
4574 reflections
245 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$W = 1/[\Sigma^2(FO^2) + (0.0677P)^2 + 0.0983P]$ WHERE $P = (FO^2 + 2FC^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å$^{-3}$
$\Delta\rho_{min} = -0.25$ e Å$^{-3}$

*Special details*

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

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.28193 (13) | 0.03509 (10) | 0.57833 (8) | 0.0383 (3) |
| O2 | 1.01220 (10) | 0.33474 (8) | 1.05582 (8) | 0.0235 (3) |
| N1 | 0.76159 (13) | −0.14643 (9) | 0.92901 (9) | 0.0233 (3) |
| N2 | 0.76767 (12) | 0.39782 (9) | 0.96609 (9) | 0.0198 (3) |
| N3 | 0.58483 (12) | 0.37175 (9) | 0.92778 (9) | 0.0213 (3) |
| C1 | 0.1780 (2) | −0.04316 (16) | 0.46126 (12) | 0.0406 (4) |
| C2 | 0.40353 (16) | −0.01850 (12) | 0.65687 (11) | 0.0261 (3) |
| C3 | 0.44402 (17) | −0.13776 (12) | 0.62313 (11) | 0.0289 (3) |
| C4 | 0.56565 (16) | −0.18727 (11) | 0.70752 (11) | 0.0264 (3) |
| C5 | 0.64546 (14) | −0.11740 (10) | 0.82602 (11) | 0.0210 (3) |
| C6 | 0.61147 (14) | 0.00623 (10) | 0.85962 (10) | 0.0190 (3) |
| C7 | 0.48756 (14) | 0.05444 (11) | 0.77385 (10) | 0.0216 (3) |
| C8 | 0.92662 (16) | −0.04883 (12) | 1.14821 (11) | 0.0266 (3) |
| C9 | 0.80675 (14) | −0.04222 (11) | 1.02468 (11) | 0.0212 (3) |
| C10 | 0.71796 (14) | 0.05319 (10) | 0.98586 (10) | 0.0190 (3) |
| C11 | 0.73269 (15) | 0.18427 (10) | 1.06001 (10) | 0.0203 (3) |
| C12 | 0.84755 (14) | 0.31009 (10) | 1.02798 (10) | 0.0184 (3) |
| C13 | 0.52187 (15) | 0.45707 (11) | 0.86269 (10) | 0.0217 (3) |
| C14 | 0.33089 (15) | 0.43345 (11) | 0.81838 (11) | 0.0235 (3) |

| | | | | |
|---|---|---|---|---|
| C15 | 0.24928 (15) | 0.51098 (12) | 0.74611 (11) | 0.0245 (3) |
| C16 | 0.05663 (15) | 0.49356 (11) | 0.70035 (10) | 0.0223 (3) |
| C17 | −0.06817 (16) | 0.38488 (12) | 0.72362 (11) | 0.0257 (3) |
| C18 | −0.24894 (16) | 0.37336 (13) | 0.68079 (11) | 0.0289 (3) |
| C19 | −0.30851 (17) | 0.47052 (15) | 0.61408 (12) | 0.0342 (4) |
| C20 | −0.18776 (18) | 0.57806 (15) | 0.58911 (12) | 0.0350 (4) |
| C21 | −0.00671 (17) | 0.58889 (13) | 0.63135 (11) | 0.0291 (3) |
| H1 | 0.827 (2) | −0.2041 (17) | 0.9322 (15) | 0.044 (4)* |
| H1A | 0.25730 | −0.05190 | 0.41690 | 0.0610* |
| H1B | 0.09370 | 0.00410 | 0.41640 | 0.0610* |
| H1C | 0.11120 | −0.13570 | 0.46960 | 0.0610* |
| H2 | 0.838 (2) | 0.4754 (16) | 0.9519 (13) | 0.032 (4)* |
| H3 | 0.38760 | −0.18480 | 0.54180 | 0.0350* |
| H4 | 0.59380 | −0.26740 | 0.68470 | 0.0320* |
| H7 | 0.46140 | 0.13600 | 0.79550 | 0.0260* |
| H8A | 0.98240 | 0.04490 | 1.19500 | 0.0400* |
| H8B | 1.02000 | −0.08780 | 1.14140 | 0.0400* |
| H8C | 0.85640 | −0.10790 | 1.18960 | 0.0400* |
| H11A | 0.78640 | 0.17950 | 1.14750 | 0.0240* |
| H11B | 0.61160 | 0.19340 | 1.04460 | 0.0240* |
| H13 | 0.59940 | 0.53380 | 0.84430 | 0.0260* |
| H14 | 0.25880 | 0.35810 | 0.84210 | 0.0280* |
| H15 | 0.32320 | 0.58470 | 0.72200 | 0.0290* |
| H17 | −0.02820 | 0.31800 | 0.76950 | 0.0310* |
| H18 | −0.33210 | 0.29880 | 0.69720 | 0.0350* |
| H19 | −0.43240 | 0.46330 | 0.58550 | 0.0410* |
| H20 | −0.22870 | 0.64450 | 0.54300 | 0.0420* |
| H21 | 0.07530 | 0.66240 | 0.61290 | 0.0350* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0382 (5) | 0.0443 (5) | 0.0258 (5) | 0.0207 (4) | −0.0034 (4) | 0.0015 (4) |
| O2 | 0.0174 (4) | 0.0182 (4) | 0.0346 (5) | 0.0081 (3) | 0.0066 (3) | 0.0048 (3) |
| N1 | 0.0215 (5) | 0.0171 (4) | 0.0333 (5) | 0.0093 (4) | 0.0093 (4) | 0.0057 (4) |
| N2 | 0.0159 (4) | 0.0161 (4) | 0.0280 (5) | 0.0064 (3) | 0.0072 (4) | 0.0047 (4) |
| N3 | 0.0161 (4) | 0.0197 (4) | 0.0277 (5) | 0.0069 (3) | 0.0064 (4) | 0.0016 (4) |
| C1 | 0.0359 (8) | 0.0526 (8) | 0.0244 (7) | 0.0121 (6) | 0.0006 (6) | 0.0028 (6) |
| C2 | 0.0228 (6) | 0.0271 (5) | 0.0261 (6) | 0.0082 (4) | 0.0052 (5) | 0.0048 (5) |
| C3 | 0.0302 (6) | 0.0265 (6) | 0.0257 (6) | 0.0048 (5) | 0.0083 (5) | −0.0029 (5) |
| C4 | 0.0277 (6) | 0.0190 (5) | 0.0338 (7) | 0.0063 (4) | 0.0138 (5) | 0.0002 (4) |
| C5 | 0.0181 (5) | 0.0161 (5) | 0.0297 (6) | 0.0054 (4) | 0.0095 (4) | 0.0040 (4) |
| C6 | 0.0162 (5) | 0.0162 (4) | 0.0252 (6) | 0.0045 (4) | 0.0084 (4) | 0.0034 (4) |
| C7 | 0.0201 (5) | 0.0191 (5) | 0.0264 (6) | 0.0074 (4) | 0.0078 (5) | 0.0041 (4) |
| C8 | 0.0227 (6) | 0.0250 (5) | 0.0329 (6) | 0.0098 (4) | 0.0076 (5) | 0.0126 (5) |
| C9 | 0.0174 (5) | 0.0180 (5) | 0.0285 (6) | 0.0057 (4) | 0.0075 (4) | 0.0071 (4) |
| C10 | 0.0165 (5) | 0.0163 (4) | 0.0244 (5) | 0.0054 (4) | 0.0068 (4) | 0.0048 (4) |
| C11 | 0.0210 (5) | 0.0183 (5) | 0.0227 (5) | 0.0075 (4) | 0.0076 (4) | 0.0044 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12 | 0.0190 (5) | 0.0159 (4) | 0.0206 (5) | 0.0075 (4) | 0.0059 (4) | 0.0004 (4) |
| C13 | 0.0205 (5) | 0.0191 (5) | 0.0265 (6) | 0.0085 (4) | 0.0076 (5) | 0.0024 (4) |
| C14 | 0.0199 (5) | 0.0220 (5) | 0.0278 (6) | 0.0080 (4) | 0.0065 (5) | 0.0021 (4) |
| C15 | 0.0204 (5) | 0.0242 (5) | 0.0301 (6) | 0.0088 (4) | 0.0084 (5) | 0.0059 (4) |
| C16 | 0.0212 (5) | 0.0248 (5) | 0.0223 (5) | 0.0115 (4) | 0.0059 (4) | 0.0019 (4) |
| C17 | 0.0239 (6) | 0.0260 (5) | 0.0281 (6) | 0.0116 (4) | 0.0071 (5) | 0.0043 (5) |
| C18 | 0.0230 (6) | 0.0335 (6) | 0.0282 (6) | 0.0074 (5) | 0.0082 (5) | 0.0003 (5) |
| C19 | 0.0224 (6) | 0.0487 (8) | 0.0317 (7) | 0.0172 (5) | 0.0049 (5) | 0.0051 (6) |
| C20 | 0.0320 (7) | 0.0429 (7) | 0.0341 (7) | 0.0226 (6) | 0.0066 (6) | 0.0131 (6) |
| C21 | 0.0272 (6) | 0.0319 (6) | 0.0307 (6) | 0.0136 (5) | 0.0089 (5) | 0.0095 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.4195 (17) | C16—C17 | 1.3991 (17) |
| O1—C2 | 1.3793 (16) | C17—C18 | 1.384 (2) |
| O2—C12 | 1.2432 (15) | C18—C19 | 1.3842 (19) |
| N1—C5 | 1.3824 (16) | C19—C20 | 1.382 (2) |
| N1—C9 | 1.3822 (15) | C20—C21 | 1.388 (2) |
| N2—N3 | 1.3758 (15) | C1—H1A | 0.9800 |
| N2—C12 | 1.3512 (15) | C1—H1B | 0.9800 |
| N3—C13 | 1.2866 (15) | C1—H1C | 0.9800 |
| N1—H1 | 0.891 (18) | C3—H3 | 0.9500 |
| N2—H2 | 0.903 (16) | C4—H4 | 0.9500 |
| C2—C7 | 1.3856 (16) | C7—H7 | 0.9500 |
| C2—C3 | 1.4073 (18) | C8—H8A | 0.9800 |
| C3—C4 | 1.3871 (18) | C8—H8B | 0.9800 |
| C4—C5 | 1.3888 (17) | C8—H8C | 0.9800 |
| C5—C6 | 1.4179 (15) | C11—H11A | 0.9900 |
| C6—C10 | 1.4354 (16) | C11—H11B | 0.9900 |
| C6—C7 | 1.3986 (16) | C13—H13 | 0.9500 |
| C8—C9 | 1.4869 (17) | C14—H14 | 0.9500 |
| C9—C10 | 1.3748 (16) | C15—H15 | 0.9500 |
| C10—C11 | 1.5030 (15) | C17—H17 | 0.9500 |
| C11—C12 | 1.5169 (15) | C18—H18 | 0.9500 |
| C13—C14 | 1.4409 (19) | C19—H19 | 0.9500 |
| C14—C15 | 1.3328 (17) | C20—H20 | 0.9500 |
| C15—C16 | 1.4645 (19) | C21—H21 | 0.9500 |
| C16—C21 | 1.3936 (17) | | |
| | | | |
| C1—O1—C2 | 117.79 (11) | C16—C21—C20 | 121.01 (13) |
| C5—N1—C9 | 109.03 (9) | O1—C1—H1A | 109.00 |
| N3—N2—C12 | 121.25 (9) | O1—C1—H1B | 109.00 |
| N2—N3—C13 | 116.02 (10) | O1—C1—H1C | 109.00 |
| C9—N1—H1 | 121.1 (11) | H1A—C1—H1B | 109.00 |
| C5—N1—H1 | 127.1 (11) | H1A—C1—H1C | 109.00 |
| N3—N2—H2 | 120.7 (11) | H1B—C1—H1C | 109.00 |
| C12—N2—H2 | 118.1 (11) | C2—C3—H3 | 120.00 |
| O1—C2—C3 | 123.67 (11) | C4—C3—H3 | 120.00 |

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| O1—C2—C7 | 115.35 (11) | C3—C4—H4 | 121.00 |
| C3—C2—C7 | 120.98 (12) | C5—C4—H4 | 121.00 |
| C2—C3—C4 | 120.58 (11) | C2—C7—H7 | 121.00 |
| C3—C4—C5 | 118.66 (11) | C6—C7—H7 | 120.00 |
| C4—C5—C6 | 121.15 (11) | C9—C8—H8A | 109.00 |
| N1—C5—C6 | 107.51 (10) | C9—C8—H8B | 109.00 |
| N1—C5—C4 | 131.34 (10) | C9—C8—H8C | 109.00 |
| C5—C6—C10 | 106.84 (10) | H8A—C8—H8B | 109.00 |
| C7—C6—C10 | 133.62 (10) | H8A—C8—H8C | 109.00 |
| C5—C6—C7 | 119.50 (10) | H8B—C8—H8C | 109.00 |
| C2—C7—C6 | 119.01 (11) | C10—C11—H11A | 109.00 |
| N1—C9—C8 | 120.64 (10) | C10—C11—H11B | 109.00 |
| N1—C9—C10 | 109.46 (10) | C12—C11—H11A | 109.00 |
| C8—C9—C10 | 129.81 (11) | C12—C11—H11B | 109.00 |
| C6—C10—C11 | 126.11 (10) | H11A—C11—H11B | 108.00 |
| C9—C10—C11 | 126.77 (10) | N3—C13—H13 | 121.00 |
| C6—C10—C9 | 107.10 (10) | C14—C13—H13 | 121.00 |
| C10—C11—C12 | 110.76 (10) | C13—C14—H14 | 118.00 |
| N2—C12—C11 | 118.76 (11) | C15—C14—H14 | 118.00 |
| O2—C12—N2 | 118.88 (10) | C14—C15—H15 | 117.00 |
| O2—C12—C11 | 122.36 (10) | C16—C15—H15 | 117.00 |
| N3—C13—C14 | 118.40 (11) | C16—C17—H17 | 119.00 |
| C13—C14—C15 | 123.95 (11) | C18—C17—H17 | 120.00 |
| C14—C15—C16 | 126.31 (11) | C17—C18—H18 | 120.00 |
| C15—C16—C17 | 122.69 (11) | C19—C18—H18 | 120.00 |
| C15—C16—C21 | 119.29 (11) | C18—C19—H19 | 120.00 |
| C17—C16—C21 | 118.02 (12) | C20—C19—H19 | 120.00 |
| C16—C17—C18 | 121.03 (11) | C19—C20—H20 | 120.00 |
| C17—C18—C19 | 119.96 (12) | C21—C20—H20 | 120.00 |
| C18—C19—C20 | 119.99 (14) | C16—C21—H21 | 119.00 |
| C19—C20—C21 | 119.99 (13) | C20—C21—H21 | 120.00 |

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| C1—O1—C2—C3 | 7.23 (19) | C5—C6—C10—C11 | 176.91 (11) |
| C1—O1—C2—C7 | −173.47 (12) | C7—C6—C10—C9 | 176.05 (13) |
| C9—N1—C5—C4 | 178.38 (13) | C7—C6—C10—C11 | −5.5 (2) |
| C9—N1—C5—C6 | −2.32 (13) | N1—C9—C10—C6 | 0.16 (14) |
| C5—N1—C9—C8 | 178.24 (11) | N1—C9—C10—C11 | −178.30 (11) |
| C5—N1—C9—C10 | 1.36 (14) | C8—C9—C10—C6 | −176.35 (12) |
| C12—N2—N3—C13 | −175.51 (10) | C8—C9—C10—C11 | 5.2 (2) |
| N3—N2—C12—O2 | 176.74 (10) | C6—C10—C11—C12 | −74.62 (15) |
| N3—N2—C12—C11 | −2.72 (16) | C9—C10—C11—C12 | 103.55 (13) |
| N2—N3—C13—C14 | 179.00 (10) | C10—C11—C12—O2 | −70.23 (14) |
| O1—C2—C3—C4 | −178.89 (12) | C10—C11—C12—N2 | 109.21 (12) |
| C7—C2—C3—C4 | 1.8 (2) | N3—C13—C14—C15 | −177.08 (12) |
| O1—C2—C7—C6 | 179.33 (11) | C13—C14—C15—C16 | −178.72 (11) |
| C3—C2—C7—C6 | −1.35 (19) | C14—C15—C16—C17 | −3.5 (2) |
| C2—C3—C4—C5 | 0.5 (2) | C14—C15—C16—C21 | 176.09 (12) |
| C3—C4—C5—N1 | 175.97 (13) | C15—C16—C17—C18 | 178.73 (11) |

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| C3—C4—C5—C6 | −3.25 (19) | C21—C16—C17—C18 | −0.85 (18) |
| N1—C5—C6—C7 | −175.64 (11) | C15—C16—C21—C20 | −178.26 (12) |
| N1—C5—C6—C10 | 2.37 (13) | C17—C16—C21—C20 | 1.33 (18) |
| C4—C5—C6—C7 | 3.75 (18) | C16—C17—C18—C19 | −0.17 (19) |
| C4—C5—C6—C10 | −178.25 (11) | C17—C18—C19—C20 | 0.7 (2) |
| C5—C6—C7—C2 | −1.38 (17) | C18—C19—C20—C21 | −0.3 (2) |
| C10—C6—C7—C2 | −178.75 (13) | C19—C20—C21—C16 | −0.8 (2) |
| C5—C6—C10—C9 | −1.55 (13) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.891 (18) | 2.093 (18) | 2.9841 (14) | 178.1 (9) |
| N2—H2···O2$^{ii}$ | 0.903 (16) | 2.012 (16) | 2.9055 (13) | 170.0 (14) |
| C7—H7···N3 | 0.95 | 2.54 | 3.3609 (15) | 145 |
| C11—H11*B*···N3 | 0.99 | 2.34 | 2.7996 (15) | 107 |

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