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# (*Z*)-3-(1*H*-Indol-3-yl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile

**Narsimha Reddy Penthala,[a] Sean Parkin[b] and Peter A. Crooks[a]***

[a]Department of Pharmaceutical Sciences, College of Pharmacy, University of Arkansas for Medical Sciences, Little Rock, AR 72205, USA, and [b]Department of Chemistry, University of Kentucky, Lexington, KY 40506, USA
Correspondence e-mail: pacrooks@uams.edu

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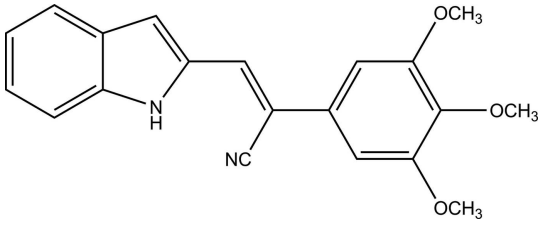
Key indicators: single-crystal X-ray study; $T$ = 90 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.037; $wR$ factor = 0.097; data-to-parameter ratio = 12.8.

In the title compound, $C_{20}H_{18}N_2O_3$, the C=C bond of the acrylonitrile group that links the indole and the 3,4,5-trimethoxyphenyl rings has $Z$ geometry, with dihedral angles between the plane of the acrylonitrile unit and the planes of the benzene and indole ring systems of 21.96 (5) and 38.94 (7)°, respectively. The acrylonitrile group is planar (r.m.s. deviation from planarity = 0.037 Å). Molecules are linked into head-to-tail chains that propagate along the *b*-axis direction by bifurcated N—H···O intermolecular hydrogen bonds, which form an $R_1^2(5)$ motif between the indole NH group and the two methoxy O atoms furthest from the nitrile group.

## Related literature

For biological activity of similar acrylonitriles, see: Naruto *et al.* (1983); Ohsumi *et al.* (1998); Saczewski *et al.* (2004). For the molecular structures of (*E*)-3-(benzo[*b*]thiophen-2-yl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile and (*Z*)-3-(benzo[*b*]thiophen-2-yl)-2-(3,4-dimethoxyphenyl)acrylonitrile, see: Sonar *et al.* (2007). For the structure of (*Z*)-4-[3-(2,5-dioxoimidazolidin-4-ylidenemethyl)-1*H*-indol-1-ylmethyl]benzonitrile, see: Penthala *et al.* (2008).




## Experimental

### Crystal data

$C_{20}H_{18}N_2O_3$
$M_r$ = 334.36
Monoclinic, $P2_1/c$
$a$ = 11.3384 (4) Å
$b$ = 21.1383 (8) Å
$c$ = 6.9570 (3) Å
$\beta$ = 93.610 (2)°
$V$ = 1664.11 (11) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.74 mm$^{-1}$
$T$ = 90 K
0.24 × 0.07 × 0.02 mm

### Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
$T_{min}$ = 0.843, $T_{max}$ = 0.929
20812 measured reflections
2979 independent reflections
2679 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.053

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
$S$ = 1.07
2979 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1N···O2$^{i}$ | 0.851 (18) | 2.075 (18) | 2.8635 (15) | 153.8 (16) |
| N1—H1N···O3$^{i}$ | 0.851 (18) | 2.301 (17) | 2.9476 (15) | 133.0 (15) |

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* and local procedures.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2125).

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Sonar, V. N., Parkin, S. & Crooks, P. A. (2007). *Acta Cryst.* C**63**, o743–o745.

# supplementary materials

*Acta Cryst.* (2012). E**68**, o729 [doi:10.1107/S1600536812005855]

## (*Z*)-3-(1*H*-Indol-3-yl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile

**Narsimha Reddy Penthala, Sean Parkin and Peter A. Crooks**

### Comment

A number of 2,3-diarylacrylonitrile analogs have been found to possess interesting biological properties such as spasmolytic (Naruto *et al.*, 1983), and cytotoxic activities (Ohsumi *et al.*, 1998 and Saczewski *et al.*, 2004). Previously, we have reported on the crystallographic data of benzothiophene acrylonitrile analogs (Sonar *et al.*, 2007). In continuation of the synthesis of structurally related analogs and to compare the structure–activity relationships of different substituted acrylonitrile analogs, we have now prepared the title compound, (I), by the reaction of indole-3-carbaldehyde with (3,4,5-trimethoxyphenyl)acetonitrile in methanolic sodium methoxide at reflux temperature. Recrystallization from methanol afforded yellow needles of (I) that were suitable for X-ray analysis. The X-ray studies revealed that the title compound is the *Z* isomer. The olefinic bond linking the indole ring and the 3,4,5-trimethoxyphenyl units has a planar atomic arrangement. The r.m.s. deviation from the mean plane passing through atoms of C1–C2–C3–C8–N1 is 0.0133 Å. The acrylonitrile group is planar (r.m.s. deviation from planarity is 0.037 Å). In the trimethoxyphenyl group, one methyl is essentially in the plane of the benzene ring and the three O atoms [deviation = 0.0253 (18) Å]. The middle methyl has the largest [deviation = 1.0600 (17) Å], while the methyl that is on the same side as the nitrile group is in between [0.3788 (19)Å out of plane]. Significant deviations from the ideal bond-angle geometry around the C$sp^2$ atoms of the double bond are observed. The C1–C2–C3 and C1–C2–C9, C4–C3–C2 and C8–C3–C2 bond angles [105.88 (12)°, 127.93 (13)°, 134.18 (13)°, 106.90 (12)°, respectively] are distorted owing to steric hindrance around the double bond linking the two ring systems. Neither the indole ring nor the benzene ring of the 3,4,5-trimethoxyphenyl group is coplanar with the vinyl double bond, making dihedral angles of 38.94 (7)° and 21.96 (5)° respectively. Molecules are linked into head-to-tail chains that propagate along the *b* axis direction by bifurcated N—H···O intermolecular hydrogen bonds that form an $R^2_1(5)$ motif between the indole NH and the two methoxy O atoms furthest from the nitrile group, as shown in Figure 2.

### Experimental

A mixture of indole-3-carbaldehyde (0.3 g, 2.06 mmol), and 2-(3,4,5-trimethoxyphenyl)acetonitrile (0.45 g, 2.17 mmol) were refluxed in methanolic 5% sodium methoxide solution for 5 hrs. The reaction mixture was cooled to room temperature and added to ice–cold water to afford a yellow crude solid, which was collected by filtration, washed with a 1:1 mixture of cold water and methanol, and suction–dried to afford the crude product. Crystallization during slow evaporation of methanol gave a yellow crystalline product of (*Z*)-3-(1*H*-indol-3-yl)-2- (3,4,5-trimethoxyphenyl)acrylonitrile that was suitable for X-ray crystallographic analysis. $^1$H NMR ($CDCl_3$): $\delta$ 3.91 (s, 3H), 3.93 (s, 6H), 6.89 (s, 2H), 7.26–7.35 (m, 2H), 7.47–7.50 (d, 1H), 7.79 (s, 1H), 7.81 (s, 1H), 8.45–8.46 (d, 1H), 8.91 (bs, 1H, NH); $^{13}$C NMR ($CDCl_3$): $\delta$ 56.57, 61.28, 102.92, 105.11, 106.03, 111.83, 112.91, 121.19, 121.75, 125.77, 127.47, 129.27, 130.94, 132.38, 138.19, 153.76.

### Refinement

H atoms were found in difference Fourier maps. H atoms attached to carbon were subsequently placed in idealized positions with constrained distances of 0.98 Å ($RCH_3$) and 0.95 Å ($C_{sp2}H$). The N—H hydrogen coordinates were freely refined, to a distance N—H = 0.851 (18) Å. $U_{iso}$(H) values were set to either $1.2U_{eq}$ or $1.5U_{eq}$ ($RCH_3$) of the attached atom.

### Computing details

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2* (Bruker, 2006); data reduction: *APEX2* (Bruker, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and local procedures.

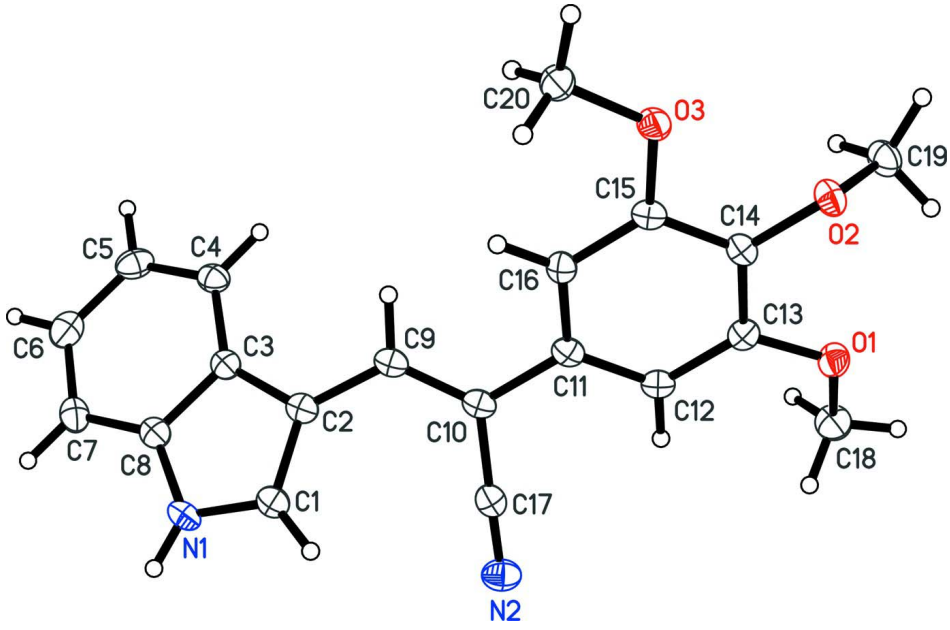


**Figure 1**

The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

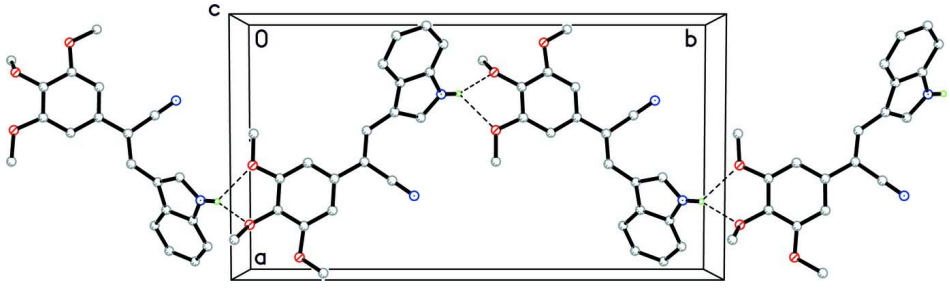


**Figure 2**

Bifurcated N–H···O bonding interactions (dashed lines) in the crystal structure of (I)

**(*Z*)-3-(1*H*-Indol-3-yl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile**

*Crystal data*

$C_{20}H_{18}N_2O_3$
$M_r = 334.36$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.3384$ (4) Å
$b = 21.1383$ (8) Å
$c = 6.9570$ (3) Å
$\beta = 93.610$ (2)°
$V = 1664.11$ (11) Å$^3$
$Z = 4$
$F(000) = 704$
$D_x = 1.335$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 9970 reflections
$\theta = 3.9$–$68.1°$
$\mu = 0.74$ mm$^{-1}$
$T = 90$ K
Lath, yellow
0.24 × 0.07 × 0.02 mm

*Data collection*

Bruker X8 Proteum diffractometer
Radiation source: fine-focus rotating anode
Graded multilayer optics monochromator
Detector resolution: 5.6 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS* in *APEX2*; Bruker, 2006)
$T_{min} = 0.843$, $T_{max} = 0.929$
20812 measured reflections
2979 independent reflections
2679 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 68.1°$, $\theta_{min} = 3.9°$
$h = -13 \rightarrow 13$
$k = -25 \rightarrow 25$
$l = -7 \rightarrow 8$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.097$
$S = 1.07$
2979 reflections
233 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.7731P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0016 (3)

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against all reflections. The weighted *R*-value *wR* and goodness of fit *S* are based on $F^2$. Conventional *R*-values *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*-values based on $F^2$ are statistically about twice as large as those based on *F*, and *R*-values based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.29609 (11) | 0.42543 (6) | 0.78546 (16) | 0.0203 (3) |
| H1N | 0.2929 (15) | 0.4617 (8) | 0.732 (2) | 0.024* |

| | | | | |
|---|---|---|---|---|
| N2 | 0.68309 (12) | 0.37056 (6) | 0.89979 (17) | 0.0251 (3) |
| O1 | 0.91232 (9) | 0.14011 (4) | 1.03760 (14) | 0.0220 (2) |
| O2 | 0.79293 (9) | 0.03501 (4) | 0.91759 (13) | 0.0202 (2) |
| O3 | 0.56732 (8) | 0.03817 (4) | 0.81372 (14) | 0.0203 (2) |
| C1 | 0.38533 (13) | 0.38300 (6) | 0.77536 (19) | 0.0195 (3) |
| H1 | 0.4520 | 0.3883 | 0.7009 | 0.023* |
| C2 | 0.36515 (12) | 0.33101 (6) | 0.88911 (18) | 0.0177 (3) |
| C3 | 0.25524 (12) | 0.34334 (6) | 0.97547 (18) | 0.0172 (3) |
| C4 | 0.18967 (13) | 0.31113 (6) | 1.1088 (2) | 0.0206 (3) |
| H4 | 0.2151 | 0.2712 | 1.1585 | 0.025* |
| C5 | 0.08734 (14) | 0.33867 (7) | 1.1662 (2) | 0.0253 (3) |
| H5 | 0.0426 | 0.3174 | 1.2574 | 0.030* |
| C6 | 0.04772 (14) | 0.39732 (7) | 1.0932 (2) | 0.0266 (3) |
| H6 | −0.0241 | 0.4145 | 1.1335 | 0.032* |
| C7 | 0.11116 (13) | 0.43046 (7) | 0.9640 (2) | 0.0225 (3) |
| H7 | 0.0850 | 0.4704 | 0.9153 | 0.027* |
| C8 | 0.21522 (13) | 0.40300 (6) | 0.90769 (19) | 0.0188 (3) |
| C9 | 0.43611 (12) | 0.27458 (6) | 0.91389 (18) | 0.0175 (3) |
| H9 | 0.3947 | 0.2367 | 0.9389 | 0.021* |
| C10 | 0.55447 (13) | 0.26926 (6) | 0.90571 (18) | 0.0170 (3) |
| C11 | 0.61787 (12) | 0.20801 (6) | 0.91199 (18) | 0.0166 (3) |
| C12 | 0.73704 (12) | 0.20546 (6) | 0.97301 (18) | 0.0173 (3) |
| H12 | 0.7781 | 0.2431 | 1.0103 | 0.021* |
| C13 | 0.79597 (12) | 0.14770 (6) | 0.97940 (18) | 0.0173 (3) |
| C14 | 0.73641 (12) | 0.09257 (6) | 0.92416 (18) | 0.0166 (3) |
| C15 | 0.61699 (12) | 0.09519 (6) | 0.86363 (18) | 0.0166 (3) |
| C16 | 0.55781 (12) | 0.15264 (6) | 0.85555 (18) | 0.0163 (3) |
| H16 | 0.4768 | 0.1544 | 0.8118 | 0.020* |
| C17 | 0.62542 (13) | 0.32580 (6) | 0.89881 (18) | 0.0185 (3) |
| C18 | 0.96763 (14) | 0.19066 (7) | 1.1428 (2) | 0.0263 (3) |
| H18A | 0.9721 | 0.2277 | 1.0590 | 0.039* |
| H18B | 1.0476 | 0.1780 | 1.1892 | 0.039* |
| H18C | 0.9214 | 0.2012 | 1.2529 | 0.039* |
| C19 | 0.84428 (13) | 0.01136 (7) | 1.0993 (2) | 0.0242 (3) |
| H19A | 0.9256 | 0.0269 | 1.1196 | 0.036* |
| H19B | 0.8445 | −0.0350 | 1.0971 | 0.036* |
| H19C | 0.7976 | 0.0262 | 1.2042 | 0.036* |
| C20 | 0.44638 (13) | 0.03775 (7) | 0.7438 (2) | 0.0227 (3) |
| H20A | 0.3972 | 0.0538 | 0.8440 | 0.034* |
| H20B | 0.4225 | −0.0056 | 0.7100 | 0.034* |
| H20C | 0.4363 | 0.0648 | 0.6295 | 0.034* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0269 (7) | 0.0156 (6) | 0.0183 (6) | 0.0011 (5) | −0.0004 (5) | 0.0033 (5) |
| N2 | 0.0311 (7) | 0.0229 (7) | 0.0218 (6) | −0.0038 (5) | 0.0048 (5) | −0.0011 (5) |
| O1 | 0.0174 (5) | 0.0210 (5) | 0.0270 (5) | −0.0010 (4) | −0.0031 (4) | −0.0036 (4) |
| O2 | 0.0221 (5) | 0.0176 (5) | 0.0202 (5) | 0.0035 (4) | −0.0040 (4) | −0.0021 (4) |
| O3 | 0.0198 (5) | 0.0160 (5) | 0.0245 (5) | −0.0015 (4) | −0.0042 (4) | −0.0019 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.0237 (8) | 0.0196 (7) | 0.0153 (6) | 0.0002 (6) | 0.0008 (6) | 0.0000 (5) |
| C2 | 0.0222 (7) | 0.0176 (7) | 0.0131 (6) | −0.0018 (5) | −0.0017 (5) | −0.0013 (5) |
| C3 | 0.0196 (7) | 0.0160 (7) | 0.0153 (6) | −0.0018 (5) | −0.0030 (5) | −0.0031 (5) |
| C4 | 0.0240 (8) | 0.0172 (7) | 0.0205 (7) | −0.0027 (6) | −0.0007 (6) | −0.0008 (5) |
| C5 | 0.0271 (8) | 0.0232 (7) | 0.0262 (8) | −0.0059 (6) | 0.0074 (6) | −0.0054 (6) |
| C6 | 0.0227 (8) | 0.0252 (8) | 0.0320 (8) | −0.0002 (6) | 0.0034 (6) | −0.0095 (6) |
| C7 | 0.0230 (8) | 0.0181 (7) | 0.0257 (7) | 0.0025 (6) | −0.0046 (6) | −0.0054 (5) |
| C8 | 0.0223 (7) | 0.0169 (7) | 0.0167 (6) | −0.0014 (5) | −0.0030 (6) | −0.0027 (5) |
| C9 | 0.0241 (8) | 0.0166 (7) | 0.0118 (6) | −0.0017 (5) | 0.0006 (5) | −0.0002 (5) |
| C10 | 0.0238 (8) | 0.0174 (7) | 0.0098 (6) | −0.0011 (5) | 0.0007 (5) | 0.0003 (5) |
| C11 | 0.0225 (7) | 0.0182 (7) | 0.0095 (6) | 0.0004 (5) | 0.0035 (5) | 0.0014 (5) |
| C12 | 0.0214 (7) | 0.0173 (7) | 0.0133 (6) | −0.0028 (5) | 0.0014 (5) | −0.0006 (5) |
| C13 | 0.0184 (7) | 0.0220 (7) | 0.0116 (6) | −0.0010 (5) | 0.0005 (5) | 0.0007 (5) |
| C14 | 0.0206 (7) | 0.0166 (7) | 0.0125 (6) | 0.0020 (5) | 0.0005 (5) | −0.0003 (5) |
| C15 | 0.0218 (7) | 0.0169 (7) | 0.0110 (6) | −0.0023 (5) | 0.0007 (5) | −0.0004 (5) |
| C16 | 0.0178 (7) | 0.0195 (7) | 0.0115 (6) | 0.0011 (5) | −0.0001 (5) | 0.0010 (5) |
| C17 | 0.0230 (7) | 0.0202 (7) | 0.0124 (6) | 0.0035 (6) | 0.0024 (5) | −0.0006 (5) |
| C18 | 0.0250 (8) | 0.0240 (8) | 0.0288 (8) | −0.0042 (6) | −0.0060 (6) | −0.0033 (6) |
| C19 | 0.0243 (8) | 0.0216 (7) | 0.0257 (7) | 0.0022 (6) | −0.0062 (6) | 0.0033 (6) |
| C20 | 0.0201 (8) | 0.0210 (7) | 0.0261 (7) | −0.0018 (6) | −0.0050 (6) | −0.0013 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.3572 (19) | C7—H7 | 0.9500 |
| N1—C8 | 1.3737 (19) | C9—C10 | 1.351 (2) |
| N1—H1N | 0.851 (18) | C9—H9 | 0.9500 |
| N2—C17 | 1.1499 (19) | C10—C17 | 1.4433 (19) |
| O1—C13 | 1.3649 (17) | C10—C11 | 1.4801 (18) |
| O1—C18 | 1.4188 (17) | C11—C12 | 1.392 (2) |
| O2—C14 | 1.3773 (16) | C11—C16 | 1.3980 (19) |
| O2—C19 | 1.4472 (17) | C12—C13 | 1.3910 (19) |
| O3—C15 | 1.3661 (16) | C12—H12 | 0.9500 |
| O3—C20 | 1.4257 (17) | C13—C14 | 1.3890 (19) |
| C1—C2 | 1.3818 (19) | C14—C15 | 1.394 (2) |
| C1—H1 | 0.9500 | C15—C16 | 1.3868 (19) |
| C2—C3 | 1.4409 (19) | C16—H16 | 0.9500 |
| C2—C9 | 1.4430 (19) | C18—H18A | 0.9800 |
| C3—C4 | 1.4011 (19) | C18—H18B | 0.9800 |
| C3—C8 | 1.4113 (19) | C18—H18C | 0.9800 |
| C4—C5 | 1.380 (2) | C19—H19A | 0.9800 |
| C4—H4 | 0.9500 | C19—H19B | 0.9800 |
| C5—C6 | 1.403 (2) | C19—H19C | 0.9800 |
| C5—H5 | 0.9500 | C20—H20A | 0.9800 |
| C6—C7 | 1.377 (2) | C20—H20B | 0.9800 |
| C6—H6 | 0.9500 | C20—H20C | 0.9800 |
| C7—C8 | 1.393 (2) | | |

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| C1—N1—C8 | 109.44 (12) | C12—C11—C10 | 120.23 (12) |
| C1—N1—H1N | 125.7 (11) | C16—C11—C10 | 119.80 (13) |
| C8—N1—H1N | 124.7 (11) | C13—C12—C11 | 119.93 (12) |

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| C13—O1—C18 | 116.87 (11) | C13—C12—H12 | 120.0 |
| C14—O2—C19 | 116.02 (10) | C11—C12—H12 | 120.0 |
| C15—O3—C20 | 117.67 (11) | O1—C13—C14 | 115.30 (12) |
| N1—C1—C2 | 110.19 (12) | O1—C13—C12 | 124.47 (12) |
| N1—C1—H1 | 124.9 | C14—C13—C12 | 120.23 (13) |
| C2—C1—H1 | 124.9 | O2—C14—C13 | 122.10 (12) |
| C1—C2—C3 | 105.88 (12) | O2—C14—C15 | 118.06 (12) |
| C1—C2—C9 | 127.93 (13) | C13—C14—C15 | 119.76 (12) |
| C3—C2—C9 | 126.16 (12) | O3—C15—C16 | 124.87 (13) |
| C4—C3—C8 | 118.85 (13) | O3—C15—C14 | 114.77 (11) |
| C4—C3—C2 | 134.18 (13) | C16—C15—C14 | 120.36 (12) |
| C8—C3—C2 | 106.90 (12) | C15—C16—C11 | 119.73 (13) |
| C5—C4—C3 | 118.52 (13) | C15—C16—H16 | 120.1 |
| C5—C4—H4 | 120.7 | C11—C16—H16 | 120.1 |
| C3—C4—H4 | 120.7 | N2—C17—C10 | 177.68 (14) |
| C4—C5—C6 | 121.61 (13) | O1—C18—H18A | 109.5 |
| C4—C5—H5 | 119.2 | O1—C18—H18B | 109.5 |
| C6—C5—H5 | 119.2 | H18A—C18—H18B | 109.5 |
| C7—C6—C5 | 121.15 (14) | O1—C18—H18C | 109.5 |
| C7—C6—H6 | 119.4 | H18A—C18—H18C | 109.5 |
| C5—C6—H6 | 119.4 | H18B—C18—H18C | 109.5 |
| C6—C7—C8 | 117.25 (13) | O2—C19—H19A | 109.5 |
| C6—C7—H7 | 121.4 | O2—C19—H19B | 109.5 |
| C8—C7—H7 | 121.4 | H19A—C19—H19B | 109.5 |
| N1—C8—C7 | 129.82 (13) | O2—C19—H19C | 109.5 |
| N1—C8—C3 | 107.59 (12) | H19A—C19—H19C | 109.5 |
| C7—C8—C3 | 122.59 (13) | H19B—C19—H19C | 109.5 |
| C10—C9—C2 | 127.70 (13) | O3—C20—H20A | 109.5 |
| C10—C9—H9 | 116.1 | O3—C20—H20B | 109.5 |
| C2—C9—H9 | 116.1 | H20A—C20—H20B | 109.5 |
| C9—C10—C17 | 119.30 (12) | O3—C20—H20C | 109.5 |
| C9—C10—C11 | 123.60 (12) | H20A—C20—H20C | 109.5 |
| C17—C10—C11 | 117.04 (12) | H20B—C20—H20C | 109.5 |
| C12—C11—C16 | 119.97 (12) | | |

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| C8—N1—C1—C2 | 0.67 (16) | C9—C10—C11—C16 | 23.89 (19) |
| N1—C1—C2—C3 | −0.36 (15) | C17—C10—C11—C16 | −158.85 (12) |
| N1—C1—C2—C9 | 177.84 (13) | C16—C11—C12—C13 | −0.58 (18) |
| C1—C2—C3—C4 | −176.92 (15) | C10—C11—C12—C13 | 179.70 (11) |
| C9—C2—C3—C4 | 4.8 (2) | C18—O1—C13—C14 | −163.42 (12) |
| C1—C2—C3—C8 | −0.07 (15) | C18—O1—C13—C12 | 16.64 (18) |
| C9—C2—C3—C8 | −178.31 (13) | C11—C12—C13—O1 | −179.74 (11) |
| C8—C3—C4—C5 | 1.0 (2) | C11—C12—C13—C14 | 0.32 (19) |
| C2—C3—C4—C5 | 177.59 (14) | C19—O2—C14—C13 | 62.90 (16) |
| C3—C4—C5—C6 | 0.6 (2) | C19—O2—C14—C15 | −120.39 (13) |
| C4—C5—C6—C7 | −1.5 (2) | O1—C13—C14—O2 | −3.80 (18) |
| C5—C6—C7—C8 | 0.7 (2) | C12—C13—C14—O2 | 176.14 (11) |
| C1—N1—C8—C7 | 179.00 (14) | O1—C13—C14—C15 | 179.55 (11) |
| C1—N1—C8—C3 | −0.69 (15) | C12—C13—C14—C15 | −0.51 (19) |

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| C6—C7—C8—N1 | −178.69 (14) | C20—O3—C15—C16 | 1.54 (18) |
| C6—C7—C8—C3 | 1.0 (2) | C20—O3—C15—C14 | −177.79 (11) |
| C4—C3—C8—N1 | 177.88 (12) | O2—C14—C15—O3 | 3.54 (17) |
| C2—C3—C8—N1 | 0.46 (15) | C13—C14—C15—O3 | −179.67 (11) |
| C4—C3—C8—C7 | −1.8 (2) | O2—C14—C15—C16 | −175.82 (11) |
| C2—C3—C8—C7 | −179.26 (13) | C13—C14—C15—C16 | 0.97 (19) |
| C1—C2—C9—C10 | 31.1 (2) | O3—C15—C16—C11 | 179.48 (11) |
| C3—C2—C9—C10 | −151.04 (14) | C14—C15—C16—C11 | −1.22 (18) |
| C2—C9—C10—C17 | 9.7 (2) | C12—C11—C16—C15 | 1.03 (18) |
| C2—C9—C10—C11 | −173.07 (12) | C10—C11—C16—C15 | −179.25 (11) |
| C9—C10—C11—C12 | −156.39 (13) | C9—C10—C17—N2 | 103 (4) |
| C17—C10—C11—C12 | 20.87 (17) | C11—C10—C17—N2 | −74 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O2$^{i}$ | 0.851 (18) | 2.075 (18) | 2.8635 (15) | 153.8 (16) |
| N1—H1*N*···O3$^{i}$ | 0.851 (18) | 2.301 (17) | 2.9476 (15) | 133.0 (15) |

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