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# 2-Amino-4-(4-fluorophenyl)-6-methoxy-4*H*-benzo[*h*]chromene-3-carbonitrile

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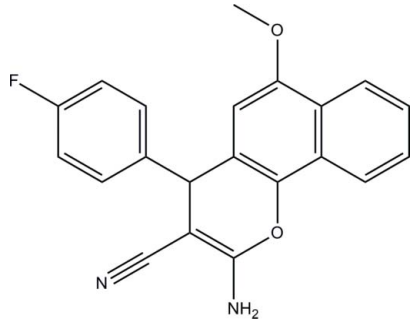
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.047; $wR$ factor = 0.132; data-to-parameter ratio = 13.1.

In the title molecule, $C_{21}H_{15}FN_2O_2$, the dihedral angle between the fluoro-substituted benzene ring and the mean plane of the 4*H*-benzo[*h*]chromene ring system [maximum deviation = 0.109 (2) Å] is 83.35 (7)°. The pyran ring adopts a slight sofa conformation with the tertiary C(H) atom forming the flap. The methoxy group is slightly twisted from the attached benzene ring of the 4*H*-benzo[*h*]chromene moiety [C—O—C—C = −4.3 (3)°]. In the crystal, molecules are linked by intermolecular N—H···N hydrogen bonds into infinite wave-like chains along the *b* axis. The crystal packing is further stabilized by $\pi$–$\pi$ interactions [centroid–centroid distance = 3.7713 (9) Å].

## Related literature

For the synthesis of 4*H*-chromene derivatives, see: Sayed *et al.* (2000); Bedair *et al.* (2001); El-Agrody *et al.* (2000, 2002). For the chemical and pharmacological properties of 4*H*-chromene and fused 4*H*-chromene derivatives, see: El-Agrody *et al.* (2000); Abd-El-Aziz *et al.* (2004, 2007); Sabry *et al.* (2011). For ring puckering parameters, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_{21}H_{15}FN_2O_2$
$M_r$ = 346.35
Monoclinic, $P2_1/c$
$a$ = 12.6336 (4) Å
$b$ = 11.9333 (3) Å
$c$ = 12.0471 (4) Å
$\beta$ = 113.581 (2)°
$V$ = 1664.56 (9) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.81 mm$^{-1}$
$T$ = 296 K
0.88 × 0.68 × 0.06 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.537, $T_{max}$ = 0.953
11390 measured reflections
3199 independent reflections
2770 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.132
$S$ = 1.05
3199 reflections
245 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H2*N*1···N2$^{i}$ | 0.89 (2) | 2.17 (2) | 3.054 (2) | 175 (2) |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

HKF and TSC thank Universiti Sains Malaysia (USM) for the Research University Grant (1001/PFIZIK/811160). TSC also thanks the Malaysian Government and USM for the award of a research fellowship. The authors extend their appreciation to the Deanship of Scientific Research at King Saud University for funding the work through the research group project No. RGP-VPP-172.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5476).

‡ Thomson Reuters ResearcherID: A-3561-2009.

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o1934–o1935 [doi:10.1107/S1600536812023021]

## 2-Amino-4-(4-fluorophenyl)-6-methoxy-4*H*-benzo[*h*]chromene-3-carbonitrile

**Al-Anood M. Al-Dies, Abdel-Galil E. Amr, Ahmed M. El-Agrody, Tze Shyang Chia and Hoong-Kun Fun**

### Comment

In our previous work, we have reported the synthesis of 4*H*-chromene derivatives using *α*-cyanocinnamonitriles and ethyl *α*-cyanocinnamates (Sayed *et al.*, 2000; Bedair *et al.*, 2001; El-Agrody *et al.*, 2000, 2002), study of their characterization and evaluation of their antimicrobial and antitumor activities. In continuation of our interest in the chemical and pharmacological properties of 4*H*-chromene and fused 4*H*-chromene derivatives (El-Agrody *et al.*, 2000; Abd-El-Aziz *et al.*, 2004, 2007; Sabry *et al.*, 2011), we report herein the crystal structure of the title compound.

The asymmetric unit of the title compound is shown in Fig. 1. The fluoro-substituted benzene ring (C14–C19) is approximately perpendicular to the 4*H*-benzo[*h*]chromene ring system [O1/C1–C13, maximum deviation = 0.109 (2) Å at atom C3] as indicated by the dihedral angle of 83.35 (7)°. The pyran ring (O1/C1–C5) adopts slight sofa conformation [puckering parameters (Cremer & Pople, 1975), $Q$ = 0.0980 (16) Å, $\theta$ = 69.5 (9)° and $\varphi$ = 167.7 (10)°] with C3 as the flap atom. The methoxy group (C20/O2) is slightly twisted from the attached benzene ring (C4–C6/C11–C13) of the 4*H*-benzo[*h*]chromene moiety with torsion angle C20—O2—C12—C13 of -4.3 (3)°.

In the crystal (Fig. 2), molecules are linked by intermolecular N1—H2N1···N2$^{i}$ hydrogen bonds (Table 1) into infinite wave-like chains along *b* axis. The crystal packing is further stabilized by *π*–*π* interaction with *Cg*1-*Cg*1 distance of 3.7713 (9) Å, where *Cg*1 is the centroid of O1/C1–C5 ring [symmetry code: 1-*x*, 1-*y*, -*z*].

### Experimental

A solution of 4-methoxy-1-naphthol (0.01 mol) in EtOH (30 ml) was treated with *α*-cyano-*p*-fluorocinnamonitrile (0.01 mol) and piperidine (0.5 ml). The reaction mixture was heated until complete precipitation occurred (reaction time: 60 min). The solid product formed was collected by filtration and recrystallized from ethanol to give the title compound. *M.p.*: 493–494 K.

### Refinement

The atoms H2N1 and H1N1 were located in a difference fourier map and refined freely [N—H = 0.89 (2) and 0.90 (2) Å]. The remaining H atoms were positioned geometrically [C—H = 0.93, 0.96 and 0.98 Å] and refined using a riding model with $U_{iso}$(H) = 1.2 or 1.5 $U_{eq}$(C). A rotating group model was applied to the methyl group.

### Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

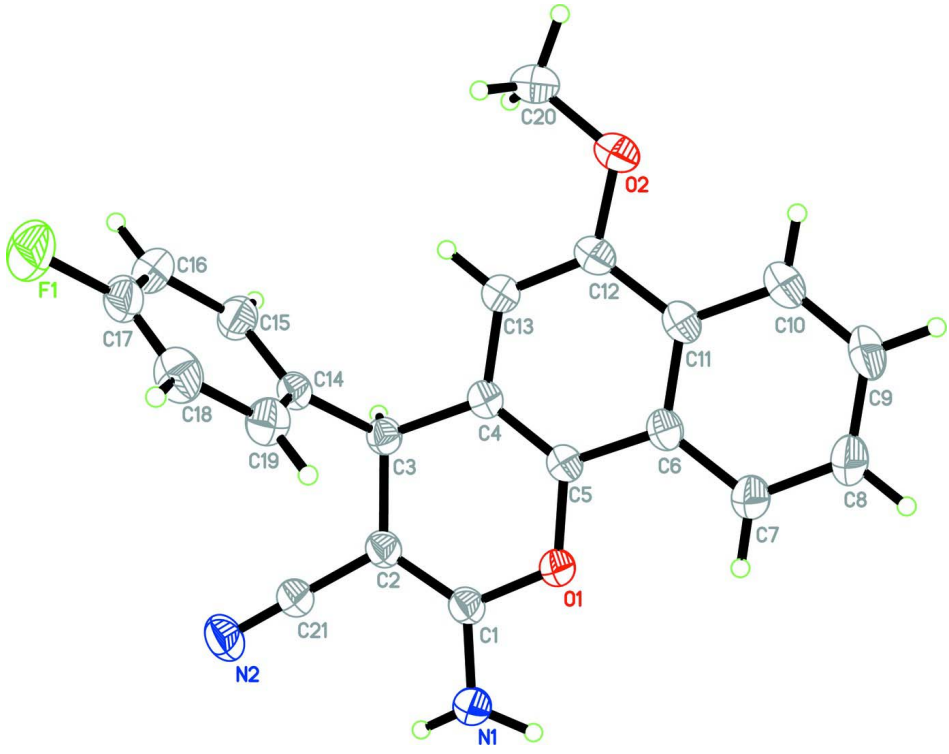


**Figure 1**

The molecular structure of the title compound with 30% probability displacement ellipsoids.

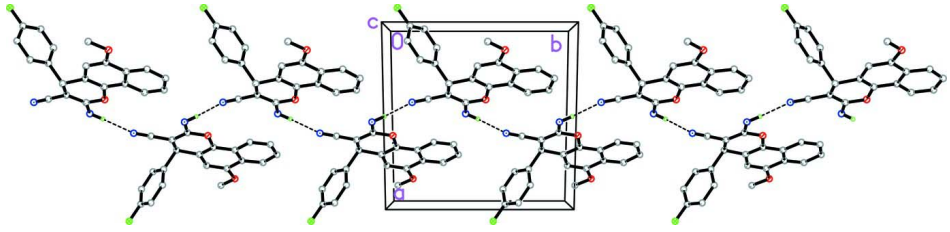


**Figure 2**

The crystal packing of the title compound. The dashed lines represent the hydrogen bonds. For clarity, hydrogen atoms not involved in hydrogen bonding have been omitted.

### 2-Amino-4-(4-fluorophenyl)-6-methoxy-4*H*-benzo[*h*]chromene- 3-carbonitrile

*Crystal data*

$C_{21}H_{15}FN_2O_2$
$M_r = 346.35$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.6336$ (4) Å
$b = 11.9333$ (3) Å
$c = 12.0471$ (4) Å
$\beta = 113.581$ (2)°
$V = 1664.56$ (9) Å$^3$
$Z = 4$

$F(000) = 720$
$D_x = 1.382$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 3014 reflections
$\theta = 3.8$–71.5°
$\mu = 0.81$ mm$^{-1}$
$T = 296$ K
Plate, yellow
0.88 × 0.68 × 0.06 mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.537, $T_{max}$ = 0.953
11390 measured reflections
3199 independent reflections
2770 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035
$\theta_{max}$ = 71.9°, $\theta_{min}$ = 3.8°
$h$ = −15→15
$k$ = −12→14
$l$ = −12→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.132
$S$ = 1.05
3199 reflections
245 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
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.3162P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0030 (5)

*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 $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F1 | 1.08340 (11) | 0.92571 (13) | 0.19221 (15) | 0.0946 (5) |
| O1 | 0.59888 (10) | 0.46944 (9) | 0.14761 (10) | 0.0507 (3) |
| O2 | 0.85128 (13) | 0.37355 (11) | −0.12356 (13) | 0.0677 (4) |
| N1 | 0.52279 (14) | 0.57508 (14) | 0.24812 (14) | 0.0569 (4) |
| N2 | 0.58458 (15) | 0.85793 (13) | 0.21110 (16) | 0.0666 (4) |
| C1 | 0.58225 (13) | 0.57477 (13) | 0.17674 (13) | 0.0442 (3) |
| C2 | 0.62297 (13) | 0.66625 (13) | 0.13969 (13) | 0.0439 (3) |
| C3 | 0.68193 (12) | 0.66037 (12) | 0.05215 (13) | 0.0416 (3) |
| H3A | 0.6274 | 0.6888 | −0.0262 | 0.050* |
| C4 | 0.70666 (12) | 0.53883 (12) | 0.03481 (12) | 0.0412 (3) |
| C5 | 0.66493 (12) | 0.45325 (12) | 0.07952 (13) | 0.0422 (3) |
| C6 | 0.68132 (12) | 0.33927 (13) | 0.05802 (13) | 0.0437 (3) |
| C7 | 0.63629 (15) | 0.24988 (14) | 0.10278 (16) | 0.0541 (4) |
| H7A | 0.5972 | 0.2650 | 0.1522 | 0.065* |
| C8 | 0.64953 (19) | 0.14210 (15) | 0.07431 (19) | 0.0656 (5) |

| | | | | |
|---|---|---|---|---|
| H8A | 0.6191 | 0.0841 | 0.1040 | 0.079* |
| C9 | 0.70845 (18) | 0.11761 (15) | 0.00090 (19) | 0.0683 (5) |
| H9A | 0.7161 | 0.0435 | −0.0187 | 0.082* |
| C10 | 0.75491 (17) | 0.20137 (15) | −0.04230 (17) | 0.0592 (4) |
| H10A | 0.7947 | 0.1839 | −0.0904 | 0.071* |
| C11 | 0.74309 (13) | 0.31434 (13) | −0.01470 (14) | 0.0466 (4) |
| C12 | 0.78985 (14) | 0.40492 (14) | −0.05799 (14) | 0.0490 (4) |
| C13 | 0.77181 (13) | 0.51312 (13) | −0.03431 (14) | 0.0469 (4) |
| H13A | 0.8026 | 0.5710 | −0.0638 | 0.056* |
| C14 | 0.79036 (13) | 0.73268 (12) | 0.09176 (13) | 0.0433 (3) |
| C15 | 0.80585 (16) | 0.80610 (15) | 0.01145 (16) | 0.0573 (4) |
| H15A | 0.7487 | 0.8125 | −0.0664 | 0.069* |
| C16 | 0.90543 (18) | 0.87111 (17) | 0.04453 (19) | 0.0682 (5) |
| H16A | 0.9157 | 0.9203 | −0.0102 | 0.082* |
| C17 | 0.98691 (16) | 0.86051 (16) | 0.15903 (19) | 0.0629 (5) |
| C18 | 0.97531 (16) | 0.78960 (18) | 0.24158 (19) | 0.0675 (5) |
| H18A | 1.0327 | 0.7844 | 0.3194 | 0.081* |
| C19 | 0.87556 (15) | 0.72490 (15) | 0.20702 (16) | 0.0567 (4) |
| H19A | 0.8663 | 0.6758 | 0.2624 | 0.068* |
| C20 | 0.8921 (2) | 0.46109 (19) | −0.1767 (2) | 0.0726 (6) |
| H20A | 0.9294 | 0.4291 | −0.2248 | 0.109* |
| H20B | 0.9463 | 0.5066 | −0.1139 | 0.109* |
| H20C | 0.8282 | 0.5064 | −0.2271 | 0.109* |
| C21 | 0.60126 (14) | 0.77220 (13) | 0.17819 (14) | 0.0479 (4) |
| H2N1 | 0.4932 (18) | 0.510 (2) | 0.2566 (19) | 0.065 (6)* |
| H1N1 | 0.4912 (19) | 0.642 (2) | 0.251 (2) | 0.073 (6)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.0660 (7) | 0.0911 (9) | 0.1290 (11) | −0.0335 (7) | 0.0416 (7) | −0.0317 (8) |
| O1 | 0.0622 (7) | 0.0378 (6) | 0.0650 (7) | −0.0010 (5) | 0.0391 (5) | −0.0020 (5) |
| O2 | 0.0845 (9) | 0.0567 (8) | 0.0838 (9) | 0.0070 (6) | 0.0566 (7) | −0.0061 (6) |
| N1 | 0.0703 (9) | 0.0447 (8) | 0.0724 (9) | −0.0022 (7) | 0.0461 (8) | −0.0035 (7) |
| N2 | 0.0771 (10) | 0.0491 (9) | 0.0787 (10) | 0.0032 (7) | 0.0364 (8) | −0.0149 (7) |
| C1 | 0.0464 (7) | 0.0404 (8) | 0.0472 (8) | 0.0021 (6) | 0.0202 (6) | −0.0029 (6) |
| C2 | 0.0450 (7) | 0.0399 (8) | 0.0486 (8) | 0.0018 (6) | 0.0206 (6) | −0.0025 (6) |
| C3 | 0.0441 (7) | 0.0378 (7) | 0.0420 (7) | 0.0014 (6) | 0.0162 (6) | 0.0007 (6) |
| C4 | 0.0429 (7) | 0.0383 (7) | 0.0411 (7) | 0.0010 (5) | 0.0155 (6) | −0.0019 (6) |
| C5 | 0.0444 (7) | 0.0390 (7) | 0.0440 (7) | 0.0017 (6) | 0.0185 (6) | −0.0025 (6) |
| C6 | 0.0433 (7) | 0.0392 (8) | 0.0443 (7) | 0.0030 (6) | 0.0131 (6) | −0.0014 (6) |
| C7 | 0.0605 (9) | 0.0427 (8) | 0.0615 (9) | 0.0019 (7) | 0.0269 (8) | 0.0031 (7) |
| C8 | 0.0794 (12) | 0.0396 (9) | 0.0821 (12) | 0.0006 (8) | 0.0366 (10) | 0.0040 (8) |
| C9 | 0.0804 (13) | 0.0379 (9) | 0.0857 (13) | 0.0083 (8) | 0.0322 (10) | −0.0052 (9) |
| C10 | 0.0653 (10) | 0.0477 (9) | 0.0664 (10) | 0.0085 (8) | 0.0283 (8) | −0.0084 (8) |
| C11 | 0.0460 (8) | 0.0428 (8) | 0.0471 (8) | 0.0058 (6) | 0.0146 (6) | −0.0042 (6) |
| C12 | 0.0494 (8) | 0.0503 (9) | 0.0499 (8) | 0.0046 (7) | 0.0227 (6) | −0.0053 (7) |
| C13 | 0.0506 (8) | 0.0447 (8) | 0.0488 (8) | −0.0014 (6) | 0.0234 (6) | −0.0021 (6) |
| C14 | 0.0455 (7) | 0.0367 (7) | 0.0509 (8) | 0.0004 (6) | 0.0227 (6) | −0.0051 (6) |
| C15 | 0.0639 (10) | 0.0539 (10) | 0.0538 (9) | −0.0093 (8) | 0.0232 (8) | 0.0002 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.0781 (13) | 0.0595 (11) | 0.0786 (12) | −0.0178 (9) | 0.0434 (11) | −0.0029 (9) |
| C17 | 0.0534 (9) | 0.0539 (10) | 0.0883 (13) | −0.0127 (8) | 0.0357 (9) | −0.0207 (9) |
| C18 | 0.0507 (10) | 0.0709 (12) | 0.0684 (11) | −0.0004 (8) | 0.0107 (8) | −0.0124 (9) |
| C19 | 0.0566 (9) | 0.0543 (10) | 0.0550 (9) | −0.0006 (7) | 0.0180 (7) | 0.0028 (7) |
| C20 | 0.0858 (14) | 0.0711 (13) | 0.0848 (13) | −0.0002 (10) | 0.0591 (12) | −0.0052 (10) |
| C21 | 0.0510 (8) | 0.0442 (9) | 0.0516 (8) | −0.0004 (6) | 0.0237 (7) | −0.0039 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C17 | 1.364 (2) | C8—C9 | 1.396 (3) |
| O1—C1 | 1.3440 (18) | C8—H8A | 0.9300 |
| O1—C5 | 1.3979 (18) | C9—C10 | 1.364 (3) |
| O2—C12 | 1.3627 (19) | C9—H9A | 0.9300 |
| O2—C20 | 1.425 (2) | C10—C11 | 1.411 (2) |
| N1—C1 | 1.349 (2) | C10—H10A | 0.9300 |
| N1—H2N1 | 0.89 (2) | C11—C12 | 1.427 (2) |
| N1—H1N1 | 0.90 (2) | C12—C13 | 1.361 (2) |
| N2—C21 | 1.147 (2) | C13—H13A | 0.9300 |
| C1—C2 | 1.356 (2) | C14—C15 | 1.376 (2) |
| C2—C21 | 1.411 (2) | C14—C19 | 1.378 (2) |
| C2—C3 | 1.517 (2) | C15—C16 | 1.394 (3) |
| C3—C4 | 1.516 (2) | C15—H15A | 0.9300 |
| C3—C14 | 1.525 (2) | C16—C17 | 1.357 (3) |
| C3—H3A | 0.9800 | C16—H16A | 0.9300 |
| C4—C5 | 1.356 (2) | C17—C18 | 1.358 (3) |
| C4—C13 | 1.419 (2) | C18—C19 | 1.392 (3) |
| C5—C6 | 1.415 (2) | C18—H18A | 0.9300 |
| C6—C7 | 1.413 (2) | C19—H19A | 0.9300 |
| C6—C11 | 1.418 (2) | C20—H20A | 0.9600 |
| C7—C8 | 1.359 (3) | C20—H20B | 0.9600 |
| C7—H7A | 0.9300 | C20—H20C | 0.9600 |
| | | | |
| C1—O1—C5 | 118.35 (12) | C9—C10—H10A | 119.7 |
| C12—O2—C20 | 116.83 (14) | C11—C10—H10A | 119.7 |
| C1—N1—H2N1 | 115.9 (14) | C10—C11—C6 | 118.83 (15) |
| C1—N1—H1N1 | 113.3 (15) | C10—C11—C12 | 122.65 (15) |
| H2N1—N1—H1N1 | 124.2 (19) | C6—C11—C12 | 118.51 (14) |
| O1—C1—N1 | 110.73 (14) | C13—C12—O2 | 124.33 (15) |
| O1—C1—C2 | 123.18 (13) | C13—C12—C11 | 120.91 (14) |
| N1—C1—C2 | 126.07 (15) | O2—C12—C11 | 114.76 (14) |
| C1—C2—C21 | 117.63 (13) | C12—C13—C4 | 120.87 (15) |
| C1—C2—C3 | 123.24 (13) | C12—C13—H13A | 119.6 |
| C21—C2—C3 | 118.96 (13) | C4—C13—H13A | 119.6 |
| C4—C3—C2 | 109.01 (12) | C15—C14—C19 | 118.55 (15) |
| C4—C3—C14 | 112.07 (12) | C15—C14—C3 | 120.19 (14) |
| C2—C3—C14 | 112.70 (12) | C19—C14—C3 | 121.25 (14) |
| C4—C3—H3A | 107.6 | C14—C15—C16 | 121.29 (17) |
| C2—C3—H3A | 107.6 | C14—C15—H15A | 119.4 |
| C14—C3—H3A | 107.6 | C16—C15—H15A | 119.4 |
| C5—C4—C13 | 118.64 (14) | C17—C16—C15 | 118.01 (18) |

| | | | |
|---|---|---|---|
| C5—C4—C3 | 122.12 (13) | C17—C16—H16A | 121.0 |
| C13—C4—C3 | 119.19 (13) | C15—C16—H16A | 121.0 |
| C4—C5—O1 | 123.18 (13) | C16—C17—C18 | 122.84 (17) |
| C4—C5—C6 | 122.86 (14) | C16—C17—F1 | 118.04 (19) |
| O1—C5—C6 | 113.93 (13) | C18—C17—F1 | 119.11 (19) |
| C7—C6—C5 | 123.01 (14) | C17—C18—C19 | 118.47 (17) |
| C7—C6—C11 | 118.83 (14) | C17—C18—H18A | 120.8 |
| C5—C6—C11 | 118.13 (14) | C19—C18—H18A | 120.8 |
| C8—C7—C6 | 120.61 (17) | C14—C19—C18 | 120.84 (17) |
| C8—C7—H7A | 119.7 | C14—C19—H19A | 119.6 |
| C6—C7—H7A | 119.7 | C18—C19—H19A | 119.6 |
| C7—C8—C9 | 120.59 (18) | O2—C20—H20A | 109.5 |
| C7—C8—H8A | 119.7 | O2—C20—H20B | 109.5 |
| C9—C8—H8A | 119.7 | H20A—C20—H20B | 109.5 |
| C10—C9—C8 | 120.56 (16) | O2—C20—H20C | 109.5 |
| C10—C9—H9A | 119.7 | H20A—C20—H20C | 109.5 |
| C8—C9—H9A | 119.7 | H20B—C20—H20C | 109.5 |
| C9—C10—C11 | 120.55 (17) | N2—C21—C2 | 179.06 (19) |

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|---|---|---|---|
| C5—O1—C1—N1 | −176.40 (13) | C9—C10—C11—C12 | −179.82 (17) |
| C5—O1—C1—C2 | 2.4 (2) | C7—C6—C11—C10 | 1.6 (2) |
| O1—C1—C2—C21 | −178.96 (14) | C5—C6—C11—C10 | −176.68 (14) |
| N1—C1—C2—C21 | −0.3 (2) | C7—C6—C11—C12 | −179.03 (14) |
| O1—C1—C2—C3 | 5.8 (2) | C5—C6—C11—C12 | 2.7 (2) |
| N1—C1—C2—C3 | −175.55 (15) | C20—O2—C12—C13 | −4.3 (3) |
| C1—C2—C3—C4 | −10.64 (19) | C20—O2—C12—C11 | 175.28 (16) |
| C21—C2—C3—C4 | 174.21 (13) | C10—C11—C12—C13 | 176.76 (16) |
| C1—C2—C3—C14 | −135.72 (15) | C6—C11—C12—C13 | −2.6 (2) |
| C21—C2—C3—C14 | 49.13 (18) | C10—C11—C12—O2 | −2.8 (2) |
| C2—C3—C4—C5 | 8.57 (19) | C6—C11—C12—O2 | 177.82 (14) |
| C14—C3—C4—C5 | 134.03 (14) | O2—C12—C13—C4 | 179.97 (14) |
| C2—C3—C4—C13 | −173.90 (12) | C11—C12—C13—C4 | 0.4 (2) |
| C14—C3—C4—C13 | −48.44 (17) | C5—C4—C13—C12 | 1.6 (2) |
| C13—C4—C5—O1 | −179.35 (13) | C3—C4—C13—C12 | −176.02 (14) |
| C3—C4—C5—O1 | −1.8 (2) | C4—C3—C14—C15 | 106.56 (16) |
| C13—C4—C5—C6 | −1.4 (2) | C2—C3—C14—C15 | −130.03 (15) |
| C3—C4—C5—C6 | 176.10 (13) | C4—C3—C14—C19 | −72.26 (18) |
| C1—O1—C5—C4 | −4.4 (2) | C2—C3—C14—C19 | 51.14 (19) |
| C1—O1—C5—C6 | 177.49 (12) | C19—C14—C15—C16 | 0.4 (3) |
| C4—C5—C6—C7 | −178.91 (15) | C3—C14—C15—C16 | −178.43 (16) |
| O1—C5—C6—C7 | −0.8 (2) | C14—C15—C16—C17 | −0.4 (3) |
| C4—C5—C6—C11 | −0.7 (2) | C15—C16—C17—C18 | 0.1 (3) |
| O1—C5—C6—C11 | 177.36 (12) | C15—C16—C17—F1 | −178.75 (17) |
| C5—C6—C7—C8 | 176.61 (16) | C16—C17—C18—C19 | 0.1 (3) |
| C11—C6—C7—C8 | −1.6 (2) | F1—C17—C18—C19 | 178.99 (16) |
| C6—C7—C8—C9 | 0.4 (3) | C15—C14—C19—C18 | −0.2 (3) |
| C7—C8—C9—C10 | 0.8 (3) | C3—C14—C19—C18 | 178.68 (15) |
| C8—C9—C10—C11 | −0.7 (3) | C17—C18—C19—C14 | −0.1 (3) |
| C9—C10—C11—C6 | −0.5 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H2*N*1···N2$^{i}$ | 0.89 (2) | 2.17 (2) | 3.054 (2) | 175 (2) |

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