



Crystal structure of 2-methylamino-3-nitro-4-*p*-tolylpyrano[3,2-*c*]chromen-5(4*H*)-one

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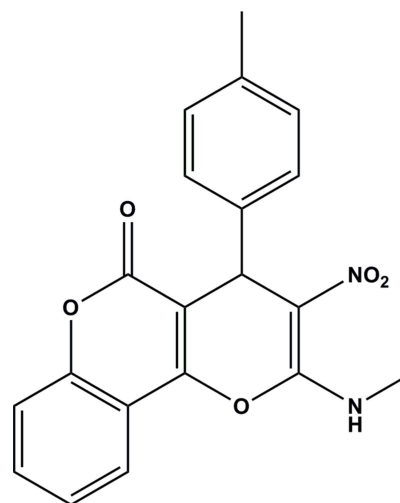
In the racemic title compound, C₂₀H₁₆N₂O₅, the pyran ring adopts a shallow envelope conformation, with the stereogenic C atom displaced from the other atoms by 0.273 (2) Å. The dihedral angle between the fused-ring system and the pendant *p*-tolyl group is 87.62 (7)°. The molecular conformation is consolidated by an intramolecular N—H···O hydrogen bond, which generates an *S*(6) ring. In the crystal, molecules are linked by C—H···O interactions, resulting in [010] chains.

Keywords: crystal structure; pyrano[3,2-*c*]chromenone; biological activity; chromene derivatives; hydrogen bonding; crystal structure.

CCDC reference: 1046918

1. Related literature

For background to the biological activity of chromene derivatives, see: Borges *et al.* (2005, 2009); Gibbs (2000); Varmus (2006). For a related structure, see: Narayanan *et al.* (2013).



2. Experimental

2.1. Crystal data

| | |
|---|--------------------------------------|
| C ₂₀ H ₁₆ N ₂ O ₅ | <i>V</i> = 1664.5 (3) Å ³ |
| <i>M_r</i> = 364.35 | <i>Z</i> = 4 |
| Monoclinic, <i>P</i> 2 ₁ / <i>n</i> | Mo <i>K</i> α radiation |
| <i>a</i> = 10.8336 (11) Å | <i>μ</i> = 0.11 mm ⁻¹ |
| <i>b</i> = 11.7927 (11) Å | <i>T</i> = 293 K |
| <i>c</i> = 13.7275 (14) Å | 0.21 × 0.19 × 0.18 mm |
| <i>β</i> = 108.357 (2)° | |

2.2. Data collection

| | |
|---|---|
| Bruker SMART APEXII CCD diffractometer | 28712 measured reflections |
| Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2008) | 4571 independent reflections |
| <i>T</i> _{min} = 0.978, <i>T</i> _{max} = 0.981 | 2862 reflections with <i>I</i> > 2σ(<i>I</i>) |
| | <i>R</i> _{int} = 0.039 |

2.3. Refinement

| | |
|---|---|
| <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.050 | 244 parameters |
| <i>wR</i> (<i>F</i> ²) = 0.151 | H-atom parameters constrained |
| <i>S</i> = 1.03 | Δρ _{max} = 0.26 e Å ⁻³ |
| 4571 reflections | Δρ _{min} = -0.36 e Å ⁻³ |

Table 1
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> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1···O5 | 0.86 | 1.96 | 2.590 (2) | 129 |
| C9—H9C···O4 ⁱ | 0.96 | 2.46 | 3.389 (3) | 163 |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7345).

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supporting information

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Crystal structure of 2-methylamino-3-nitro-4-*p*-tolylpyrano[3,2-*c*]chromen-5(4*H*)-one

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S1. Comment

Coumarins and their natural synthetic derivatives are pharmacologically interesting compounds due to their structural diversity and synthetic accessibility (Borges *et al.*, 2005, 2009). Cancer, a diverse group of diseases characterized by uncontrolled growth of abnormal cells, is a major worldwide problem. It is a fatal disease standing next to the cardiovascular disease in terms of morbidity and mortality. Although the cancer research has led to a number of new and effective solutions, the medicines used as treatments have clear limitations and unfortunately cancer is projected as the primary cause of death in the future (Gibbs *et al.* 2000; Varmus *et al.* 2006).

The title compound, Fig. 1, consists of a chromene moiety attached to a nitrophenyl ring, a nitro group and a methylamine group. The molecular structure is stabilized by an intra molecular N1—H1A···O5 interaction, which generates an S(6) ring motif. The chromen ring is almost coplanar with the least-square planes of the phenyl ring, making dihedral angle of 87.18 (8)°. The six-membered pyran ring (C7/O1/C8/C10/C11/C12) adopts sofa conformations, with puckering parameters $Q_2 = 0.1787$ (17) Å, $Q_3 = -0.0688$ (18) Å and $\varphi_2 = 0.9$ (6)°, respectively. The C18 atom deviates from mean plane of the phenyl ring by -0.0260 Å. The title compound exhibits structural similarities with an already reported related structure (Narayanan *et al.* 2013).

In the crystal, the molecules are linked *via* intermolecular C9—H9C···O4 hydrogen-bond interaction to generate [010] chains.

S2. Experimental

A solution of 4-methylbenzaldehyde (1.0 mmol), 4-hydroxycoumarin (1.0 mmol), NMSM (1.0 mmol), and piperidine (0.2 equiv) in EtOH (2 ml) was stirred for the three hours. After reaction was complete as indicated by TLC, the product was filtered and washed with EtOH (2 ml) to remove the excess base and other impurities. Finally, the product was recrystallized from EtOH to yield colourless blocks of the title compound.

S3. Refinement

N and C-bound H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for all other H atoms.

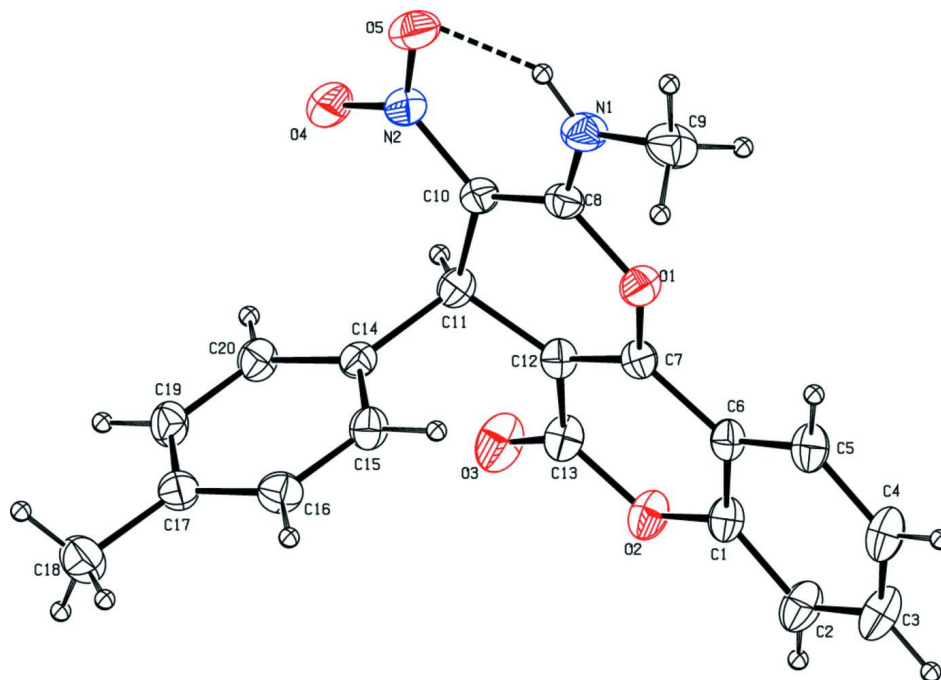
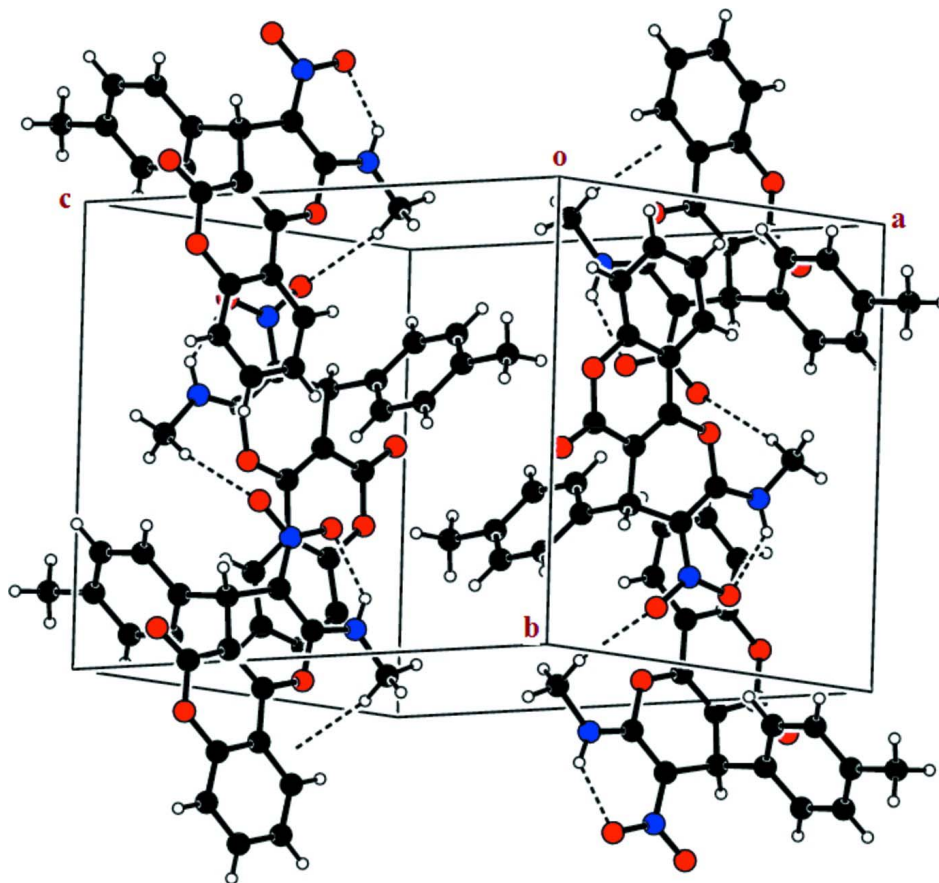


Figure 1

The molecular structure of the title molecule, with atom displacement ellipsoids drawn at the 30% probability level. The intramolecular N—H \cdots O hydrogen bond, which generates an S(6) ring motif, is shown as a dashed line.

**Figure 2**

The crystal packing of the title compound, viewed along the *b* axis, showing C9—H9C···O4 hydrogen bonds producing chains parallel to the 101 planes.

2-Methylamino-3-nitro-4-*p*-tolylpyrano[3,2-*c*]chromen-5(4*H*)-one

Crystal data

$C_{20}H_{16}N_2O_5$
 $M_r = 364.35$
 Monoclinic, $P2_1/n$
 Hall symbol: -P 2yn
 $a = 10.8336$ (11) Å
 $b = 11.7927$ (11) Å
 $c = 13.7275$ (14) Å
 $\beta = 108.357$ (2)°
 $V = 1664.5$ (3) Å³
 $Z = 4$

$F(000) = 760$
 $D_x = 1.454$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 2862 reflections
 $\theta = 2.1$ – 30.3 °
 $\mu = 0.11$ mm⁻¹
 $T = 293$ K
 Block, colourless
 $0.21 \times 0.19 \times 0.18$ mm

Data collection

Bruker SMART APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω and φ scans

Absorption correction: multi-scan
 (SADABS; Bruker, 2008)
 $T_{\min} = 0.978$, $T_{\max} = 0.981$
 28712 measured reflections
 4571 independent reflections
 2862 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$
 $\theta_{\text{max}} = 30.3^\circ$, $\theta_{\text{min}} = 2.1^\circ$
 $h = -15 \rightarrow 15$

$k = -15 \rightarrow 16$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.151$
 $S = 1.03$
 4571 reflections
 244 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.7266P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.36 \text{ e } \text{\AA}^{-3}$

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 -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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C1 | 0.39731 (17) | 0.29273 (15) | 0.11931 (14) | 0.0394 (4) |
| C2 | 0.3614 (2) | 0.18037 (17) | 0.11445 (17) | 0.0525 (5) |
| H2 | 0.2802 | 0.1594 | 0.1181 | 0.063* |
| C3 | 0.4481 (2) | 0.09986 (18) | 0.10411 (18) | 0.0569 (6) |
| H3 | 0.4256 | 0.0235 | 0.1014 | 0.068* |
| C4 | 0.5677 (2) | 0.13063 (17) | 0.09769 (17) | 0.0538 (5) |
| H4 | 0.6246 | 0.0751 | 0.0895 | 0.065* |
| C5 | 0.60377 (19) | 0.24263 (17) | 0.10328 (15) | 0.0455 (4) |
| H5 | 0.6849 | 0.2629 | 0.0990 | 0.055* |
| C6 | 0.51827 (16) | 0.32599 (14) | 0.11533 (13) | 0.0358 (4) |
| C7 | 0.54773 (16) | 0.44453 (14) | 0.13040 (13) | 0.0342 (4) |
| C8 | 0.70721 (17) | 0.58447 (15) | 0.13873 (13) | 0.0375 (4) |
| C9 | 0.9063 (2) | 0.5072 (2) | 0.11560 (18) | 0.0560 (5) |
| H9A | 0.9850 | 0.5386 | 0.1089 | 0.084* |
| H9B | 0.8626 | 0.4640 | 0.0553 | 0.084* |
| H9C | 0.9270 | 0.4587 | 0.1747 | 0.084* |
| C10 | 0.62572 (17) | 0.66705 (14) | 0.15559 (13) | 0.0365 (4) |
| C11 | 0.50320 (16) | 0.64000 (14) | 0.18012 (13) | 0.0359 (4) |
| H11 | 0.4340 | 0.6918 | 0.1422 | 0.043* |
| C12 | 0.46442 (16) | 0.52058 (15) | 0.14680 (13) | 0.0350 (4) |
| C13 | 0.33590 (17) | 0.48409 (16) | 0.14373 (14) | 0.0422 (4) |
| C14 | 0.52303 (15) | 0.65125 (14) | 0.29503 (13) | 0.0331 (4) |

| | | | | |
|------|--------------|--------------|--------------|------------|
| C15 | 0.60210 (17) | 0.57475 (15) | 0.36318 (14) | 0.0400 (4) |
| H15 | 0.6410 | 0.5159 | 0.3384 | 0.048* |
| C16 | 0.62389 (18) | 0.58497 (16) | 0.46753 (15) | 0.0445 (4) |
| H16 | 0.6773 | 0.5328 | 0.5120 | 0.053* |
| C17 | 0.56764 (17) | 0.67147 (16) | 0.50698 (14) | 0.0414 (4) |
| C18 | 0.5931 (2) | 0.6825 (2) | 0.62041 (16) | 0.0597 (6) |
| H18A | 0.6515 | 0.6236 | 0.6554 | 0.090* |
| H18B | 0.5127 | 0.6760 | 0.6354 | 0.090* |
| H18C | 0.6316 | 0.7552 | 0.6431 | 0.090* |
| C19 | 0.48681 (18) | 0.74572 (16) | 0.43833 (15) | 0.0449 (4) |
| H19 | 0.4466 | 0.8037 | 0.4630 | 0.054* |
| C20 | 0.46409 (17) | 0.73612 (15) | 0.33388 (14) | 0.0405 (4) |
| H20 | 0.4087 | 0.7872 | 0.2894 | 0.049* |
| N1 | 0.82208 (16) | 0.59828 (15) | 0.12756 (13) | 0.0480 (4) |
| H1 | 0.8497 | 0.6666 | 0.1273 | 0.058* |
| N2 | 0.66129 (17) | 0.78014 (13) | 0.15862 (12) | 0.0455 (4) |
| O1 | 0.67089 (11) | 0.47326 (10) | 0.13148 (10) | 0.0405 (3) |
| O2 | 0.30759 (12) | 0.37082 (11) | 0.12920 (11) | 0.0469 (3) |
| O3 | 0.25155 (13) | 0.54490 (13) | 0.15308 (14) | 0.0624 (4) |
| O4 | 0.58643 (16) | 0.85218 (12) | 0.17480 (12) | 0.0591 (4) |
| O5 | 0.76697 (15) | 0.80934 (12) | 0.14575 (12) | 0.0598 (4) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0360 (9) | 0.0392 (9) | 0.0391 (9) | 0.0006 (7) | 0.0063 (7) | -0.0084 (7) |
| C2 | 0.0418 (10) | 0.0437 (11) | 0.0668 (14) | -0.0075 (8) | 0.0098 (9) | -0.0126 (10) |
| C3 | 0.0534 (12) | 0.0367 (10) | 0.0722 (15) | -0.0048 (9) | 0.0077 (10) | -0.0144 (10) |
| C4 | 0.0517 (12) | 0.0410 (11) | 0.0642 (13) | 0.0066 (9) | 0.0117 (10) | -0.0147 (9) |
| C5 | 0.0421 (10) | 0.0443 (11) | 0.0492 (11) | 0.0049 (8) | 0.0129 (8) | -0.0096 (8) |
| C6 | 0.0367 (9) | 0.0356 (9) | 0.0325 (9) | 0.0017 (7) | 0.0073 (7) | -0.0051 (7) |
| C7 | 0.0324 (8) | 0.0367 (9) | 0.0324 (8) | 0.0011 (7) | 0.0089 (7) | -0.0012 (7) |
| C8 | 0.0418 (9) | 0.0384 (9) | 0.0338 (9) | 0.0000 (7) | 0.0140 (7) | 0.0048 (7) |
| C9 | 0.0470 (11) | 0.0626 (13) | 0.0669 (14) | 0.0091 (10) | 0.0301 (10) | 0.0089 (11) |
| C10 | 0.0412 (9) | 0.0320 (8) | 0.0374 (9) | 0.0011 (7) | 0.0139 (7) | 0.0033 (7) |
| C11 | 0.0342 (8) | 0.0303 (8) | 0.0417 (9) | 0.0059 (7) | 0.0097 (7) | 0.0013 (7) |
| C12 | 0.0332 (8) | 0.0357 (9) | 0.0342 (9) | 0.0034 (7) | 0.0079 (7) | -0.0016 (7) |
| C13 | 0.0346 (9) | 0.0415 (10) | 0.0470 (10) | 0.0018 (7) | 0.0081 (8) | -0.0061 (8) |
| C14 | 0.0298 (8) | 0.0308 (8) | 0.0402 (9) | -0.0002 (6) | 0.0131 (7) | -0.0001 (7) |
| C15 | 0.0393 (9) | 0.0360 (9) | 0.0457 (10) | 0.0082 (7) | 0.0149 (8) | -0.0012 (7) |
| C16 | 0.0436 (10) | 0.0439 (10) | 0.0438 (10) | 0.0067 (8) | 0.0107 (8) | 0.0084 (8) |
| C17 | 0.0385 (9) | 0.0442 (10) | 0.0441 (10) | -0.0077 (8) | 0.0165 (8) | -0.0041 (8) |
| C18 | 0.0679 (14) | 0.0687 (15) | 0.0450 (12) | -0.0062 (11) | 0.0212 (10) | -0.0041 (10) |
| C19 | 0.0460 (10) | 0.0415 (10) | 0.0517 (11) | 0.0046 (8) | 0.0220 (9) | -0.0075 (8) |
| C20 | 0.0385 (9) | 0.0366 (9) | 0.0466 (10) | 0.0071 (7) | 0.0138 (8) | -0.0001 (8) |
| N1 | 0.0472 (9) | 0.0462 (9) | 0.0595 (10) | -0.0008 (7) | 0.0295 (8) | 0.0051 (8) |
| N2 | 0.0586 (10) | 0.0366 (8) | 0.0424 (9) | -0.0015 (7) | 0.0176 (8) | 0.0045 (7) |
| O1 | 0.0364 (6) | 0.0360 (7) | 0.0525 (8) | 0.0021 (5) | 0.0189 (6) | -0.0005 (6) |

| | | | | | | |
|----|-------------|------------|-------------|-------------|------------|-------------|
| O2 | 0.0344 (6) | 0.0419 (7) | 0.0626 (9) | -0.0018 (5) | 0.0129 (6) | -0.0120 (6) |
| O3 | 0.0376 (7) | 0.0541 (9) | 0.0952 (12) | 0.0077 (6) | 0.0204 (8) | -0.0142 (8) |
| O4 | 0.0780 (10) | 0.0349 (7) | 0.0708 (10) | 0.0064 (7) | 0.0328 (8) | 0.0035 (7) |
| O5 | 0.0682 (10) | 0.0461 (8) | 0.0738 (11) | -0.0111 (7) | 0.0348 (8) | 0.0063 (7) |

Geometric parameters (Å, °)

| | | | |
|----------|-------------|-------------|-------------|
| C1—O2 | 1.376 (2) | C11—C12 | 1.499 (2) |
| C1—C2 | 1.377 (3) | C11—C14 | 1.529 (2) |
| C1—C6 | 1.385 (2) | C11—H11 | 0.9800 |
| C2—C3 | 1.374 (3) | C12—C13 | 1.445 (2) |
| C2—H2 | 0.9300 | C13—O3 | 1.200 (2) |
| C3—C4 | 1.374 (3) | C13—O2 | 1.371 (2) |
| C3—H3 | 0.9300 | C14—C20 | 1.381 (2) |
| C4—C5 | 1.373 (3) | C14—C15 | 1.385 (2) |
| C4—H4 | 0.9300 | C15—C16 | 1.382 (3) |
| C5—C6 | 1.396 (2) | C15—H15 | 0.9300 |
| C5—H5 | 0.9300 | C16—C17 | 1.383 (3) |
| C6—C7 | 1.434 (2) | C16—H16 | 0.9300 |
| C7—C12 | 1.341 (2) | C17—C19 | 1.379 (3) |
| C7—O1 | 1.372 (2) | C17—C18 | 1.499 (3) |
| C8—N1 | 1.311 (2) | C18—H18A | 0.9600 |
| C8—O1 | 1.364 (2) | C18—H18B | 0.9600 |
| C8—C10 | 1.382 (2) | C18—H18C | 0.9600 |
| C9—N1 | 1.452 (3) | C19—C20 | 1.381 (3) |
| C9—H9A | 0.9600 | C19—H19 | 0.9300 |
| C9—H9B | 0.9600 | C20—H20 | 0.9300 |
| C9—H9C | 0.9600 | N1—H1 | 0.8600 |
| C10—N2 | 1.385 (2) | N2—O4 | 1.242 (2) |
| C10—C11 | 1.503 (2) | N2—O5 | 1.260 (2) |
| O2—C1—C2 | 116.81 (17) | C7—C12—C13 | 119.29 (16) |
| O2—C1—C6 | 121.38 (16) | C7—C12—C11 | 122.74 (15) |
| C2—C1—C6 | 121.81 (17) | C13—C12—C11 | 117.61 (15) |
| C3—C2—C1 | 118.54 (19) | O3—C13—O2 | 117.06 (17) |
| C3—C2—H2 | 120.7 | O3—C13—C12 | 125.32 (18) |
| C1—C2—H2 | 120.7 | O2—C13—C12 | 117.62 (15) |
| C2—C3—C4 | 120.87 (19) | C20—C14—C15 | 118.29 (16) |
| C2—C3—H3 | 119.6 | C20—C14—C11 | 121.95 (15) |
| C4—C3—H3 | 119.6 | C15—C14—C11 | 119.76 (15) |
| C5—C4—C3 | 120.53 (19) | C16—C15—C14 | 120.72 (16) |
| C5—C4—H4 | 119.7 | C16—C15—H15 | 119.6 |
| C3—C4—H4 | 119.7 | C14—C15—H15 | 119.6 |
| C4—C5—C6 | 119.76 (19) | C15—C16—C17 | 121.18 (17) |
| C4—C5—H5 | 120.1 | C15—C16—H16 | 119.4 |
| C6—C5—H5 | 120.1 | C17—C16—H16 | 119.4 |
| C1—C6—C5 | 118.46 (17) | C19—C17—C16 | 117.64 (17) |
| C1—C6—C7 | 116.14 (15) | C19—C17—C18 | 121.61 (18) |

| | | | |
|----------------|--------------|-----------------|--------------|
| C5—C6—C7 | 125.29 (17) | C16—C17—C18 | 120.75 (19) |
| C12—C7—O1 | 122.46 (16) | C17—C18—H18A | 109.5 |
| C12—C7—C6 | 123.04 (16) | C17—C18—H18B | 109.5 |
| O1—C7—C6 | 114.43 (14) | H18A—C18—H18B | 109.5 |
| N1—C8—O1 | 111.93 (16) | C17—C18—H18C | 109.5 |
| N1—C8—C10 | 127.74 (17) | H18A—C18—H18C | 109.5 |
| O1—C8—C10 | 120.33 (15) | H18B—C18—H18C | 109.5 |
| N1—C9—H9A | 109.5 | C17—C19—C20 | 121.64 (17) |
| N1—C9—H9B | 109.5 | C17—C19—H19 | 119.2 |
| H9A—C9—H9B | 109.5 | C20—C19—H19 | 119.2 |
| N1—C9—H9C | 109.5 | C19—C20—C14 | 120.49 (17) |
| H9A—C9—H9C | 109.5 | C19—C20—H20 | 119.8 |
| H9B—C9—H9C | 109.5 | C14—C20—H20 | 119.8 |
| C8—C10—N2 | 119.80 (16) | C8—N1—C9 | 125.09 (17) |
| C8—C10—C11 | 122.95 (15) | C8—N1—H1 | 117.5 |
| N2—C10—C11 | 117.04 (15) | C9—N1—H1 | 117.5 |
| C12—C11—C10 | 108.26 (14) | O4—N2—O5 | 120.76 (16) |
| C12—C11—C14 | 109.33 (14) | O4—N2—C10 | 118.21 (16) |
| C10—C11—C14 | 111.45 (14) | O5—N2—C10 | 121.03 (16) |
| C12—C11—H11 | 109.3 | C8—O1—C7 | 119.69 (13) |
| C10—C11—H11 | 109.3 | C13—O2—C1 | 122.25 (14) |
| C14—C11—H11 | 109.3 | | |
| O2—C1—C2—C3 | 179.72 (18) | C11—C12—C13—O3 | 9.9 (3) |
| C6—C1—C2—C3 | -0.8 (3) | C7—C12—C13—O2 | 3.3 (3) |
| C1—C2—C3—C4 | -0.6 (3) | C11—C12—C13—O2 | -169.93 (15) |
| C2—C3—C4—C5 | 1.1 (3) | C12—C11—C14—C20 | -129.00 (17) |
| C3—C4—C5—C6 | -0.2 (3) | C10—C11—C14—C20 | 111.35 (18) |
| O2—C1—C6—C5 | -178.80 (16) | C12—C11—C14—C15 | 51.0 (2) |
| C2—C1—C6—C5 | 1.7 (3) | C10—C11—C14—C15 | -68.7 (2) |
| O2—C1—C6—C7 | 4.9 (2) | C20—C14—C15—C16 | -1.6 (3) |
| C2—C1—C6—C7 | -174.59 (18) | C11—C14—C15—C16 | 178.42 (16) |
| C4—C5—C6—C1 | -1.2 (3) | C14—C15—C16—C17 | 0.0 (3) |
| C4—C5—C6—C7 | 174.73 (18) | C15—C16—C17—C19 | 1.4 (3) |
| C1—C6—C7—C12 | -0.5 (3) | C15—C16—C17—C18 | -179.30 (18) |
| C5—C6—C7—C12 | -176.51 (18) | C16—C17—C19—C20 | -1.2 (3) |
| C1—C6—C7—O1 | 176.64 (15) | C18—C17—C19—C20 | 179.47 (18) |
| C5—C6—C7—O1 | 0.6 (3) | C17—C19—C20—C14 | -0.4 (3) |
| N1—C8—C10—N2 | 2.4 (3) | C15—C14—C20—C19 | 1.8 (3) |
| O1—C8—C10—N2 | -177.36 (15) | C11—C14—C20—C19 | -178.26 (16) |
| N1—C8—C10—C11 | -172.24 (17) | O1—C8—N1—C9 | -3.8 (3) |
| O1—C8—C10—C11 | 8.0 (3) | C10—C8—N1—C9 | 176.42 (19) |
| C8—C10—C11—C12 | -19.5 (2) | C8—C10—N2—O4 | -179.39 (17) |
| N2—C10—C11—C12 | 165.70 (15) | C11—C10—N2—O4 | -4.5 (2) |
| C8—C10—C11—C14 | 100.73 (19) | C8—C10—N2—O5 | 0.3 (3) |
| N2—C10—C11—C14 | -74.03 (19) | C11—C10—N2—O5 | 175.25 (16) |
| O1—C7—C12—C13 | 179.56 (15) | N1—C8—O1—C7 | -173.09 (15) |
| C6—C7—C12—C13 | -3.6 (3) | C10—C8—O1—C7 | 6.7 (2) |

| | | | |
|-----------------|--------------|---------------|--------------|
| O1—C7—C12—C11 | -7.6 (3) | C12—C7—O1—C8 | -7.0 (2) |
| C6—C7—C12—C11 | 169.29 (16) | C6—C7—O1—C8 | 175.86 (15) |
| C10—C11—C12—C7 | 19.3 (2) | O3—C13—O2—C1 | -178.85 (17) |
| C14—C11—C12—C7 | -102.29 (19) | C12—C13—O2—C1 | 1.0 (3) |
| C10—C11—C12—C13 | -167.72 (15) | C2—C1—O2—C13 | 174.23 (17) |
| C14—C11—C12—C13 | 70.70 (19) | C6—C1—O2—C13 | -5.2 (3) |
| C7—C12—C13—O3 | -176.84 (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> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1...O5 | 0.86 | 1.96 | 2.590 (2) | 129 |
| C9—H9C...O4 ⁱ | 0.96 | 2.46 | 3.389 (3) | 163 |

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