Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

# Crystal structure of 2-methoxy-1-nitronaphthalene

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Received 2 August 2015; accepted 28 August 2015

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

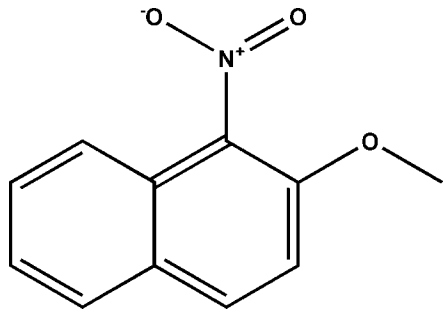
The asymmetric unit of the title compound, $C_{11}H_9NO_3$, contains two molecules, *A* and *B*. In molecule *A*, the dihedral angle between the planes of the naphthalene ring system (r.m.s. deviation = 0.003 Å) and the nitro group is 89.9 (2)°, and the C atom of the methoxy group deviates from the naphthyl plane by 0.022 (2) Å. Equivalent data for molecule *B* are 0.008 Å, 65.9 (2)° and −0.198 (2) Å, respectively. In the crystal, molecules are linked by weak C—H···O interactions, forming [100] chains of alternating *A* and *B* molecules. Weak aromatic π–π stacking contacts, with a range of centroid–centroid distances from 3.5863 (9) to 3.8048 (9) Å, are also observed.

**Keywords:** crystal structure; naphthalene derivative; weak C—H···O interactions; π–π stacking.

**CCDC reference**: 1421062

## 1. Related literature

For biological activities of naphthalene derivatives, see: Wright *et al.* (2000); Rokade & Sayyed (2009); Upadhayaya *et al.* (2010). For the title compound as an intermediate in the synthesis of antipyretic drugs, see: Stoylkova *et al.* (2000); Govindarajana *et al.* (2011); Kirumakki *et al.* (2004); Yadav *et al.* (1998). For a related structure, see: Wannalerse *et al.* (2013).



## 2. Experimental

### 2.1. Crystal data

$C_{11}H_9NO_3$
$M_r$ = 203.19
Triclinic, $P\overline{1}$
$a$ = 9.1291 (4) Å
$b$ = 10.2456 (4) Å
$c$ = 10.5215 (4) Å
$\alpha$ = 86.390 (2)°
$\beta$ = 82.964 (2)°
$\gamma$ = 85.801 (2)°
$V$ = 972.63 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
0.39 × 0.32 × 0.24 mm

### 2.2. Data collection

Bruker X8 APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.676, $T_{max}$ = 0.746
34901 measured reflections
5450 independent reflections
3446 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.141
$S$ = 1.04
5450 reflections
272 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4···O5$^{i}$ | 0.93 | 2.57 | 3.409 (2) | 150 |
| C11—H11$A$···O5$^{ii}$ | 0.96 | 2.60 | 3.462 (3) | 150 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

## Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements and the University Sultan Moulay Slimane, Beni-Mellal, for financial support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7477).

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# supporting information

*Acta Cryst.* (2015). E**71**, o701–o702 [doi:10.1107/S2056989015016114]

## Crystal structure of 2-methoxy-1-nitronaphthalene

**Hasna Yassine, Mostafa Khouili, Lahcen El Ammari, Mohamed Saadi and El Mostafa Ketatni**

### S1. Comment

Naphthalene derivatives have been extensively employed in many fields, for example, as a colorant, explosive, disinfectant, insecticide, auxin plant hormone and play a role in the chemical defence against biological (Wright *et al.*, 2000) and have diverse and interesting antibiotic properties (Rokade & Sayyed, 2009; Upadhayaya *et al.* 2010). 2-Methoxynaphthalene is an important intermediate used in the production of naproxen. It is widely used a non-steroidal anti-inflammatory, analgesic and antipyretic drug (Stoylkova *et al.*, 2000, Govindarajana *et al.*, 2011, Kirumakki *et al.*, 2004; Yadav *et al.*, 1998). Nitration of 1-methoxynaphthalene with bismuth nitrate in $CH_2Cl_2$ gives a compound (I) and describes its structure here.

The asymmetric unit of the title compound consists of two crystallographically independent molecules of nearly similar geometry as shown in Fig. 1. Bond lengths and angles of the title compound are comparable with that found in the similar structure (Wannalerse *et al.*, 2013). In the first (O1O2O3N1C1–C11) and second (O4O5O6N2C12–C22) molecules, the dihedral angles between the nitro group and the attached naphthalene system are 89.9 (2)° and 65.9 (2)°, respectively. The two naphthalene rings belonging to the both molecules form a dihedral angle of 72.02 (5)°.

In the crystal, the molecules are linked together by weak C—H···O interactions. Moreover, the *π*–*π* contacts between the naphthalene rings, may further consolidate the structure, with range of centroid– centroid distances = 3.5863 (9)—3.8048 (9) Å.

### S2. Experimental

2-Methoxynaphtalene (500 mg, 3.164 mmol) and silica gel (500 mg) was added to a suspension of bismuth nitrate pentahydrate (1.2 eqv.) in $CH_2Cl_2$ (20 ml). The mixture was refluxed for 6 h. After cooling to room temperature, the reaction mixture was filtered and watched with $CH_2Cl_2$, the filtrate obtained was concentrated, and the resulting residue was purified by column chromatography using EtOAc-Hexane (1:9 *v*/*v*). The title compound was recrystallized from the solvent mixture ethyl acetate/hexane to yield orange block crystals (yield: 74%).

### S3. Refinement

All H atoms could be located in a difference Fourier map. However, they were placed in calculated positions with C—H = 0.93–0.96 Å, and refined as riding on their parent atoms with $U_{iso}$(H) = 1.2 $U_{eq}$ for aromatic and $U_{iso}$(H) = 1.5 $U_{eq}$ (C) for methyl. Two outlier reflections, (-7 3 0) and (-1 - 3 2), were omitted in the last cycles of refinement.

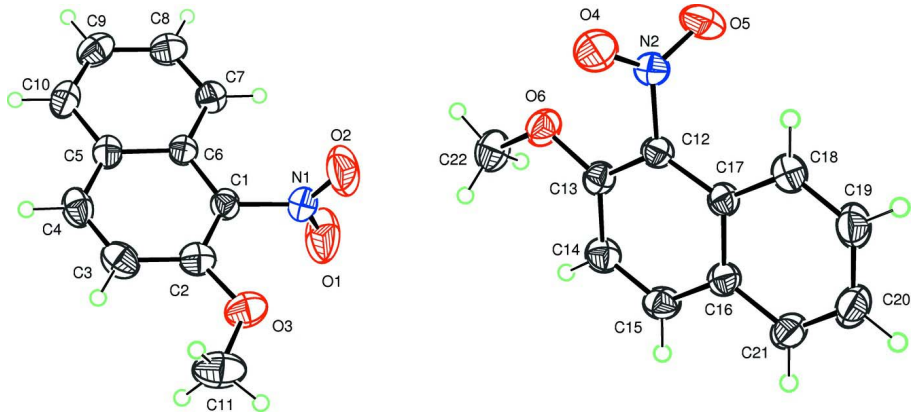


**Figure 1**

A view of the molecule of the title compound, showing displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small circles.

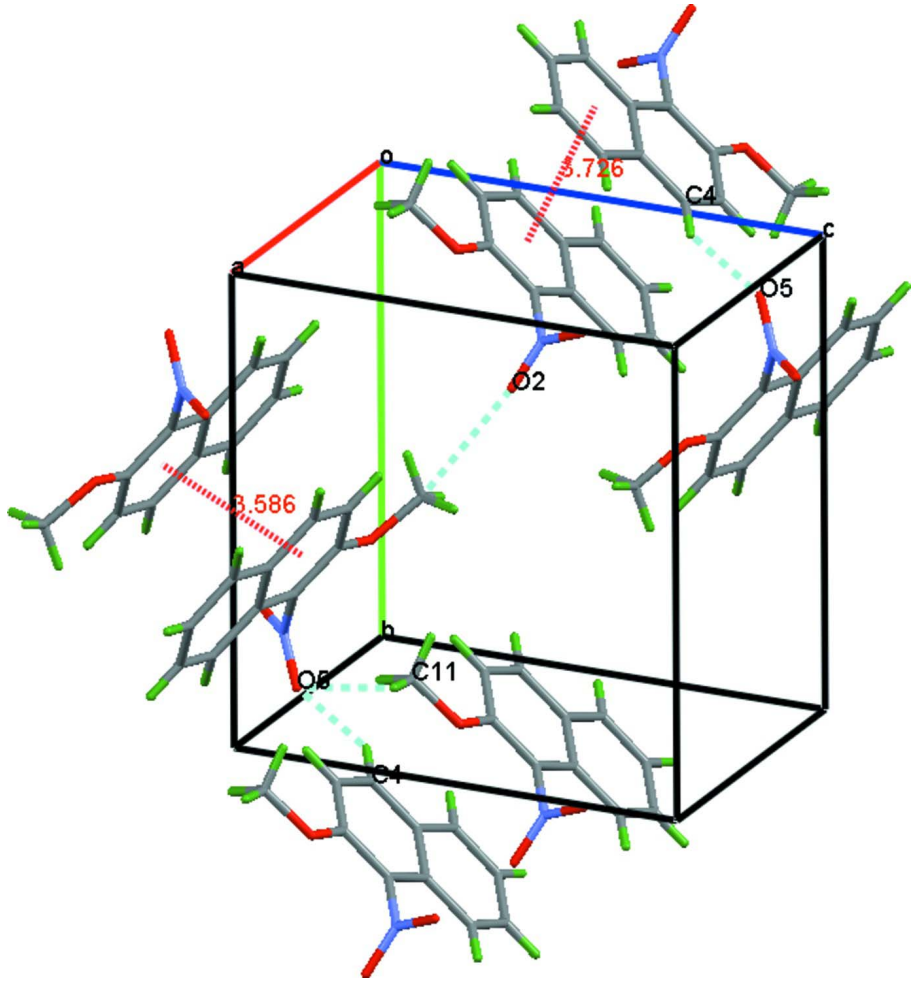


**Figure 2**

Partial crystal packing for the title compound showing molecules linked by hydrogen bonds as blue dashed lines and $\pi$–$\pi$ contacts between the naphthalene rings (red dashed lines).

**2-Methoxy-1-nitronaphthalene**

*Crystal data*

$C_{11}H_9NO_3$
$M_r = 203.19$
Triclinic, $P\overline{1}$
$a = 9.1291$ (4) Å
$b = 10.2456$ (4) Å
$c = 10.5215$ (4) Å
$\alpha = 86.390$ (2)°
$\beta = 82.964$ (2)°
$\gamma = 85.801$ (2)°
$V = 972.63$ (7) Å$^3$
$Z = 4$
$F(000) = 424$
$D_x = 1.388$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3506 reflections
$\theta = 1.7$–30.0°
$\mu = 0.10$ mm$^{-1}$
$T = 296$ K
Block, orange
0.39 × 0.32 × 0.24 mm

*Data collection*

Bruker X8 APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.676$, $T_{max} = 0.746$
34901 measured reflections
5450 independent reflections
3446 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 29.6°$, $\theta_{min} = 2.0°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 14$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.141$
$S = 1.04$
5450 reflections
272 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.2462P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å$^{-3}$
$\Delta\rho_{min} = -0.15$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (2)

*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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.82138 (17) | 0.83303 (15) | 0.59386 (15) | 0.0445 (3) |
| C2 | 0.76837 (19) | 0.92960 (16) | 0.67524 (16) | 0.0512 (4) |
| C3 | 0.8714 (2) | 0.99956 (17) | 0.72711 (17) | 0.0582 (4) |
| H3 | 0.8389 | 1.0650 | 0.7832 | 0.070* |
| C4 | 1.0189 (2) | 0.97177 (17) | 0.69542 (16) | 0.0562 (4) |
| H4 | 1.0854 | 1.0201 | 0.7298 | 0.067* |
| C5 | 1.07461 (18) | 0.87266 (15) | 0.61254 (15) | 0.0464 (4) |
| C6 | 0.97310 (17) | 0.79982 (14) | 0.55904 (14) | 0.0416 (3) |

| | | | | |
|---|---|---|---|---|
| C7 | 1.02802 (19) | 0.70018 (16) | 0.47545 (16) | 0.0515 (4) |
| H7 | 0.9625 | 0.6519 | 0.4398 | 0.062* |
| C8 | 1.1760 (2) | 0.67452 (19) | 0.44694 (18) | 0.0605 (5) |
| H8 | 1.2107 | 0.6085 | 0.3920 | 0.073* |
| C9 | 1.2771 (2) | 0.7456 (2) | 0.49869 (19) | 0.0633 (5) |
| H9 | 1.3782 | 0.7266 | 0.4781 | 0.076* |
| C10 | 1.2280 (2) | 0.84240 (19) | 0.57914 (18) | 0.0580 (5) |
| H10 | 1.2961 | 0.8895 | 0.6128 | 0.070* |
| C11 | 0.5602 (3) | 1.0473 (2) | 0.7849 (2) | 0.0859 (7) |
| H11A | 0.4542 | 1.0518 | 0.7914 | 0.129* |
| H11B | 0.5951 | 1.1311 | 0.7543 | 0.129* |
| H11C | 0.5912 | 1.0242 | 0.8678 | 0.129* |
| C12 | 0.12086 (16) | 0.31177 (13) | 0.90308 (14) | 0.0391 (3) |
| C13 | 0.19472 (16) | 0.40531 (14) | 0.82703 (14) | 0.0411 (3) |
| C14 | 0.11092 (19) | 0.51331 (15) | 0.77661 (15) | 0.0477 (4) |
| H14 | 0.1581 | 0.5779 | 0.7242 | 0.057* |
| C15 | −0.03834 (18) | 0.52299 (15) | 0.80456 (15) | 0.0483 (4) |
| H15 | −0.0915 | 0.5959 | 0.7721 | 0.058* |
| C16 | −0.11572 (17) | 0.42663 (14) | 0.88090 (14) | 0.0424 (3) |
| C17 | −0.03443 (16) | 0.31586 (14) | 0.93202 (14) | 0.0391 (3) |
| C18 | −0.11305 (19) | 0.22006 (16) | 1.00938 (17) | 0.0515 (4) |
| H18 | −0.0618 | 0.1475 | 1.0446 | 0.062* |
| C19 | −0.2632 (2) | 0.23339 (18) | 1.0326 (2) | 0.0612 (5) |
| H19 | −0.3135 | 0.1690 | 1.0827 | 0.073* |
| C20 | −0.34344 (19) | 0.34223 (19) | 0.9822 (2) | 0.0618 (5) |
| H20 | −0.4460 | 0.3497 | 0.9990 | 0.074* |
| C21 | −0.27129 (18) | 0.43676 (17) | 0.90895 (18) | 0.0532 (4) |
| H21 | −0.3251 | 0.5093 | 0.8767 | 0.064* |
| C22 | 0.4201 (2) | 0.47481 (19) | 0.71352 (18) | 0.0615 (5) |
| H22A | 0.5244 | 0.4516 | 0.7073 | 0.092* |
| H22B | 0.4010 | 0.5629 | 0.7403 | 0.092* |
| H22C | 0.3862 | 0.4688 | 0.6314 | 0.092* |
| N1 | 0.20980 (15) | 0.20198 (13) | 0.95773 (14) | 0.0483 (3) |
| N2 | 0.71338 (16) | 0.76148 (15) | 0.53852 (16) | 0.0571 (4) |
| O1 | 0.6734 (2) | 0.8025 (2) | 0.43824 (19) | 0.1118 (7) |
| O2 | 0.6708 (2) | 0.66298 (17) | 0.5941 (2) | 0.1043 (6) |
| O3 | 0.61970 (14) | 0.95078 (14) | 0.69777 (14) | 0.0703 (4) |
| O4 | 0.29054 (17) | 0.22522 (13) | 1.03577 (15) | 0.0785 (4) |
| O5 | 0.19774 (17) | 0.09259 (12) | 0.92409 (16) | 0.0767 (4) |
| O6 | 0.34371 (12) | 0.38730 (11) | 0.80514 (11) | 0.0537 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0446 (8) | 0.0428 (8) | 0.0464 (8) | −0.0077 (6) | −0.0072 (7) | 0.0025 (6) |
| C2 | 0.0539 (10) | 0.0513 (9) | 0.0462 (9) | −0.0005 (7) | −0.0013 (7) | 0.0027 (7) |
| C3 | 0.0772 (13) | 0.0527 (9) | 0.0447 (9) | −0.0034 (9) | −0.0046 (9) | −0.0084 (7) |
| C4 | 0.0708 (12) | 0.0557 (10) | 0.0465 (9) | −0.0177 (8) | −0.0179 (8) | −0.0006 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.0510 (9) | 0.0486 (8) | 0.0410 (8) | −0.0110 (7) | −0.0117 (7) | 0.0069 (7) |
| C6 | 0.0441 (8) | 0.0415 (7) | 0.0393 (8) | −0.0064 (6) | −0.0076 (6) | 0.0052 (6) |
| C7 | 0.0527 (10) | 0.0499 (9) | 0.0526 (10) | −0.0041 (7) | −0.0081 (8) | −0.0046 (7) |
| C8 | 0.0582 (11) | 0.0624 (11) | 0.0580 (11) | 0.0062 (8) | −0.0016 (8) | −0.0027 (8) |
| C9 | 0.0455 (10) | 0.0768 (13) | 0.0640 (12) | 0.0008 (9) | −0.0028 (8) | 0.0118 (10) |
| C10 | 0.0499 (10) | 0.0688 (11) | 0.0578 (11) | −0.0170 (8) | −0.0169 (8) | 0.0122 (9) |
| C11 | 0.0839 (16) | 0.0823 (15) | 0.0800 (15) | 0.0233 (12) | 0.0198 (12) | −0.0064 (12) |
| C12 | 0.0404 (8) | 0.0360 (7) | 0.0417 (8) | −0.0007 (6) | −0.0080 (6) | −0.0039 (6) |
| C13 | 0.0390 (8) | 0.0445 (8) | 0.0403 (8) | −0.0033 (6) | −0.0047 (6) | −0.0046 (6) |
| C14 | 0.0524 (9) | 0.0456 (8) | 0.0442 (8) | −0.0048 (7) | −0.0055 (7) | 0.0049 (6) |
| C15 | 0.0522 (9) | 0.0444 (8) | 0.0483 (9) | 0.0032 (7) | −0.0121 (7) | 0.0036 (7) |
| C16 | 0.0413 (8) | 0.0445 (8) | 0.0431 (8) | −0.0009 (6) | −0.0099 (6) | −0.0089 (6) |
| C17 | 0.0392 (8) | 0.0386 (7) | 0.0409 (8) | −0.0042 (6) | −0.0070 (6) | −0.0069 (6) |
| C18 | 0.0493 (9) | 0.0441 (8) | 0.0611 (10) | −0.0087 (7) | −0.0048 (8) | 0.0001 (7) |
| C19 | 0.0505 (10) | 0.0582 (10) | 0.0746 (12) | −0.0188 (8) | 0.0041 (9) | −0.0067 (9) |
| C20 | 0.0370 (9) | 0.0710 (12) | 0.0785 (13) | −0.0071 (8) | −0.0011 (8) | −0.0203 (10) |
| C21 | 0.0411 (9) | 0.0570 (10) | 0.0636 (11) | 0.0021 (7) | −0.0125 (8) | −0.0125 (8) |
| C22 | 0.0487 (10) | 0.0745 (12) | 0.0592 (11) | −0.0146 (9) | 0.0053 (8) | 0.0020 (9) |
| N1 | 0.0435 (7) | 0.0412 (7) | 0.0595 (8) | −0.0011 (5) | −0.0071 (6) | 0.0016 (6) |
| N2 | 0.0450 (8) | 0.0572 (9) | 0.0709 (10) | −0.0085 (6) | −0.0111 (7) | −0.0039 (7) |
| O1 | 0.1262 (16) | 0.1253 (15) | 0.1010 (13) | −0.0526 (12) | −0.0681 (12) | 0.0190 (11) |
| O2 | 0.1055 (13) | 0.0811 (11) | 0.1351 (16) | −0.0523 (10) | −0.0365 (11) | 0.0232 (10) |
| O3 | 0.0542 (8) | 0.0768 (9) | 0.0753 (9) | 0.0099 (6) | 0.0049 (6) | −0.0104 (7) |
| O4 | 0.0826 (10) | 0.0663 (8) | 0.0944 (11) | −0.0031 (7) | −0.0500 (9) | 0.0107 (7) |
| O5 | 0.0839 (10) | 0.0403 (7) | 0.1082 (12) | 0.0091 (6) | −0.0230 (8) | −0.0139 (7) |
| O6 | 0.0394 (6) | 0.0609 (7) | 0.0585 (7) | −0.0050 (5) | −0.0002 (5) | 0.0050 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.372 (2) | C12—N1 | 1.4678 (19) |
| C1—C6 | 1.411 (2) | C13—O6 | 1.3524 (18) |
| C1—N2 | 1.466 (2) | C13—C14 | 1.412 (2) |
| C2—O3 | 1.353 (2) | C14—C15 | 1.356 (2) |
| C2—C3 | 1.404 (3) | C14—H14 | 0.9300 |
| C3—C4 | 1.360 (3) | C15—C16 | 1.408 (2) |
| C3—H3 | 0.9300 | C15—H15 | 0.9300 |
| C4—C5 | 1.406 (2) | C16—C21 | 1.412 (2) |
| C4—H4 | 0.9300 | C16—C17 | 1.423 (2) |
| C5—C10 | 1.418 (2) | C17—C18 | 1.414 (2) |
| C5—C6 | 1.419 (2) | C18—C19 | 1.361 (2) |
| C6—C7 | 1.413 (2) | C18—H18 | 0.9300 |
| C7—C8 | 1.358 (2) | C19—C20 | 1.400 (3) |
| C7—H7 | 0.9300 | C19—H19 | 0.9300 |
| C8—C9 | 1.396 (3) | C20—C21 | 1.357 (3) |
| C8—H8 | 0.9300 | C20—H20 | 0.9300 |
| C9—C10 | 1.360 (3) | C21—H21 | 0.9300 |
| C9—H9 | 0.9300 | C22—O6 | 1.427 (2) |
| C10—H10 | 0.9300 | C22—H22A | 0.9600 |

| | | | |
|---|---|---|---|
| C11—O3 | 1.424 (2) | C22—H22B | 0.9600 |
| C11—H11A | 0.9600 | C22—H22C | 0.9600 |
| C11—H11B | 0.9600 | N1—O5 | 1.2129 (17) |
| C11—H11C | 0.9600 | N1—O4 | 1.2136 (18) |
| C12—C13 | 1.371 (2) | N2—O1 | 1.200 (2) |
| C12—C17 | 1.411 (2) | N2—O2 | 1.200 (2) |
| | | | |
| C2—C1—C6 | 124.01 (15) | O6—C13—C14 | 124.40 (14) |
| C2—C1—N2 | 117.77 (15) | C12—C13—C14 | 118.16 (14) |
| C6—C1—N2 | 118.21 (14) | C15—C14—C13 | 120.05 (14) |
| O3—C2—C1 | 116.87 (16) | C15—C14—H14 | 120.0 |
| O3—C2—C3 | 125.16 (16) | C13—C14—H14 | 120.0 |
| C1—C2—C3 | 117.96 (16) | C14—C15—C16 | 122.34 (14) |
| C4—C3—C2 | 120.12 (16) | C14—C15—H15 | 118.8 |
| C4—C3—H3 | 119.9 | C16—C15—H15 | 118.8 |
| C2—C3—H3 | 119.9 | C15—C16—C21 | 122.11 (14) |
| C3—C4—C5 | 122.44 (16) | C15—C16—C17 | 118.92 (14) |
| C3—C4—H4 | 118.8 | C21—C16—C17 | 118.97 (14) |
| C5—C4—H4 | 118.8 | C12—C17—C18 | 124.80 (14) |
| C4—C5—C10 | 123.00 (16) | C12—C17—C16 | 116.68 (13) |
| C4—C5—C6 | 118.74 (15) | C18—C17—C16 | 118.51 (14) |
| C10—C5—C6 | 118.27 (16) | C19—C18—C17 | 120.38 (16) |
| C1—C6—C7 | 124.13 (14) | C19—C18—H18 | 119.8 |
| C1—C6—C5 | 116.72 (14) | C17—C18—H18 | 119.8 |
| C7—C6—C5 | 119.15 (15) | C18—C19—C20 | 121.21 (17) |
| C8—C7—C6 | 120.33 (16) | C18—C19—H19 | 119.4 |
| C8—C7—H7 | 119.8 | C20—C19—H19 | 119.4 |
| C6—C7—H7 | 119.8 | C21—C20—C19 | 119.91 (16) |
| C7—C8—C9 | 121.14 (18) | C21—C20—H20 | 120.0 |
| C7—C8—H8 | 119.4 | C19—C20—H20 | 120.0 |
| C9—C8—H8 | 119.4 | C20—C21—C16 | 121.01 (16) |
| C10—C9—C8 | 120.03 (18) | C20—C21—H21 | 119.5 |
| C10—C9—H9 | 120.0 | C16—C21—H21 | 119.5 |
| C8—C9—H9 | 120.0 | O6—C22—H22A | 109.5 |
| C9—C10—C5 | 121.09 (17) | O6—C22—H22B | 109.5 |
| C9—C10—H10 | 119.5 | H22A—C22—H22B | 109.5 |
| C5—C10—H10 | 119.5 | O6—C22—H22C | 109.5 |
| O3—C11—H11A | 109.5 | H22A—C22—H22C | 109.5 |
| O3—C11—H11B | 109.5 | H22B—C22—H22C | 109.5 |
| H11A—C11—H11B | 109.5 | O5—N1—O4 | 123.26 (14) |
| O3—C11—H11C | 109.5 | O5—N1—C12 | 118.58 (14) |
| H11A—C11—H11C | 109.5 | O4—N1—C12 | 118.15 (13) |
| H11B—C11—H11C | 109.5 | O1—N2—O2 | 122.74 (17) |
| C13—C12—C17 | 123.81 (13) | O1—N2—C1 | 118.59 (15) |
| C13—C12—N1 | 117.52 (13) | O2—N2—C1 | 118.65 (16) |
| C17—C12—N1 | 118.66 (13) | C2—O3—C11 | 118.58 (17) |
| O6—C13—C12 | 117.43 (13) | C13—O6—C22 | 117.88 (13) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···O5$^{i}$ | 0.93 | 2.57 | 3.409 (2) | 150 |
| C11—H11*A*···O5$^{ii}$ | 0.96 | 2.60 | 3.462 (3) | 150 |

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