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# 1-(4-Benzyloxy-5-methoxy-2-nitrophenyl)ethanone

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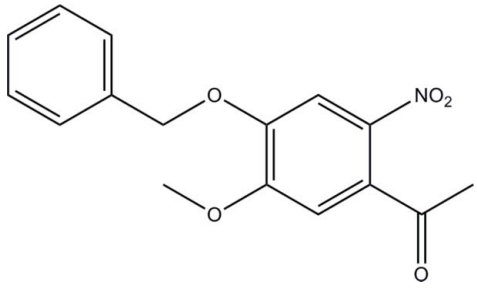
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.071; $wR$ factor = 0.223; data-to-parameter ratio = 13.2.

In the molecule of the title compound, $C_{16}H_{15}NO_5$, the aromatic rings are oriented at a dihedral angle of 74.89 (3)°. Intramolecular C—H···O interactions result in the formation of a seven-membered ring. In the crystal structure, weak intermolecular C—H···O interactions link the molecules into chains along the $b$ axis.

## Related literature

The title compound is an important pharmaceutical intermediate. For general background, see: Mizuta *et al.* (2002). For bond-length data, see: Allen *et al.* (1987).




## Experimental

### Crystal data

$C_{16}H_{15}NO_5$
$M_r$ = 301.29
Orthorhombic, *Pbca*
$a$ = 13.390 (3) Å
$b$ = 10.465 (2) Å
$c$ = 20.768 (4) Å
$V$ = 2910.1 (10) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 294 K
0.20 × 0.10 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.980, $T_{max}$ = 0.990
2634 measured reflections
2634 independent reflections
1446 reflections with $I > 2\sigma(I)$
3 standard reflections
frequency: 120 min
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.223
$S$ = 1.06
2634 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4$A$···O3$^{i}$ | 0.93 | 2.58 | 3.466 (5) | 158 |
| C16—H16$C$···O4 | 0.96 | 2.35 | 2.899 (5) | 115 |

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2654).

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# supporting information

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## 1-(4-Benzyloxy-5-methoxy-2-nitrophenyl)ethanone

**Lei Gao, Li-hua Guo, Rui Liu, Dan-hua Que and Hong-fei Ma**

### S1. Comment

The title compound contains nitro, acetyl and methoxy groups, which can react with different groups to prepare various functional organic compounds as a fine organic intermediate. We report herein its crystal structure.

In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C1-C6) and B (C8-C13) are, of course, planar and they are oriented at a dihedral angle of 74.89 (3)°. Intramolecular C-H···O interaction (Table 1) results in the formation of a seven-membered ring C (O4/N/C10/C11/C15/C16/H16C) having twisted conformation.

In the crystal structure, weak intermolecular C-H···O interactions (Table 1) link the molecules into chains along the b axis (Fig. 2), in which they may be effective in the stabilization of the structure.

### S2. Experimental

For the preparation of the title compound, 1-(4-benzyloxy-5-methoxy-2-nitro- phenyl)ethanone (20.0 g, 66.4 mmol) were dissolved in DMF (50 ml). Then, the solution was poured into ice water (100 ml). The crystalline product was isolated by filtration, washed with water (600 ml). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

### S3. Refinement

H atoms were positioned geometrically, with C-H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

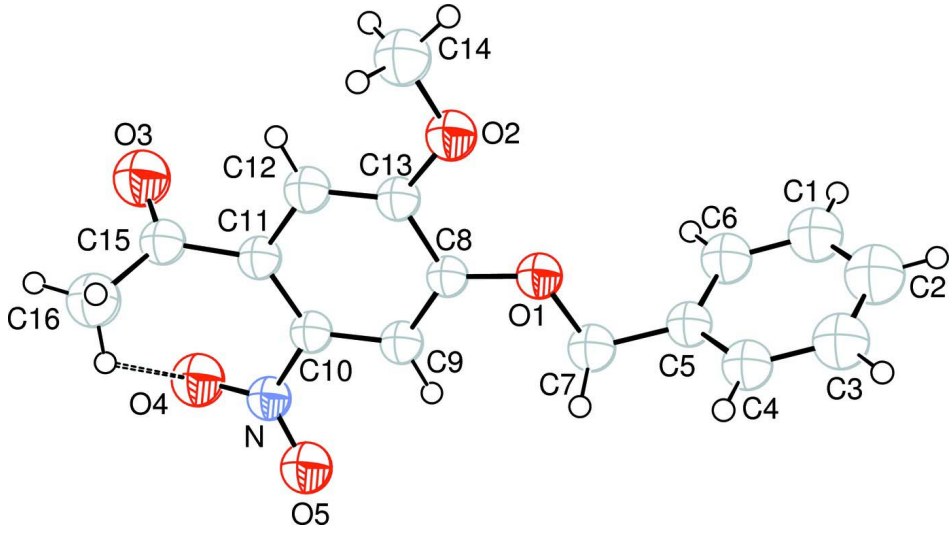


**Figure 1**

The molecular structure of the title molecule, with the atom-numbering scheme.

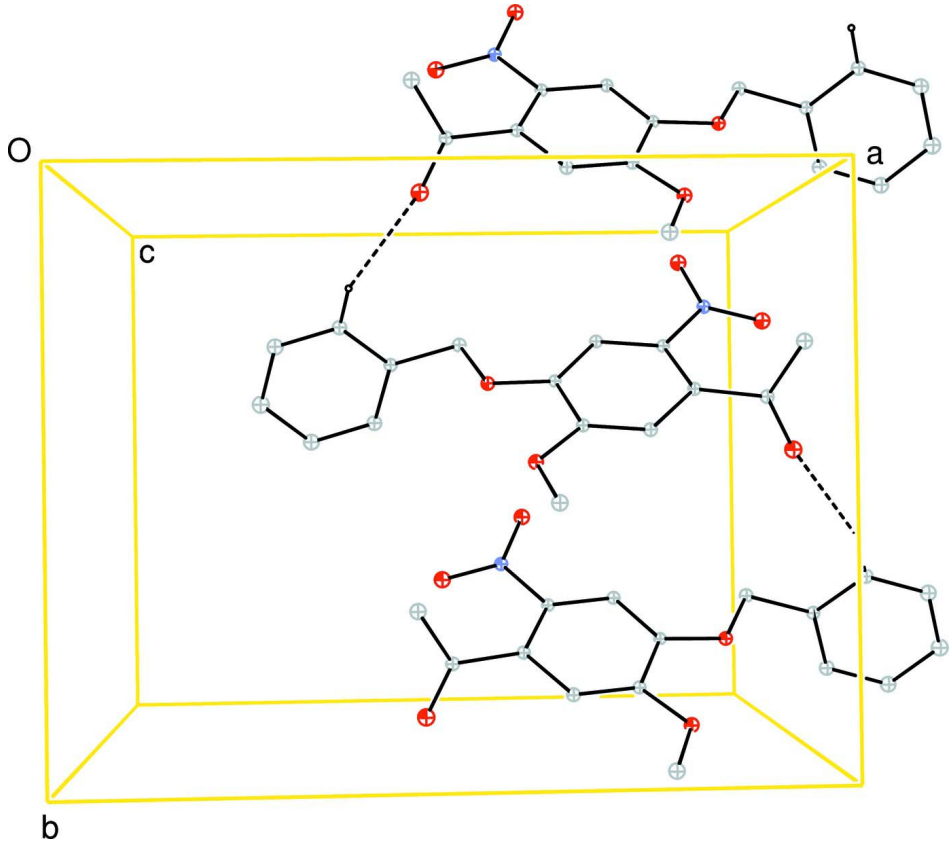


**Figure 2**

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in hydrogen bonding have been omitted.

**1-(4-Benzyloxy-5-methoxy-2-nitrophenyl)ethanone**

*Crystal data*

$C_{16}H_{15}NO_5$
$M_r = 301.29$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 13.390$ (3) Å
$b = 10.465$ (2) Å
$c = 20.768$ (4) Å
$V = 2910.1$ (10) Å$^3$
$Z = 8$
$F(000) = 1264$
$D_x = 1.375$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 1.0$–$1.0°$
$\mu = 0.10$ mm$^{-1}$
$T = 294$ K
Needle, colorless
0.20 × 0.10 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min} = 0.980$, $T_{max} = 0.990$
2634 measured reflections
2634 independent reflections
1446 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.0°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 24$
3 standard reflections every 120 min
intensity decay: none

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.223$
$S = 1.06$
2634 reflections
199 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
$w = 1/[\sigma^2(F_o^2) + (0.118P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N | 0.4084 (2) | 0.3120 (3) | 0.33235 (15) | 0.0456 (8) |
| O1 | 0.07225 (17) | 0.1644 (3) | 0.38239 (11) | 0.0460 (7) |
| O2 | 0.13768 (18) | 0.0124 (3) | 0.46870 (13) | 0.0538 (8) |
| O3 | 0.5275 (2) | 0.0290 (3) | 0.42219 (18) | 0.0748 (10) |

| | | | | |
|---|---|---|---|---|
| O4 | 0.4987 (2) | 0.2830 (3) | 0.33126 (15) | 0.0684 (10) |
| O5 | 0.3740 (2) | 0.4020 (3) | 0.30338 (14) | 0.0633 (9) |
| C1 | −0.1972 (3) | 0.0558 (5) | 0.2841 (2) | 0.0683 (13) |
| H1A | −0.2128 | −0.0196 | 0.2626 | 0.082* |
| C2 | −0.2731 (4) | 0.1305 (6) | 0.3092 (3) | 0.0793 (16) |
| H2A | −0.3394 | 0.1054 | 0.3051 | 0.095* |
| C3 | −0.2488 (3) | 0.2420 (5) | 0.3400 (3) | 0.0682 (13) |
| H3A | −0.2994 | 0.2942 | 0.3558 | 0.082* |
| C4 | −0.1506 (3) | 0.2778 (4) | 0.3478 (2) | 0.0546 (11) |
| H4A | −0.1357 | 0.3519 | 0.3706 | 0.065* |
| C5 | −0.0736 (3) | 0.2049 (4) | 0.32217 (18) | 0.0461 (10) |
| C6 | −0.0996 (3) | 0.0917 (4) | 0.2906 (2) | 0.0592 (12) |
| H6A | −0.0495 | 0.0399 | 0.2737 | 0.071* |
| C7 | 0.0318 (3) | 0.2420 (4) | 0.3310 (2) | 0.0510 (10) |
| H7A | 0.0690 | 0.2277 | 0.2915 | 0.061* |
| H7B | 0.0364 | 0.3318 | 0.3420 | 0.061* |
| C8 | 0.1732 (3) | 0.1672 (3) | 0.39057 (17) | 0.0380 (9) |
| C9 | 0.2392 (3) | 0.2446 (4) | 0.35806 (18) | 0.0428 (9) |
| H9A | 0.2159 | 0.3038 | 0.3282 | 0.051* |
| C10 | 0.3402 (2) | 0.2336 (4) | 0.37010 (17) | 0.0409 (9) |
| C11 | 0.3807 (3) | 0.1481 (4) | 0.41517 (18) | 0.0430 (9) |
| C12 | 0.3098 (3) | 0.0707 (4) | 0.44838 (19) | 0.0478 (10) |
| H12A | 0.3323 | 0.0118 | 0.4786 | 0.057* |
| C13 | 0.2105 (3) | 0.0807 (4) | 0.43705 (18) | 0.0422 (9) |
| C14 | 0.1676 (3) | −0.0629 (5) | 0.5236 (2) | 0.0661 (13) |
| H14A | 0.1103 | −0.1054 | 0.5413 | 0.099* |
| H14B | 0.1964 | −0.0080 | 0.5557 | 0.099* |
| H14C | 0.2160 | −0.1253 | 0.5104 | 0.099* |
| C15 | 0.4875 (3) | 0.1311 (4) | 0.43162 (19) | 0.0478 (10) |
| C16 | 0.5384 (3) | 0.2346 (5) | 0.4663 (2) | 0.0652 (13) |
| H16A | 0.6068 | 0.2115 | 0.4735 | 0.098* |
| H16B | 0.5059 | 0.2486 | 0.5069 | 0.098* |
| H16C | 0.5356 | 0.3115 | 0.4411 | 0.098* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N | 0.0501 (17) | 0.049 (2) | 0.0377 (18) | −0.0016 (16) | 0.0013 (14) | −0.0030 (16) |
| O1 | 0.0448 (14) | 0.0553 (18) | 0.0378 (15) | −0.0030 (12) | −0.0076 (11) | 0.0163 (13) |
| O2 | 0.0561 (15) | 0.0561 (19) | 0.0492 (16) | −0.0062 (13) | −0.0013 (13) | 0.0211 (14) |
| O3 | 0.0644 (19) | 0.056 (2) | 0.104 (3) | 0.0188 (17) | −0.0083 (17) | −0.0031 (19) |
| O4 | 0.0493 (16) | 0.083 (2) | 0.073 (2) | −0.0008 (15) | 0.0050 (14) | 0.0105 (18) |
| O5 | 0.0683 (18) | 0.057 (2) | 0.065 (2) | −0.0072 (15) | −0.0065 (15) | 0.0197 (17) |
| C1 | 0.076 (3) | 0.059 (3) | 0.070 (3) | −0.014 (3) | −0.017 (3) | −0.009 (2) |
| C2 | 0.059 (3) | 0.081 (4) | 0.098 (4) | −0.018 (3) | −0.016 (3) | 0.019 (3) |
| C3 | 0.049 (2) | 0.067 (3) | 0.089 (3) | 0.004 (2) | 0.003 (2) | 0.009 (3) |
| C4 | 0.055 (2) | 0.048 (3) | 0.060 (3) | 0.003 (2) | 0.005 (2) | 0.000 (2) |
| C5 | 0.055 (2) | 0.041 (2) | 0.043 (2) | 0.0047 (18) | −0.0095 (17) | 0.0119 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C6 | 0.064 (3) | 0.050 (3) | 0.063 (3) | 0.001 (2) | −0.009 (2) | −0.001 (2) |
| C7 | 0.053 (2) | 0.054 (3) | 0.046 (2) | 0.001 (2) | −0.0072 (18) | 0.009 (2) |
| C8 | 0.0472 (19) | 0.037 (2) | 0.0300 (18) | −0.0084 (16) | −0.0018 (15) | 0.0034 (16) |
| C9 | 0.0491 (19) | 0.037 (2) | 0.042 (2) | 0.0030 (17) | −0.0059 (17) | 0.0082 (17) |
| C10 | 0.0400 (18) | 0.043 (2) | 0.039 (2) | −0.0042 (16) | −0.0018 (16) | −0.0059 (17) |
| C11 | 0.050 (2) | 0.038 (2) | 0.040 (2) | 0.0024 (18) | −0.0055 (17) | 0.0017 (18) |
| C12 | 0.053 (2) | 0.040 (2) | 0.050 (3) | 0.0087 (18) | −0.0106 (19) | 0.0086 (18) |
| C13 | 0.050 (2) | 0.036 (2) | 0.041 (2) | −0.0040 (17) | −0.0026 (17) | 0.0077 (17) |
| C14 | 0.075 (3) | 0.066 (3) | 0.057 (3) | −0.010 (2) | −0.008 (2) | 0.019 (2) |
| C15 | 0.050 (2) | 0.052 (3) | 0.041 (2) | 0.0042 (19) | −0.0028 (17) | −0.002 (2) |
| C16 | 0.059 (2) | 0.073 (3) | 0.063 (3) | 0.001 (2) | −0.016 (2) | −0.016 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N—O5 | 1.209 (4) | C6—H6A | 0.9300 |
| N—O4 | 1.247 (4) | C7—H7A | 0.9700 |
| N—C10 | 1.456 (5) | C7—H7B | 0.9700 |
| O1—C8 | 1.363 (4) | C8—C9 | 1.376 (5) |
| O1—C7 | 1.446 (4) | C8—C13 | 1.414 (5) |
| O2—C13 | 1.376 (4) | C9—C10 | 1.380 (5) |
| O2—C14 | 1.443 (5) | C9—H9A | 0.9300 |
| O3—C15 | 1.212 (5) | C10—C11 | 1.404 (5) |
| C1—C6 | 1.367 (5) | C11—C12 | 1.426 (5) |
| C1—C2 | 1.384 (7) | C11—C15 | 1.480 (5) |
| C1—H1A | 0.9300 | C12—C13 | 1.355 (5) |
| C2—C3 | 1.370 (7) | C12—H12A | 0.9300 |
| C2—H2A | 0.9300 | C14—H14A | 0.9600 |
| C3—C4 | 1.377 (5) | C14—H14B | 0.9600 |
| C3—H3A | 0.9300 | C14—H14C | 0.9600 |
| C4—C5 | 1.389 (5) | C15—C16 | 1.468 (6) |
| C4—H4A | 0.9300 | C16—H16A | 0.9600 |
| C5—C6 | 1.398 (6) | C16—H16B | 0.9600 |
| C5—C7 | 1.475 (5) | C16—H16C | 0.9600 |
| | | | |
| O5—N—O4 | 123.3 (3) | C9—C8—C13 | 119.0 (3) |
| O5—N—C10 | 117.9 (3) | C8—C9—C10 | 119.4 (3) |
| O4—N—C10 | 118.7 (3) | C8—C9—H9A | 120.3 |
| C8—O1—C7 | 116.8 (3) | C10—C9—H9A | 120.3 |
| C13—O2—C14 | 117.7 (3) | C9—C10—C11 | 123.5 (3) |
| C6—C1—C2 | 120.6 (5) | C9—C10—N | 118.0 (3) |
| C6—C1—H1A | 119.7 | C11—C10—N | 118.4 (3) |
| C2—C1—H1A | 119.7 | C10—C11—C12 | 115.3 (3) |
| C3—C2—C1 | 118.9 (4) | C10—C11—C15 | 127.1 (3) |
| C3—C2—H2A | 120.6 | C12—C11—C15 | 117.6 (3) |
| C1—C2—H2A | 120.6 | C13—C12—C11 | 121.7 (4) |
| C2—C3—C4 | 120.9 (5) | C13—C12—H12A | 119.1 |
| C2—C3—H3A | 119.5 | C11—C12—H12A | 119.1 |
| C4—C3—H3A | 119.5 | C12—C13—O2 | 124.9 (4) |

| | | | |
|---|---|---|---|
| C3—C4—C5 | 120.9 (4) | C12—C13—C8 | 121.0 (3) |
| C3—C4—H4A | 119.5 | O2—C13—C8 | 114.1 (3) |
| C5—C4—H4A | 119.5 | O2—C14—H14A | 109.5 |
| C4—C5—C6 | 117.4 (4) | O2—C14—H14B | 109.5 |
| C4—C5—C7 | 121.2 (4) | H14A—C14—H14B | 109.5 |
| C6—C5—C7 | 121.3 (4) | O2—C14—H14C | 109.5 |
| C1—C6—C5 | 121.1 (4) | H14A—C14—H14C | 109.5 |
| C1—C6—H6A | 119.4 | H14B—C14—H14C | 109.5 |
| C5—C6—H6A | 119.4 | O3—C15—C16 | 121.6 (4) |
| O1—C7—C5 | 107.6 (3) | O3—C15—C11 | 119.7 (4) |
| O1—C7—H7A | 110.2 | C16—C15—C11 | 118.2 (4) |
| C5—C7—H7A | 110.2 | C15—C16—H16A | 109.5 |
| O1—C7—H7B | 110.2 | C15—C16—H16B | 109.5 |
| C5—C7—H7B | 110.2 | H16A—C16—H16B | 109.5 |
| H7A—C7—H7B | 108.5 | C15—C16—H16C | 109.5 |
| O1—C8—C9 | 126.0 (3) | H16A—C16—H16C | 109.5 |
| O1—C8—C13 | 115.0 (3) | H16B—C16—H16C | 109.5 |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 0.8 (8) | O4—N—C10—C11 | 14.6 (5) |
| C1—C2—C3—C4 | −2.0 (8) | C9—C10—C11—C12 | 0.4 (5) |
| C2—C3—C4—C5 | 2.9 (7) | N—C10—C11—C12 | −177.4 (3) |
| C3—C4—C5—C6 | −2.5 (6) | C9—C10—C11—C15 | −179.2 (4) |
| C3—C4—C5—C7 | −179.4 (4) | N—C10—C11—C15 | 3.0 (6) |
| C2—C1—C6—C5 | −0.5 (7) | C10—C11—C12—C13 | −0.4 (6) |
| C4—C5—C6—C1 | 1.3 (6) | C15—C11—C12—C13 | 179.2 (4) |
| C7—C5—C6—C1 | 178.1 (4) | C11—C12—C13—O2 | −177.7 (3) |
| C8—O1—C7—C5 | 168.6 (3) | C11—C12—C13—C8 | 1.3 (6) |
| C4—C5—C7—O1 | 100.4 (4) | C14—O2—C13—C12 | 8.6 (6) |
| C6—C5—C7—O1 | −76.4 (5) | C14—O2—C13—C8 | −170.5 (4) |
| C7—O1—C8—C9 | 5.7 (5) | O1—C8—C13—C12 | 177.8 (4) |
| C7—O1—C8—C13 | −174.0 (3) | C9—C8—C13—C12 | −2.0 (6) |
| O1—C8—C9—C10 | −177.9 (3) | O1—C8—C13—O2 | −3.1 (5) |
| C13—C8—C9—C10 | 1.9 (5) | C9—C8—C13—O2 | 177.1 (3) |
| C8—C9—C10—C11 | −1.1 (6) | C10—C11—C15—O3 | −118.1 (5) |
| C8—C9—C10—N | 176.7 (3) | C12—C11—C15—O3 | 62.3 (5) |
| O5—N—C10—C9 | 16.4 (5) | C10—C11—C15—C16 | 69.2 (5) |
| O4—N—C10—C9 | −163.3 (3) | C12—C11—C15—C16 | −110.4 (4) |
| O5—N—C10—C11 | −165.6 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4*A*···O3$^{i}$ | 0.93 | 2.58 | 3.466 (5) | 158 |
| C16—H16*C*···O4 | 0.96 | 2.35 | 2.899 (5) | 115 |

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