Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Dimethyl 2-(2-quinolylmethyl)malonate

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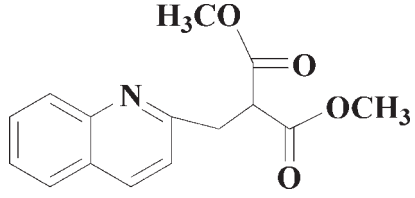
Received 29 March 2010; accepted 15 April 2010

Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.035; $wR$ factor = 0.095; data-to-parameter ratio = 12.8.

In the title compound, $C_{15}H_{15}NO_4$, the quinoline ring system and one of the malonate side chains are essentially coplanar (r.m.s. deviation = 0.0297 Å). The two malonate C—C(=O)—O—$CH_3$ side chains are oriented at right angles [89.68 (8)°] with respect to each other. The crystal packing is stabilized by weak non-classical intermolecular C—H···O hydrogen bonds, which link the molecules into dimers about inversion centers.

## Related literature

For general background to the synthesis of halomalonates, see: Okimoto & Takahashi (2002).




## Experimental

### Crystal data

$C_{15}H_{15}NO_4$ $M_r$ = 273.28
Monoclinic, $P2_1/c$
$a$ = 10.626 (2) Å
$b$ = 16.198 (3) Å
$c$ = 8.1859 (16) Å
$\beta$ = 107.92 (3)°
$V$ = 1340.6 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 113 K
0.20 × 0.18 × 0.12 mm

### Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min}$ = 0.981, $T_{max}$ = 0.988
8841 measured reflections
2355 independent reflections
2094 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.095
$S$ = 1.07
2355 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C11—H11···O1$^{i}$ | 0.98 | 2.49 | 3.410 (2) | 157 |

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported by the NSFC (grant No. 30873139).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2271).

## References

Okimoto, M. & Takahashi, Y. (2002). *Synthesis*, **15**, 2215–2219
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2010). E**66**, o1383 [https://doi.org/10.1107/S1600536810013930]

## Dimethyl 2-(2-quinolylmethyl)malonate

**Shou-Xin Liu, Zhao-Hui Zhang, Yu-Feng Yang, Xiao-Li Zhen and Jian-Rong Han**

### S1. Comment

Substituted malonate is a very important organic intermediate. It is electrooxidized in methanol in the presence of halogen ions to afford the corresponding halomalonates (Okimoto & Takahashi, 2002). We have synthesized the title compound, (I), in our laboratory and report in this article its synthesis and crystal structure.

The molecular structure of (I) is presented in Fig. 1. In (I), the quinoline ring and the side chain atoms C10/C11/C14 are essentially planar [r.m.s. deviation, 0.0297 Å]. The two malonate side chains comprising C/C/O/C atoms (C11/C12/O1/C13 and C11/C14/O3/C15) are oriented at right angles (89.68 (8)°) with respect to each other. The crystal packing is stabilized by weak non-classical intermolecular C—H···O hydrogen bonds which link the molecules into dimers about inversion centers.

### S2. Experimental

An anhydrous methanol solution (130 ml) of 2-brommethyl quinoline was added to an anhydrous methanol solution (180 ml) of sodium methoxide (5.4 g, 0.1 mol) and dimethyl malonate (26.4 g, 0.2 mol). The mixture was refluxed for 4 h and the product was isolated with silica gel column. The solvent was removed and a little petroleum ether was added to the resultant to give pale-yellow precipitates which were isolated, recrystallized from n-hexane, and dried under vacuum to give the title compound (60% yield). Colorless single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an n-hexane solution.

### S3. Refinement

The H atoms were included in calculated positions (C—H = 0.93–0.98 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

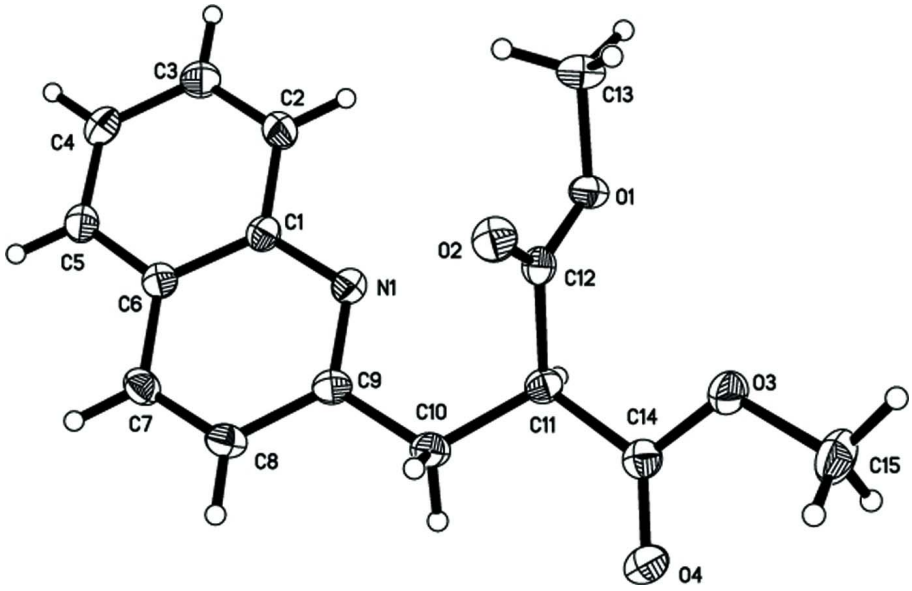



**Figure 1**
Ellipsoid plot.

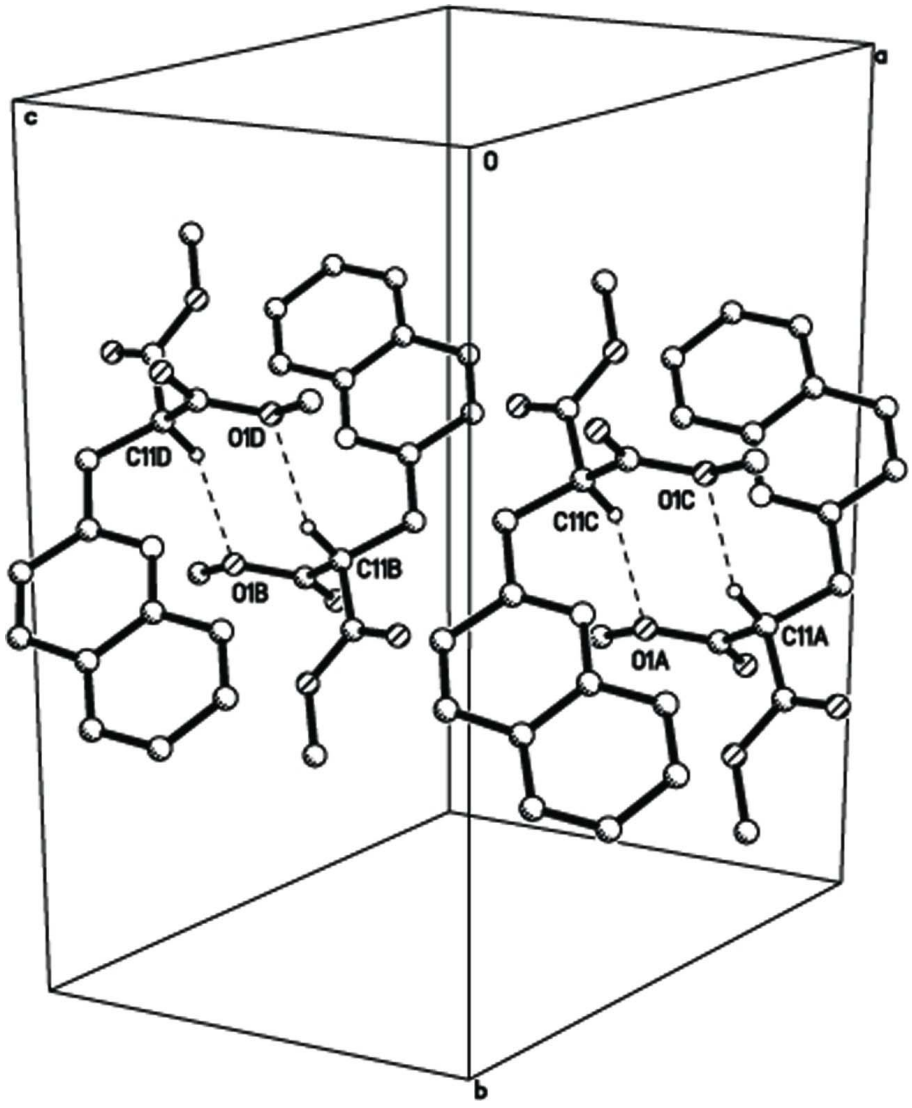



**Figure 2**

Packing diagram.

**Dimethyl 2-(2-quinolylmethyl)malonate**

*Crystal data*

$C_{15}H_{15}NO_4$
$M_r = 273.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.626\ (2)$ Å
$b = 16.198\ (3)$ Å
$c = 8.1859\ (16)$ Å
$\beta = 107.92\ (3)°$
$V = 1340.6\ (5)$ Å$^3$
$Z = 4$

$F(000) = 576$
$D_x = 1.354$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4229 reflections
$\theta = 2.0–27.9°$
$\mu = 0.10$ mm$^{-1}$
$T = 113$ K
Prism, colourless
0.20 × 0.18 × 0.12 mm

*Data collection*

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm$^{-1}$
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min}$ = 0.981, $T_{max}$ = 0.988
8841 measured reflections
2355 independent reflections
2094 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.4°
$h$ = −12→7
$k$ = −19→18
$l$ = −9→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.095
$S$ = 1.07
2355 reflections
184 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
$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.2353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.133 (8)

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.42646 (9) | 0.11002 (5) | 0.37324 (10) | 0.0208 (2) |
| O2 | 0.28197 (9) | 0.17212 (5) | 0.48439 (11) | 0.0238 (3) |
| O3 | 0.57807 (9) | 0.20028 (5) | 0.71040 (11) | 0.0259 (3) |
| O4 | 0.58734 (10) | 0.12269 (6) | 0.94065 (12) | 0.0315 (3) |
| N1 | 0.24000 (10) | −0.02029 (6) | 0.47394 (13) | 0.0193 (3) |
| C1 | 0.14301 (12) | −0.06967 (7) | 0.37093 (16) | 0.0186 (3) |
| C2 | 0.13159 (14) | −0.07574 (8) | 0.19515 (17) | 0.0249 (3) |
| H2 | 0.1898 | −0.0466 | 0.1519 | 0.030* |
| C3 | 0.03559 (14) | −0.12414 (8) | 0.08742 (17) | 0.0275 (3) |
| H3 | 0.0287 | −0.1271 | −0.0285 | 0.033* |
| C4 | −0.05277 (13) | −0.16937 (8) | 0.15063 (17) | 0.0240 (3) |
| H4 | −0.1164 | −0.2029 | 0.0771 | 0.029* |
| C5 | −0.04480 (13) | −0.16388 (8) | 0.32021 (17) | 0.0217 (3) |
| H5 | −0.1036 | −0.1937 | 0.3612 | 0.026* |

| | | | | |
|---|---|---|---|---|
| C6 | 0.05144 (13) | −0.11361 (7) | 0.43356 (16) | 0.0186 (3) |
| C7 | 0.06142 (13) | −0.10242 (8) | 0.60895 (16) | 0.0220 (3) |
| H7 | 0.0030 | −0.1293 | 0.6554 | 0.026* |
| C8 | 0.15648 (13) | −0.05239 (8) | 0.70838 (16) | 0.0224 (3) |
| H8 | 0.1631 | −0.0444 | 0.8232 | 0.027* |
| C9 | 0.24603 (12) | −0.01216 (7) | 0.63656 (15) | 0.0189 (3) |
| C10 | 0.35008 (13) | 0.04359 (8) | 0.74929 (15) | 0.0210 (3) |
| H10A | 0.4004 | 0.0127 | 0.8496 | 0.025* |
| H10B | 0.3065 | 0.0886 | 0.7886 | 0.025* |
| C11 | 0.44620 (12) | 0.07996 (7) | 0.66261 (15) | 0.0183 (3) |
| H11 | 0.4954 | 0.0344 | 0.6324 | 0.022* |
| C12 | 0.37365 (12) | 0.12590 (7) | 0.49881 (15) | 0.0182 (3) |
| C13 | 0.35706 (15) | 0.14582 (9) | 0.20836 (16) | 0.0306 (3) |
| H13A | 0.2709 | 0.1213 | 0.1651 | 0.046* |
| H13B | 0.4060 | 0.1357 | 0.1295 | 0.046* |
| H13C | 0.3481 | 0.2042 | 0.2211 | 0.046* |
| C14 | 0.54437 (12) | 0.13576 (8) | 0.78861 (16) | 0.0203 (3) |
| C15 | 0.67016 (15) | 0.25858 (9) | 0.81881 (19) | 0.0318 (4) |
| H15A | 0.6337 | 0.2801 | 0.9038 | 0.048* |
| H15B | 0.6855 | 0.3030 | 0.7498 | 0.048* |
| H15C | 0.7522 | 0.2312 | 0.8747 | 0.048* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0226 (5) | 0.0238 (5) | 0.0158 (5) | 0.0004 (4) | 0.0055 (4) | 0.0020 (3) |
| O2 | 0.0218 (5) | 0.0216 (5) | 0.0273 (5) | 0.0050 (4) | 0.0067 (4) | 0.0024 (4) |
| O3 | 0.0279 (5) | 0.0222 (5) | 0.0243 (5) | −0.0075 (4) | 0.0032 (4) | −0.0002 (4) |
| O4 | 0.0320 (6) | 0.0367 (6) | 0.0204 (5) | −0.0099 (4) | 0.0004 (4) | 0.0007 (4) |
| N1 | 0.0180 (6) | 0.0198 (6) | 0.0194 (5) | 0.0002 (4) | 0.0048 (4) | 0.0000 (4) |
| C1 | 0.0166 (7) | 0.0180 (6) | 0.0205 (6) | 0.0025 (5) | 0.0046 (5) | 0.0006 (5) |
| C2 | 0.0233 (7) | 0.0314 (7) | 0.0216 (7) | −0.0043 (6) | 0.0095 (6) | −0.0014 (5) |
| C3 | 0.0283 (8) | 0.0353 (8) | 0.0188 (7) | −0.0035 (6) | 0.0070 (6) | −0.0043 (6) |
| C4 | 0.0198 (7) | 0.0245 (7) | 0.0247 (7) | −0.0015 (5) | 0.0021 (6) | −0.0039 (5) |
| C5 | 0.0190 (7) | 0.0194 (6) | 0.0262 (7) | 0.0003 (5) | 0.0062 (6) | 0.0026 (5) |
| C6 | 0.0179 (7) | 0.0156 (6) | 0.0218 (6) | 0.0039 (5) | 0.0054 (5) | 0.0032 (5) |
| C7 | 0.0240 (7) | 0.0212 (7) | 0.0224 (7) | −0.0013 (5) | 0.0094 (6) | 0.0043 (5) |
| C8 | 0.0272 (7) | 0.0225 (6) | 0.0176 (6) | 0.0000 (5) | 0.0072 (6) | 0.0024 (5) |
| C9 | 0.0207 (7) | 0.0166 (6) | 0.0184 (6) | 0.0035 (5) | 0.0045 (5) | 0.0022 (5) |
| C10 | 0.0243 (7) | 0.0212 (6) | 0.0171 (6) | −0.0012 (5) | 0.0059 (5) | 0.0012 (5) |
| C11 | 0.0193 (7) | 0.0174 (6) | 0.0173 (6) | 0.0010 (5) | 0.0044 (5) | −0.0003 (5) |
| C12 | 0.0189 (7) | 0.0155 (6) | 0.0197 (6) | −0.0034 (5) | 0.0052 (5) | −0.0015 (5) |
| C13 | 0.0328 (8) | 0.0384 (8) | 0.0182 (7) | −0.0002 (6) | 0.0044 (6) | 0.0070 (6) |
| C14 | 0.0180 (7) | 0.0215 (7) | 0.0212 (7) | 0.0028 (5) | 0.0059 (5) | 0.0002 (5) |
| C15 | 0.0280 (8) | 0.0252 (7) | 0.0368 (8) | −0.0083 (6) | 0.0019 (7) | −0.0041 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C12 | 1.3389 (15) | C6—C7 | 1.4184 (18) |
| O1—C13 | 1.4454 (16) | C7—C8 | 1.3542 (19) |
| O2—C12 | 1.2054 (15) | C7—H7 | 0.9300 |
| O3—C14 | 1.3309 (15) | C8—C9 | 1.4212 (18) |
| O3—C15 | 1.4500 (16) | C8—H8 | 0.9300 |
| O4—C14 | 1.2052 (16) | C9—C10 | 1.5039 (18) |
| N1—C9 | 1.3195 (16) | C10—C11 | 1.5296 (17) |
| N1—C1 | 1.3705 (17) | C10—H10A | 0.9700 |
| C1—C2 | 1.4098 (18) | C10—H10B | 0.9700 |
| C1—C6 | 1.4222 (18) | C11—C14 | 1.5189 (18) |
| C2—C3 | 1.3710 (19) | C11—C12 | 1.5197 (17) |
| C2—H2 | 0.9300 | C11—H11 | 0.9800 |
| C3—C4 | 1.4089 (19) | C13—H13A | 0.9600 |
| C3—H3 | 0.9300 | C13—H13B | 0.9600 |
| C4—C5 | 1.3673 (18) | C13—H13C | 0.9600 |
| C4—H4 | 0.9300 | C15—H15A | 0.9600 |
| C5—C6 | 1.4093 (19) | C15—H15B | 0.9600 |
| C5—H5 | 0.9300 | C15—H15C | 0.9600 |
| | | | |
| C12—O1—C13 | 115.21 (10) | C9—C10—C11 | 114.61 (10) |
| C14—O3—C15 | 116.60 (10) | C9—C10—H10A | 108.6 |
| C9—N1—C1 | 118.22 (11) | C11—C10—H10A | 108.6 |
| N1—C1—C2 | 118.74 (11) | C9—C10—H10B | 108.6 |
| N1—C1—C6 | 122.59 (11) | C11—C10—H10B | 108.6 |
| C2—C1—C6 | 118.65 (12) | H10A—C10—H10B | 107.6 |
| C3—C2—C1 | 120.65 (12) | C14—C11—C12 | 111.33 (10) |
| C3—C2—H2 | 119.7 | C14—C11—C10 | 109.35 (10) |
| C1—C2—H2 | 119.7 | C12—C11—C10 | 111.59 (10) |
| C2—C3—C4 | 120.61 (12) | C14—C11—H11 | 108.1 |
| C2—C3—H3 | 119.7 | C12—C11—H11 | 108.1 |
| C4—C3—H3 | 119.7 | C10—C11—H11 | 108.1 |
| C5—C4—C3 | 119.93 (12) | O2—C12—O1 | 124.13 (11) |
| C5—C4—H4 | 120.0 | O2—C12—C11 | 124.46 (11) |
| C3—C4—H4 | 120.0 | O1—C12—C11 | 111.39 (10) |
| C4—C5—C6 | 120.78 (12) | O1—C13—H13A | 109.5 |
| C4—C5—H5 | 119.6 | O1—C13—H13B | 109.5 |
| C6—C5—H5 | 119.6 | H13A—C13—H13B | 109.5 |
| C5—C6—C7 | 123.60 (12) | O1—C13—H13C | 109.5 |
| C5—C6—C1 | 119.35 (11) | H13A—C13—H13C | 109.5 |
| C7—C6—C1 | 117.04 (12) | H13B—C13—H13C | 109.5 |
| C8—C7—C6 | 119.63 (11) | O4—C14—O3 | 124.21 (12) |
| C8—C7—H7 | 120.2 | O4—C14—C11 | 124.03 (12) |
| C6—C7—H7 | 120.2 | O3—C14—C11 | 111.76 (10) |
| C7—C8—C9 | 119.76 (11) | O3—C15—H15A | 109.5 |
| C7—C8—H8 | 120.1 | O3—C15—H15B | 109.5 |
| C9—C8—H8 | 120.1 | H15A—C15—H15B | 109.5 |

| | | | |
|---|---|---|---|
| N1—C9—C8 | 122.73 (12) | O3—C15—H15C | 109.5 |
| N1—C9—C10 | 118.51 (11) | H15A—C15—H15C | 109.5 |
| C8—C9—C10 | 118.74 (11) | H15B—C15—H15C | 109.5 |
| | | | |
| C9—N1—C1—C2 | 177.02 (11) | C7—C8—C9—N1 | 1.02 (19) |
| C9—N1—C1—C6 | −1.52 (17) | C7—C8—C9—C10 | 179.55 (11) |
| N1—C1—C2—C3 | −179.71 (12) | N1—C9—C10—C11 | −4.71 (16) |
| C6—C1—C2—C3 | −1.11 (19) | C8—C9—C10—C11 | 176.70 (11) |
| C1—C2—C3—C4 | −0.6 (2) | C9—C10—C11—C14 | 179.17 (10) |
| C2—C3—C4—C5 | 1.3 (2) | C9—C10—C11—C12 | 55.55 (14) |
| C3—C4—C5—C6 | −0.27 (19) | C13—O1—C12—O2 | −6.25 (17) |
| C4—C5—C6—C7 | 177.20 (12) | C13—O1—C12—C11 | 175.19 (10) |
| C4—C5—C6—C1 | −1.43 (18) | C14—C11—C12—O2 | −79.24 (15) |
| N1—C1—C6—C5 | −179.36 (11) | C10—C11—C12—O2 | 43.25 (16) |
| C2—C1—C6—C5 | 2.10 (17) | C14—C11—C12—O1 | 99.32 (11) |
| N1—C1—C6—C7 | 1.92 (17) | C10—C11—C12—O1 | −138.19 (11) |
| C2—C1—C6—C7 | −176.62 (11) | C15—O3—C14—O4 | −1.50 (18) |
| C5—C6—C7—C8 | −179.49 (12) | C15—O3—C14—C11 | 179.13 (10) |
| C1—C6—C7—C8 | −0.82 (18) | C12—C11—C14—O4 | 159.19 (13) |
| C6—C7—C8—C9 | −0.55 (19) | C10—C11—C14—O4 | 35.42 (17) |
| C1—N1—C9—C8 | 0.03 (17) | C12—C11—C14—O3 | −21.44 (14) |
| C1—N1—C9—C10 | −178.51 (10) | C10—C11—C14—O3 | −145.21 (10) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C11—H11···O1$^{i}$ | 0.98 | 2.49 | 3.410 (2) | 157 |

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