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

# 5-[(8-Hydroxyquinolin-2-ylamino)-methylene]-2,2-dimethyl-1,3-dioxane-4,6-dione

**Luiz Everson da Silva,[a] Paulo Teixeira de Sousa Jr,[a] Evandro Luiz Dall'Oglio[a] and Sabine Foro[b]***

[a]Departamento de Química, Universidale Federal de Mato Grosso, 78060-900 Cuiabá, MT, Brazil, and [b]Clemens Schöpf-Institut für Organische Chemie und Biochemie, Technische Universität Darmstadt, Petersenstrasse 22, D-64287 Darmstadt, Germany
Correspondence e-mail: foro@tu-darmstadt.de

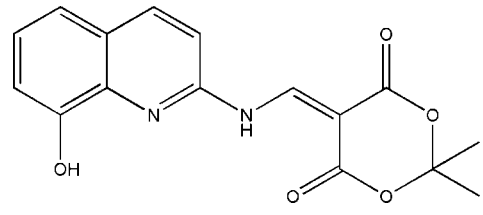
Received 20 June 2007; accepted 2 July 2007

Key indicators: single-crystal X-ray study; $T$ = 299 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.046; $wR$ factor = 0.119; data-to-parameter ratio = 11.6.

In the title compound, $C_{16}H_{14}N_2O_5$, the 1,3-dioxane-4,6-dione ring is in an envelope conformation. The C—N—C—C torsion angle between the nearly planar quinoline ring system carrying the OH group and the 1,3-dioxane-4,6-dione unit is −177.1 (1)°. The NH group has an intramolecular contact to a carbonyl O atom, forming a six-membered ring, and also an intermolecular contact to the carbonyl O atom. One intramolecular O—H···N and two intermolecular C—H···O hydrogen bonds are also observed. Two molecules are connected by these hydrogen bonds, forming layers.

## Related literature

For related literature, see: Cassis *et al.* (1985); Gaber & McNab (2001); da Silva *et al.* (2002, 2007); Storz (2004).




## Experimental

### Crystal data

$C_{16}H_{14}N_2O_5$
$M_r$ = 314.29
Triclinic, $P\overline{1}$
$a$ = 7.445 (2) Å
$b$ = 8.440 (2) Å
$c$ = 12.906 (3) Å
$\alpha$ = 77.31 (1)°
$\beta$ = 86.81 (2)°
$\gamma$ = 73.15 (1)°
$V$ = 757.2 (3) Å$^3$
$Z$ = 2
Cu $K\alpha$ radiation
$\mu$ = 0.88 mm$^{-1}$
$T$ = 299 (2) K
0.60 × 0.40 × 0.33 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
5325 measured reflections
2704 independent reflections
2436 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.053
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.120
$S$ = 1.08
2704 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2N···O4 | 0.85 (2) | 2.140 (19) | 2.7428 (17) | 127.6 (14) |
| N2—H2N···O4$^{i}$ | 0.85 (2) | 2.498 (18) | 3.2943 (17) | 156.2 (15) |
| O3—H3O···N1 | 0.79 (2) | 2.22 (2) | 2.7028 (16) | 120.0 (18) |
| C2—H2···O1$^{i}$ | 0.97 (2) | 2.564 (19) | 3.4151 (19) | 146.6 (14) |
| C2—H2···O4$^{i}$ | 0.97 (2) | 2.35 (2) | 3.2533 (19) | 155.4 (14) |

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC Software*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank Professor Dr Hartmut Fuess, Technische Universität Darmstadt, for diffractometer time.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2044).

## References

Cassis, R., Tapia, R. & Valerama, J. A. (1985). *Synth. Commun.* **15**, 125–129.
Enraf–Nonius (1996). *CAD-4-PC Software*. Version 2.0. Enraf–Nonius GmbH, Solingen, Germany.
Gaber, A. E.-A. M. & McNab, H. (2001). *Synthesis*, **14**, 2059–2074.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Silva, L. E. da, Joussef, A. C., Pacheco, L. K., Albino, D. B. L., Duarte, A. M. C., Steindel, M. & Rebelo, R. A. (2007). *Lett. Drug. Des. Discov.* **4**, 154–159.
Silva, L. E. da, Rebelo, R. A., Graf, G. I., Hastreiter, D., Montalban, A. G. & McKillop, A. (2002). *Tetrahedron*, **58**, 9095–9100.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1987). *REDU4*. Version 6.2c. Stoe & Cie GmbH, Darmstadt, Germany.
Storz, T. (2004). *Org. Process. Res. Dev.* **8**, 663–665.

# supplementary materials

*Acta Cryst.* (2007). E**63**, o3439 [ **doi:10.1107/S160053680703214X** ]

# 5-[(8-Hydroxyquinolin-2-ylamino)methylene]-2,2-dimethyl-1,3-dioxane-4,6-dione

**L. E. da Silva, P. T. de Sousa Jr, E. L. Dall'Oglio and S. Foro**

## Comment

Aminoquinolines are important constituents in a variety of pharmaceutically important compound classes, most notably perhaps the antimalarials quinine, chloroquine, and their derivatives (Storz, 2004). On the other hands, Meldrum's acid and its derivatives serve as key intermediates for the synthesis of heterocyclic compounds with pharmacological activity (Gaber & McNab, 2001). In order to investigate the structure-function relationships and develop more effective antiparasitic drugs based on nitrogen heterocyclic derivatives (da Silva *et al.*, 2007; da Silva *et al.*, 2002), we report here the crystal structure of the title compound (I).

In (I), the 1,3-dioxane-4,6-dione ring is an envelope conformation with C13 in the flap position. The quinoline ring system, with the OH group, is nearly planar with maximum deviations from the mean plane of −0.0127 (9) Å for atom N1 and 0.0097 (9) Å for atom O3. The dihedral angle C11—C10—N2—C1 is −177.1 (1)° and the distances C1—N2 and C10—C11 indicate delocalization of the conjugated system. The amino H atom forms an intra- and an intermolecular contact to the carbonyl O4 atom. One intramolecular O—H···N and two intermolecular C—H···O hydrogen bonds were also observed. The packing diagram illustrates that two hydrogen-bonded molecules are related by an inversion centre building layers (Fig. 2). Details of the hydrogen-bonding parameters are given in Table 1.

## Experimental

The title compound was prepared according to a literature procedure (Cassis *et al.*, 1985). Single crystals of (I) suitable for X-ray data collection were obtained by slow evaporation from a solution of methanol:DCM (1:1). m.p.: 501–502 K.

## Refinement

The methyl H atoms were positioned with idealized geometry using a riding model with C—H = 0.96 Å. The other H atoms were located in difference map, and their positional parameters were refined freely [N—H = 0.85 (2) Å, O—H = 0.78 (2) Å and C—H = 0.87 (2)–1.00 (2) Å]. $U_{iso}$(H) values were set to 1.2$U_{eq}$ (1.5$U_{eq}$ for methyl) of the parent atom.

## Figures

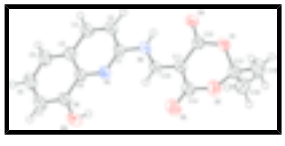


Fig. 1. Molecular structure of (I), showing the atom labeling and displacement ellipsoids drawn at the 50% probability level.

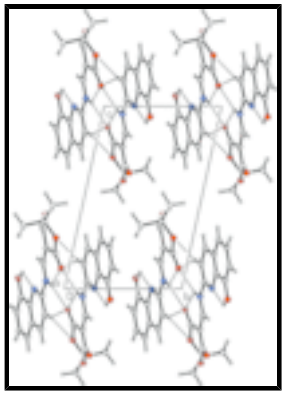


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

**5-[(8-Hydroxyquinolin-2-ylamino)methylene]-2,2-dimethyl-1,3-dioxane-4,6-dione**

*Crystal data*

| | |
|---|---|
| $C_{16}H_{14}N_2O_5$ | $Z = 2$ |
| $M_r = 314.29$ | $F_{000} = 328$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.379$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Cu $K\alpha$ radiation<br>$\lambda = 1.54180$ Å |
| $a = 7.445$ (2) Å | Cell parameters from 25 reflections |
| $b = 8.440$ (2) Å | $\theta = 5.6$–$25.2°$ |
| $c = 12.906$ (3) Å | $\mu = 0.88$ mm$^{-1}$ |
| $\alpha = 77.31$ (1)° | $T = 299$ (2) K |
| $\beta = 86.81$ (2)° | Prism, colourless |
| $\gamma = 73.15$ (1)° | 0.60 × 0.40 × 0.33 mm |
| $V = 757.2$ (3) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD4 diffractometer | $R_{int} = 0.053$ |
| Radiation source: fine-focus sealed tube | $\theta_{max} = 66.9°$ |
| Monochromator: graphite | $\theta_{min} = 3.5°$ |
| $T = 299(2)$ K | $h = -8 \rightarrow 8$ |
| $\omega/2\theta$ scans | $k = -10 \rightarrow 10$ |
| Absorption correction: none | $l = -15 \rightarrow 15$ |
| 5325 measured reflections | 3 standard reflections |
| 2704 independent reflections | every 120 min |
| 2436 reflections with $I > 2\sigma(I)$ | intensity decay: 1.0% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.120$

$S = 1.08$

2704 reflections

233 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.1625P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.27$ e Å$^{-3}$

$\Delta\rho_{min} = -0.18$ e Å$^{-3}$

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0292 (18)

*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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.87562 (18) | 0.18229 (16) | 1.03493 (11) | 0.0356 (3) |
| C2 | 0.8419 (2) | 0.11498 (19) | 1.14199 (12) | 0.0433 (4) |
| H2 | 0.742 (3) | 0.061 (2) | 1.1559 (13) | 0.052* |
| C3 | 0.9522 (2) | 0.1279 (2) | 1.21805 (12) | 0.0465 (4) |
| H3 | 0.936 (3) | 0.084 (2) | 1.2920 (15) | 0.056* |
| C4 | 1.0967 (2) | 0.20732 (18) | 1.18985 (11) | 0.0415 (3) |
| C5 | 1.2181 (3) | 0.2261 (2) | 1.26336 (14) | 0.0532 (4) |
| H5 | 1.203 (3) | 0.191 (2) | 1.3366 (16) | 0.064* |
| C6 | 1.3538 (2) | 0.3041 (2) | 1.22804 (15) | 0.0559 (4) |
| H6 | 1.442 (3) | 0.319 (2) | 1.2787 (16) | 0.067* |
| C7 | 1.3758 (2) | 0.3669 (2) | 1.11993 (14) | 0.0493 (4) |
| H7 | 1.471 (3) | 0.422 (2) | 1.0951 (14) | 0.059* |
| C8 | 1.26047 (19) | 0.34956 (17) | 1.04695 (12) | 0.0402 (3) |
| C9 | 1.11806 (18) | 0.26817 (16) | 1.08037 (11) | 0.0358 (3) |
| C10 | 0.7677 (2) | 0.23140 (17) | 0.85433 (11) | 0.0391 (3) |
| H10 | 0.855 (3) | 0.281 (2) | 0.8346 (13) | 0.047* |
| C11 | 0.65092 (19) | 0.22806 (17) | 0.77695 (11) | 0.0384 (3) |
| C12 | 0.50124 (19) | 0.14834 (17) | 0.80186 (10) | 0.0367 (3) |
| C13 | 0.3749 (2) | 0.2999 (2) | 0.62899 (11) | 0.0469 (4) |
| C14 | 0.6902 (2) | 0.2936 (2) | 0.66727 (12) | 0.0484 (4) |
| C15 | 0.2853 (3) | 0.2547 (3) | 0.54213 (13) | 0.0674 (5) |
| H15A | 0.2819 | 0.3378 | 0.4776 | 0.101* |

| | | | | |
|---|---|---|---|---|
| H15B | 0.1598 | 0.2525 | 0.5620 | 0.101* |
| H15C | 0.3572 | 0.1452 | 0.5314 | 0.101* |
| C16 | 0.2690 (3) | 0.4675 (2) | 0.65367 (14) | 0.0610 (5) |
| H16A | 0.3288 | 0.4863 | 0.7120 | 0.091* |
| H16B | 0.1424 | 0.4673 | 0.6724 | 0.091* |
| H16C | 0.2679 | 0.5561 | 0.5924 | 0.091* |
| N1 | 1.00832 (15) | 0.25491 (13) | 1.00330 (9) | 0.0360 (3) |
| N2 | 0.75720 (16) | 0.16946 (14) | 0.95745 (9) | 0.0384 (3) |
| H2N | 0.677 (3) | 0.116 (2) | 0.9798 (13) | 0.046* |
| O1 | 0.38313 (15) | 0.16567 (14) | 0.72143 (8) | 0.0478 (3) |
| O2 | 0.56184 (16) | 0.30024 (16) | 0.59423 (8) | 0.0546 (3) |
| O3 | 1.27855 (16) | 0.41060 (14) | 0.94136 (9) | 0.0502 (3) |
| H3O | 1.205 (3) | 0.386 (3) | 0.9114 (16) | 0.060* |
| O4 | 0.47832 (15) | 0.06465 (13) | 0.88781 (8) | 0.0445 (3) |
| O5 | 0.82846 (18) | 0.3362 (2) | 0.63562 (10) | 0.0747 (4) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0278 (6) | 0.0356 (7) | 0.0447 (7) | −0.0111 (5) | −0.0029 (5) | −0.0076 (5) |
| C2 | 0.0353 (7) | 0.0496 (8) | 0.0488 (8) | −0.0221 (6) | −0.0007 (6) | −0.0043 (6) |
| C3 | 0.0444 (8) | 0.0572 (9) | 0.0408 (8) | −0.0239 (7) | −0.0012 (6) | −0.0036 (6) |
| C4 | 0.0354 (7) | 0.0445 (7) | 0.0471 (8) | −0.0145 (6) | −0.0034 (6) | −0.0096 (6) |
| C5 | 0.0518 (9) | 0.0652 (10) | 0.0493 (9) | −0.0250 (8) | −0.0077 (7) | −0.0131 (7) |
| C6 | 0.0468 (9) | 0.0632 (10) | 0.0678 (11) | −0.0230 (8) | −0.0127 (8) | −0.0217 (8) |
| C7 | 0.0353 (8) | 0.0478 (8) | 0.0728 (11) | −0.0201 (6) | −0.0014 (7) | −0.0182 (7) |
| C8 | 0.0305 (7) | 0.0364 (7) | 0.0559 (8) | −0.0117 (5) | 0.0026 (6) | −0.0120 (6) |
| C9 | 0.0269 (6) | 0.0348 (6) | 0.0475 (8) | −0.0098 (5) | −0.0022 (5) | −0.0107 (5) |
| C10 | 0.0305 (7) | 0.0401 (7) | 0.0481 (8) | −0.0147 (6) | −0.0007 (6) | −0.0060 (6) |
| C11 | 0.0324 (7) | 0.0423 (7) | 0.0411 (7) | −0.0151 (6) | −0.0007 (5) | −0.0038 (6) |
| C12 | 0.0361 (7) | 0.0406 (7) | 0.0359 (7) | −0.0157 (5) | −0.0030 (5) | −0.0061 (5) |
| C13 | 0.0392 (8) | 0.0658 (9) | 0.0350 (7) | −0.0225 (7) | −0.0019 (6) | 0.0012 (6) |
| C14 | 0.0371 (8) | 0.0610 (9) | 0.0463 (8) | −0.0198 (7) | 0.0024 (6) | −0.0028 (7) |
| C15 | 0.0645 (11) | 0.1003 (14) | 0.0435 (9) | −0.0365 (11) | −0.0114 (8) | −0.0077 (9) |
| C16 | 0.0528 (10) | 0.0647 (10) | 0.0560 (10) | −0.0123 (8) | −0.0026 (8) | 0.0017 (8) |
| N1 | 0.0290 (6) | 0.0366 (6) | 0.0438 (6) | −0.0120 (4) | −0.0005 (5) | −0.0075 (5) |
| N2 | 0.0311 (6) | 0.0424 (6) | 0.0447 (7) | −0.0175 (5) | −0.0040 (5) | −0.0055 (5) |
| O1 | 0.0475 (6) | 0.0612 (6) | 0.0404 (6) | −0.0314 (5) | −0.0096 (4) | 0.0017 (4) |
| O2 | 0.0433 (6) | 0.0845 (8) | 0.0362 (6) | −0.0261 (6) | 0.0033 (4) | −0.0037 (5) |
| O3 | 0.0437 (6) | 0.0561 (6) | 0.0573 (7) | −0.0265 (5) | 0.0087 (5) | −0.0110 (5) |
| O4 | 0.0484 (6) | 0.0523 (6) | 0.0381 (5) | −0.0292 (5) | −0.0038 (4) | 0.0008 (4) |
| O5 | 0.0494 (7) | 0.1174 (11) | 0.0581 (7) | −0.0452 (7) | 0.0062 (6) | 0.0067 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.3095 (18) | C10—H10 | 0.867 (19) |
| C1—N2 | 1.4107 (18) | C11—C12 | 1.4483 (19) |
| C1—C2 | 1.412 (2) | C11—C14 | 1.449 (2) |
| C2—C3 | 1.356 (2) | C12—O4 | 1.2096 (17) |

| | | | |
|---|---|---|---|
| C2—H2 | 0.97 (2) | C12—O1 | 1.3537 (17) |
| C3—C4 | 1.416 (2) | C13—O2 | 1.4392 (18) |
| C3—H3 | 0.959 (18) | C13—O1 | 1.4437 (17) |
| C4—C5 | 1.409 (2) | C13—C16 | 1.499 (2) |
| C4—C9 | 1.410 (2) | C13—C15 | 1.505 (2) |
| C5—C6 | 1.366 (3) | C14—O5 | 1.208 (2) |
| C5—H5 | 0.94 (2) | C14—O2 | 1.360 (2) |
| C6—C7 | 1.398 (3) | C15—H15A | 0.9600 |
| C6—H6 | 1.00 (2) | C15—H15B | 0.9600 |
| C7—C8 | 1.367 (2) | C15—H15C | 0.9600 |
| C7—H7 | 0.96 (2) | C16—H16A | 0.9600 |
| C8—O3 | 1.3608 (19) | C16—H16B | 0.9600 |
| C8—C9 | 1.4224 (19) | C16—H16C | 0.9600 |
| C9—N1 | 1.3644 (18) | N2—H2N | 0.85 (2) |
| C10—N2 | 1.3255 (19) | O3—H3O | 0.79 (2) |
| C10—C11 | 1.370 (2) | | |
| N1—C1—N2 | 118.34 (12) | C12—C11—C14 | 119.95 (13) |
| N1—C1—C2 | 124.55 (13) | O4—C12—O1 | 118.43 (12) |
| N2—C1—C2 | 117.11 (12) | O4—C12—C11 | 124.72 (13) |
| C3—C2—C1 | 118.21 (13) | O1—C12—C11 | 116.81 (11) |
| C3—C2—H2 | 124.4 (10) | O2—C13—O1 | 109.88 (12) |
| C1—C2—H2 | 117.4 (10) | O2—C13—C16 | 110.65 (14) |
| C2—C3—C4 | 120.36 (14) | O1—C13—C16 | 110.18 (12) |
| C2—C3—H3 | 122.0 (12) | O2—C13—C15 | 106.47 (13) |
| C4—C3—H3 | 117.7 (12) | O1—C13—C15 | 106.14 (14) |
| C5—C4—C9 | 119.49 (14) | C16—C13—C15 | 113.36 (15) |
| C5—C4—C3 | 124.25 (14) | O5—C14—O2 | 118.07 (14) |
| C9—C4—C3 | 116.25 (13) | O5—C14—C11 | 125.82 (15) |
| C6—C5—C4 | 119.78 (16) | O2—C14—C11 | 116.05 (13) |
| C6—C5—H5 | 119.4 (14) | C13—C15—H15A | 109.5 |
| C4—C5—H5 | 120.7 (14) | C13—C15—H15B | 109.5 |
| C5—C6—C7 | 121.53 (15) | H15A—C15—H15B | 109.5 |
| C5—C6—H6 | 121.2 (12) | C13—C15—H15C | 109.5 |
| C7—C6—H6 | 117.3 (12) | H15A—C15—H15C | 109.5 |
| C8—C7—C6 | 119.85 (15) | H15B—C15—H15C | 109.5 |
| C8—C7—H7 | 118.6 (11) | C13—C16—H16A | 109.5 |
| C6—C7—H7 | 121.6 (11) | C13—C16—H16B | 109.5 |
| O3—C8—C7 | 120.85 (14) | H16A—C16—H16B | 109.5 |
| O3—C8—C9 | 118.76 (13) | C13—C16—H16C | 109.5 |
| C7—C8—C9 | 120.38 (14) | H16A—C16—H16C | 109.5 |
| N1—C9—C4 | 123.75 (12) | H16B—C16—H16C | 109.5 |
| N1—C9—C8 | 117.29 (12) | C1—N1—C9 | 116.85 (12) |
| C4—C9—C8 | 118.96 (13) | C10—N2—C1 | 124.05 (13) |
| N2—C10—C11 | 125.99 (14) | C10—N2—H2N | 119.6 (11) |
| N2—C10—H10 | 116.6 (11) | C1—N2—H2N | 116.4 (12) |
| C11—C10—H10 | 117.4 (11) | C12—O1—C13 | 118.49 (11) |
| C10—C11—C12 | 121.53 (13) | C14—O2—C13 | 118.71 (11) |
| C10—C11—C14 | 118.32 (13) | C8—O3—H3O | 107.0 (15) |

| | | | |
|---|---|---|---|
| N1—C1—C2—C3 | 1.0 (2) | C10—C11—C12—O1 | 174.03 (12) |
| N2—C1—C2—C3 | −179.08 (13) | C14—C11—C12—O1 | −11.3 (2) |
| C1—C2—C3—C4 | 0.1 (2) | C10—C11—C14—O5 | 8.0 (3) |
| C2—C3—C4—C5 | −179.85 (15) | C12—C11—C14—O5 | −166.78 (17) |
| C2—C3—C4—C9 | −0.7 (2) | C10—C11—C14—O2 | −174.86 (13) |
| C9—C4—C5—C6 | 0.6 (2) | C12—C11—C14—O2 | 10.3 (2) |
| C3—C4—C5—C6 | 179.72 (16) | N2—C1—N1—C9 | 178.78 (10) |
| C4—C5—C6—C7 | 0.2 (3) | C2—C1—N1—C9 | −1.3 (2) |
| C5—C6—C7—C8 | −0.5 (3) | C4—C9—N1—C1 | 0.58 (19) |
| C6—C7—C8—O3 | 179.61 (13) | C8—C9—N1—C1 | −178.91 (11) |
| C6—C7—C8—C9 | 0.1 (2) | C11—C10—N2—C1 | −177.11 (13) |
| C5—C4—C9—N1 | 179.58 (13) | N1—C1—N2—C10 | −2.3 (2) |
| C3—C4—C9—N1 | 0.4 (2) | C2—C1—N2—C10 | 177.76 (12) |
| C5—C4—C9—C8 | −0.9 (2) | O4—C12—O1—C13 | 163.07 (13) |
| C3—C4—C9—C8 | 179.84 (12) | C11—C12—O1—C13 | −18.90 (18) |
| O3—C8—C9—N1 | 0.60 (18) | O2—C13—O1—C12 | 47.43 (17) |
| C7—C8—C9—N1 | −179.88 (12) | C16—C13—O1—C12 | −74.74 (17) |
| O3—C8—C9—C4 | −178.92 (11) | C15—C13—O1—C12 | 162.17 (13) |
| C7—C8—C9—C4 | 0.6 (2) | O5—C14—O2—C13 | −161.71 (16) |
| N2—C10—C11—C12 | −1.8 (2) | C11—C14—O2—C13 | 21.0 (2) |
| N2—C10—C11—C14 | −176.57 (13) | O1—C13—O2—C14 | −48.65 (18) |
| C10—C11—C12—O4 | −8.1 (2) | C16—C13—O2—C14 | 73.23 (17) |
| C14—C11—C12—O4 | 166.58 (13) | C15—C13—O2—C14 | −163.18 (14) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2N···O4 | 0.85 (2) | 2.140 (19) | 2.7428 (17) | 127.6 (14) |
| N2—H2N···O4$^{i}$ | 0.85 (2) | 2.498 (18) | 3.2943 (17) | 156.2 (15) |
| O3—H3O···N1 | 0.79 (2) | 2.22 (2) | 2.7028 (16) | 120.0 (18) |
| C2—H2···O1$^{i}$ | 0.97 (2) | 2.564 (19) | 3.4151 (19) | 146.6 (14) |
| C2—H2···O4$^{i}$ | 0.97 (2) | 2.35 (2) | 3.2533 (19) | 155.4 (14) |

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

**Fig. 1**

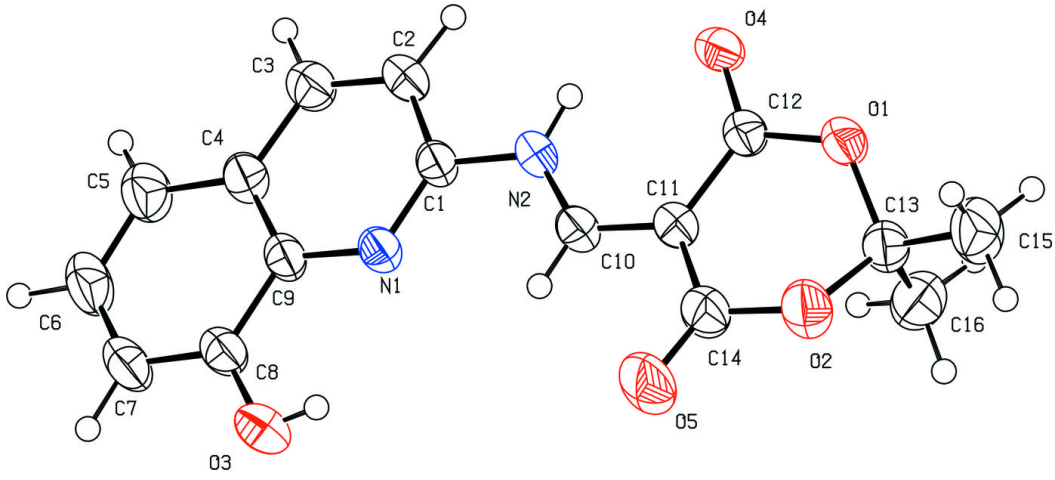

O4
C2
C3
C12
O1
C4
C5
C1
N2
C11
C13
C15
N1
C10
C9
C6
C16
C14
O2
C8
C7
O5
O3

**Fig. 2**