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

# 1-(4-Aminophenyl)-2-ethyl-3-hydroxy-1,4-dihydropyridin-4-one monohydrate

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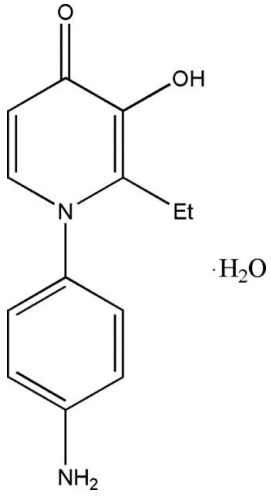
Received 9 April 2007; accepted 12 April 2007

Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.043; $wR$ factor = 0.125; data-to-parameter ratio = 12.6.

In the title compound, $C_{13}H_{14}N_2O_2\cdot H_2O$, the dihedral angle between the benzene and pyridinone rings is 74.86 (7)°. In the crystal structure, the molecules are linked by a pair of O—H···O hydrogen bonds [O···O = 2.662 (2) Å], forming a centrosymmetric dimer. In addition, the molecules are linked *via* O—H···O, O—H···N and N—H···O intermolecular hydrogen bonds involving the water molecules, and C—H···O interactions.

## Related literature

O—H···O hydrogen-bonded dimer formation is observed in 3-hydroxypyridin-4-one and 3-hydroxypyran-4-one compounds (Xiao *et al.*, 1992; Burgess *et al.*, 1993; Burgess, Fawcett, Russell & Zaisheng, 1998; Burgess, Fawcett, Russell & Waltham, 1998; Brown *et al.*, 1995). For related literature, see: Burgess (1993); Burgess *et al.* (1996); Chan *et al.* (1992); Clevette & Orvig (1990); Lu *et al.* (2003).




## Experimental

### Crystal data

$C_{13}H_{14}N_2O_2\cdot H_2O$
$M_r$ = 248.28
Triclinic, $P\overline{1}$
$a$ = 7.8992 (14) Å
$b$ = 8.0162 (15) Å
$c$ = 11.4759 (19) Å
$\alpha$ = 96.835 (2)°
$\beta$ = 106.273 (2)°
$\gamma$ = 113.143 (3)°
$V$ = 619.43 (19) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 298 (2) K
0.58 × 0.47 × 0.22 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*: Sheldrick, 1996)
$T_{min}$ = 0.947, $T_{max}$ = 0.979
3228 measured reflections
2155 independent reflections
1494 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.019

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.125
$S$ = 1.03
2155 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H15···N2$^{i}$ | 0.85 | 2.17 | 2.987 (3) | 160 |
| O3—H14···O1$^{ii}$ | 0.85 | 2.01 | 2.857 (2) | 174 |
| O2—H2···O1 | 0.82 | 2.34 | 2.7630 (19) | 113 |
| O2—H2···O1$^{iii}$ | 0.82 | 2.01 | 2.662 (2) | 136 |
| N2—H2*B*···O3 | 0.92 (3) | 2.15 (3) | 3.053 (3) | 170 (2) |
| C11—H11···O1$^{iv}$ | 0.93 | 2.45 | 3.324 (3) | 157 |

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x, y+1, z+1$; (iii) $-x+1, -y, -z$; (iv) $-x+1, -y+1, -z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Natural Science Foundation of Jiangsu Education Department (grant No. 02KJB150007)

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2360).

## References

Brown, S. D., Burgess, J., Fawcett, J., Parsons, S. A., Russell, D. R. & Waltham, E. (1995). *Acta Cryst.* C**51**, 1335–1338.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burgess, J. (1993). *Transition Met. Chem.* **18**, 439–448.
Burgess, J., Fawcett, J., Parsons, S. A., Patel, M. S. & Russell, D. R. (1993). *J. Chem. Res. S.*, pp. 50–51.
Burgess, J., Fawcett, J., Russell, D. R., Hider, R. C., Hossain, M. B., Stoner, C. R. & van der Helm, D. (1996). *Acta Cryst.* C**52**, 2917–2920.

  doi:10.1107/S1600536807018284 

Burgess, J., Fawcett, J., Russell, D. R. & Waltham, E. (1998). *Acta Cryst.* C**54**, 2011–2015.
Burgess, J., Fawcett, J., Russell, D. R. & Zaisheng, L. (1998). *Acta Cryst.* C**54**, 430–433.
Chan, H.-K., Ghosh, S., Venkataram, S., Rahman, Y.-E. & Grant, D. J. W. (1992). *J. Pharm. Sci.* **81**, 353–358.
Clevette, D. J. & Orvig, C. (1990). *Polyhedron*, **9**, 151–161.
Lu, Z. S., Ni, Z. P., Niu, D. Z. & Tu, S. J. (2003). *Chin. J. Org. Chem.* **23**, 89–91.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xiao, G. Y., van der Helm, D., Hider, R. C. & Dobbin, P. S. (1992). *J. Chem. Soc. Dalton Trans.* pp. 3265–3271.

# supplementary materials

*Acta Cryst.* (2007). E**63**, o2614-o2615 [ **doi:10.1107/S1600536807018284** ]

# 1-(4-Aminophenyl)-2-ethyl-3-hydroxy-1,4-dihydropyridin-4-one monohydrate

**Z.-S. Lu, X.-F. Xia, F. Gao, C.-S. Yao and D.-Z. Niu**

## Comment

A number of hydroxypyrones and hydroxypyridinones are being assessed or considered as orally effective chelators for treatment iron or aluminum overload (Burgess, 1993; Clevette *et al.*, 1990). In our continuing research on the hydroxypyridinone (Lu *et al.*, 2003), we present here the crystal structure of the title compound, (I).

Bond distances (Table 1) in compound (I) agree with those observed for the *N*-benzyl analogues (Burgess, Fawcett, Russell & Zaisheng, 1998;

Burgess, Fawcett, Russell & Waltham, 1998). The dihedral angle between the benzene and pyridinone rings is 74.86 (7)°.

Molecules of compound (I) are linked by a pair of O2—H2···O1(1-x, -y, -z) hydrogen bonds into a centrosymmetric dimer (Fig. 1). This type of dimeric structure is commonly found in anhydrous or hydrous 3-hydroxypyridin-4-one (Xiao *et al.*, 1992; Burgess *et al.*, 1993; Burgess, Fawcett, Russell & Zaisheng, 1998; Burgess, Fawcett, Russell & Waltham, 1998 and 3-hydroxypyran-4-one compounds (Brown *et al.*, 1995). In the crystal structure, there is a water molecule associated with each pyridinone molecule, which are linked through N2—H2B···O3 hydrogen bonds (Fig. 1). The water molecule links the O1 and N2 atoms of adjacent pyridinones through O—H···O and O—H···O hydrogen bonds, with O3···O1(x, 1+y, 1+z) and O3···N2(-x, 2-y, 1-z) distances of 2.857 (2) and 2.987 (3) Å, respectively. In addition, the molecules are linked by weak C—H···O contacts of ca C···O 3.3 Å, as previously observed for this type of compounds (Burgess *et al.*, 1996; Burgess, Fawcett, Russell & Zaisheng, 1998; Burgess, Fawcett, Russell & Waltham, 1998).

## Experimental

The title compound was prepared according to the literature method of Lu *et al.* (2003). Crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol-water solution at room temperature after several weeks.

## Refinement

H atoms of amino group were located in a difference map and refined freely. The remaining H atoms were placed in calculated positions, with C—H = 0.93-0.97 Å and O—H = 0.82-0.85 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(O)$.

## Figures

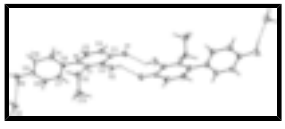


Fig. 1. A view of the hydrogen-bonded (dashed lines) dimer of (I). Displacement ellipsoids are drawn at the 30% probability level.

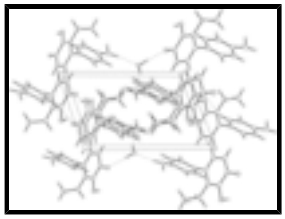


Fig. 2. The packing diagram of (I). Hydrogen bonds are shown as dashed lines.

### 1-(4-Aminophenyl)-2-ethyl-3-hydroxy-1,4-dihydropyridin-4-one monohydrate

*Crystal data*

| | |
|---|---|
| $C_{13}H_{14}N_2O_2{\cdot}H_2O$ | $Z = 2$ |
| $M_r = 248.28$ | $F_{000} = 264$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.331$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 7.8992$ (14) Å | Cell parameters from 1277 reflections |
| $b = 8.0162$ (15) Å | $\theta = 2.9$–$27.8^\circ$ |
| $c = 11.4759$ (19) Å | $\mu = 0.10$ mm$^{-1}$ |
| $\alpha = 96.835$ (2)° | $T = 298$ (2) K |
| $\beta = 106.273$ (2)° | Plate, yellow |
| $\gamma = 113.143$ (3)° | 0.58 × 0.47 × 0.22 mm |
| $V = 619.43$ (19) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 2155 independent reflections |
| Radiation source: fine-focus sealed tube | 1494 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.019$ |
| $T = 298$(2) K | $\theta_{max} = 25.0^\circ$ |
| φ and ω scans | $\theta_{min} = 1.9^\circ$ |
| Absorption correction: multi-scan (SADABS: Sheldrick, 1996) | $h = -9\rightarrow9$ |
| $T_{min} = 0.947$, $T_{max} = 0.979$ | $k = -9\rightarrow6$ |
| 3228 measured reflections | $l = -12\rightarrow13$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H atoms treated by a mixture of independent and constrained refinement |
| Least-squares matrix: full | $w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.141P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | $(\Delta/\sigma)_{max} = 0.001$ |
| $wR(F^2) = 0.125$ | $\Delta\rho_{max} = 0.15$ e Å$^{-3}$ |
| $S = 1.03$ | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |
| 2155 reflections | Extinction correction: none |

171 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

*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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.2932 (2) | 0.4626 (2) | 0.13373 (15) | 0.0410 (4) |
| N2 | 0.2456 (4) | 1.0252 (4) | 0.4479 (2) | 0.0598 (6) |
| O1 | 0.3004 (2) | 0.02104 (19) | −0.07892 (12) | 0.0487 (4) |
| O2 | 0.6375 (2) | 0.2808 (2) | 0.11625 (14) | 0.0555 (4) |
| H2 | 0.6220 | 0.1884 | 0.0671 | 0.083* |
| O3 | 0.0972 (2) | 0.8939 (2) | 0.65506 (14) | 0.0663 (5) |
| H14 | 0.1591 | 0.9252 | 0.7339 | 0.100* |
| H15 | −0.0016 | 0.9169 | 0.6437 | 0.100* |
| C1 | 0.4659 (3) | 0.4445 (3) | 0.15894 (17) | 0.0376 (5) |
| C2 | 0.4666 (3) | 0.2958 (3) | 0.08741 (17) | 0.0392 (5) |
| C3 | 0.2953 (3) | 0.1587 (3) | −0.01476 (17) | 0.0386 (5) |
| C4 | 0.1269 (3) | 0.1900 (3) | −0.03696 (19) | 0.0465 (5) |
| H4 | 0.0120 | 0.1085 | −0.1040 | 0.056* |
| C5 | 0.1280 (3) | 0.3360 (3) | 0.0369 (2) | 0.0476 (5) |
| H5 | 0.0128 | 0.3498 | 0.0209 | 0.057* |
| C6 | 0.2799 (3) | 0.6082 (3) | 0.21302 (18) | 0.0390 (5) |
| C7 | 0.1834 (3) | 0.5618 (3) | 0.2967 (2) | 0.0485 (5) |
| H7 | 0.1250 | 0.4377 | 0.3004 | 0.058* |
| C8 | 0.1741 (3) | 0.6998 (3) | 0.3745 (2) | 0.0502 (5) |
| H8 | 0.1093 | 0.6679 | 0.4307 | 0.060* |
| C9 | 0.2597 (3) | 0.8856 (3) | 0.37046 (18) | 0.0444 (5) |
| C10 | 0.3514 (3) | 0.9284 (3) | 0.28396 (19) | 0.0465 (5) |
| H10 | 0.4068 | 1.0518 | 0.2784 | 0.056* |
| C11 | 0.3620 (3) | 0.7914 (3) | 0.20597 (18) | 0.0430 (5) |
| H11 | 0.4246 | 0.8226 | 0.1486 | 0.052* |
| C12 | 0.6474 (3) | 0.5818 (3) | 0.26744 (18) | 0.0449 (5) |
| H12A | 0.6434 | 0.7017 | 0.2829 | 0.054* |

| | | | | |
|---|---|---|---|---|
| H12B | 0.7629 | 0.6010 | 0.2461 | 0.054* |
| C13 | 0.6645 (4) | 0.5139 (4) | 0.3857 (2) | 0.0671 (7) |
| H13A | 0.7824 | 0.6047 | 0.4528 | 0.101* |
| H13B | 0.6702 | 0.3961 | 0.3711 | 0.101* |
| H13C | 0.5519 | 0.4978 | 0.4083 | 0.101* |
| H2A | 0.346 (5) | 1.136 (5) | 0.464 (3) | 0.091 (11)* |
| H2B | 0.214 (4) | 0.983 (4) | 0.513 (2) | 0.071 (8)* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0404 (10) | 0.0359 (9) | 0.0441 (9) | 0.0184 (8) | 0.0109 (8) | 0.0073 (7) |
| N2 | 0.0670 (15) | 0.0683 (15) | 0.0520 (12) | 0.0413 (13) | 0.0205 (11) | 0.0054 (11) |
| O1 | 0.0524 (9) | 0.0468 (9) | 0.0433 (8) | 0.0258 (7) | 0.0108 (7) | 0.0023 (7) |
| O2 | 0.0439 (9) | 0.0527 (9) | 0.0588 (9) | 0.0254 (7) | 0.0058 (7) | −0.0070 (7) |
| O3 | 0.0696 (11) | 0.0737 (11) | 0.0486 (9) | 0.0372 (9) | 0.0089 (8) | 0.0027 (8) |
| C1 | 0.0385 (11) | 0.0363 (11) | 0.0388 (10) | 0.0174 (9) | 0.0128 (8) | 0.0121 (8) |
| C2 | 0.0397 (11) | 0.0417 (11) | 0.0391 (10) | 0.0208 (9) | 0.0130 (9) | 0.0138 (9) |
| C3 | 0.0457 (11) | 0.0361 (11) | 0.0327 (10) | 0.0177 (9) | 0.0126 (9) | 0.0095 (8) |
| C4 | 0.0424 (12) | 0.0417 (12) | 0.0438 (11) | 0.0173 (9) | 0.0031 (9) | 0.0060 (9) |
| C5 | 0.0397 (11) | 0.0442 (12) | 0.0537 (12) | 0.0218 (10) | 0.0067 (10) | 0.0081 (10) |
| C6 | 0.0404 (11) | 0.0385 (11) | 0.0413 (10) | 0.0210 (9) | 0.0144 (9) | 0.0100 (9) |
| C7 | 0.0477 (12) | 0.0453 (12) | 0.0602 (13) | 0.0230 (10) | 0.0234 (11) | 0.0223 (11) |
| C8 | 0.0527 (13) | 0.0649 (15) | 0.0512 (12) | 0.0349 (12) | 0.0281 (10) | 0.0239 (11) |
| C9 | 0.0427 (11) | 0.0537 (13) | 0.0405 (11) | 0.0309 (10) | 0.0080 (9) | 0.0086 (9) |
| C10 | 0.0498 (12) | 0.0389 (11) | 0.0525 (12) | 0.0213 (10) | 0.0185 (10) | 0.0119 (10) |
| C11 | 0.0472 (12) | 0.0416 (12) | 0.0436 (11) | 0.0201 (10) | 0.0199 (9) | 0.0139 (9) |
| C12 | 0.0424 (11) | 0.0401 (11) | 0.0478 (12) | 0.0197 (9) | 0.0109 (9) | 0.0049 (9) |
| C13 | 0.0633 (15) | 0.0707 (17) | 0.0441 (13) | 0.0200 (13) | 0.0051 (11) | 0.0052 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N1—C5 | 1.358 (2) | C5—H5 | 0.93 |
| N1—C1 | 1.382 (2) | C6—C11 | 1.376 (3) |
| N1—C6 | 1.449 (2) | C6—C7 | 1.384 (3) |
| N2—C9 | 1.404 (3) | C7—C8 | 1.375 (3) |
| N2—H2A | 0.88 (3) | C7—H7 | 0.93 |
| N2—H2B | 0.92 (3) | C8—C9 | 1.386 (3) |
| O1—C3 | 1.273 (2) | C8—H8 | 0.93 |
| O2—C2 | 1.354 (2) | C9—C10 | 1.384 (3) |
| O2—H2 | 0.82 | C10—C11 | 1.376 (3) |
| O3—H14 | 0.85 | C10—H10 | 0.93 |
| O3—H15 | 0.85 | C11—H11 | 0.93 |
| C1—C2 | 1.367 (3) | C12—C13 | 1.512 (3) |
| C1—C12 | 1.500 (3) | C12—H12A | 0.97 |
| C2—C3 | 1.431 (3) | C12—H12B | 0.97 |
| C3—C4 | 1.409 (3) | C13—H13A | 0.96 |
| C4—C5 | 1.356 (3) | C13—H13B | 0.96 |
| C4—H4 | 0.93 | C13—H13C | 0.96 |

| | | | |
|---|---|---|---|
| C5—N1—C1 | 120.00 (17) | C8—C7—H7 | 120.1 |
| C5—N1—C6 | 118.45 (16) | C6—C7—H7 | 120.1 |
| C1—N1—C6 | 121.45 (16) | C7—C8—C9 | 121.07 (19) |
| C9—N2—H2A | 111.3 (19) | C7—C8—H8 | 119.5 |
| C9—N2—H2B | 109.7 (16) | C9—C8—H8 | 119.5 |
| H2A—N2—H2B | 119 (3) | C10—C9—C8 | 118.19 (19) |
| C2—O2—H2 | 109.5 | C10—C9—N2 | 120.9 (2) |
| H14—O3—H15 | 107.1 | C8—C9—N2 | 120.8 (2) |
| C2—C1—N1 | 119.21 (17) | C11—C10—C9 | 121.2 (2) |
| C2—C1—C12 | 120.64 (17) | C11—C10—H10 | 119.4 |
| N1—C1—C12 | 120.09 (17) | C9—C10—H10 | 119.4 |
| O2—C2—C1 | 117.57 (17) | C6—C11—C10 | 119.89 (18) |
| O2—C2—C3 | 119.97 (17) | C6—C11—H11 | 120.1 |
| C1—C2—C3 | 122.46 (18) | C10—C11—H11 | 120.1 |
| O1—C3—C4 | 124.17 (18) | C1—C12—C13 | 111.82 (17) |
| O1—C3—C2 | 120.92 (18) | C1—C12—H12A | 109.3 |
| C4—C3—C2 | 114.92 (17) | C13—C12—H12A | 109.3 |
| C5—C4—C3 | 121.69 (19) | C1—C12—H12B | 109.3 |
| C5—C4—H4 | 119.2 | C13—C12—H12B | 109.3 |
| C3—C4—H4 | 119.2 | H12A—C12—H12B | 107.9 |
| C4—C5—N1 | 121.67 (19) | C12—C13—H13A | 109.5 |
| C4—C5—H5 | 119.2 | C12—C13—H13B | 109.5 |
| N1—C5—H5 | 119.2 | H13A—C13—H13B | 109.5 |
| C11—C6—C7 | 119.82 (19) | C12—C13—H13C | 109.5 |
| C11—C6—N1 | 120.78 (17) | H13A—C13—H13C | 109.5 |
| C7—C6—N1 | 119.41 (18) | H13B—C13—H13C | 109.5 |
| C8—C7—C6 | 119.8 (2) | | |
| C5—N1—C1—C2 | −1.6 (3) | C5—N1—C6—C11 | −107.4 (2) |
| C6—N1—C1—C2 | 174.75 (17) | C1—N1—C6—C11 | 76.2 (2) |
| C5—N1—C1—C12 | −178.74 (17) | C5—N1—C6—C7 | 72.6 (2) |
| C6—N1—C1—C12 | −2.4 (3) | C1—N1—C6—C7 | −103.8 (2) |
| N1—C1—C2—O2 | −178.87 (16) | C11—C6—C7—C8 | −1.5 (3) |
| C12—C1—C2—O2 | −1.7 (3) | N1—C6—C7—C8 | 178.45 (18) |
| N1—C1—C2—C3 | 1.1 (3) | C6—C7—C8—C9 | 0.1 (3) |
| C12—C1—C2—C3 | 178.27 (17) | C7—C8—C9—C10 | 1.4 (3) |
| O2—C2—C3—O1 | 0.6 (3) | C7—C8—C9—N2 | 178.5 (2) |
| C1—C2—C3—O1 | −179.44 (18) | C8—C9—C10—C11 | −1.6 (3) |
| O2—C2—C3—C4 | −179.23 (17) | N2—C9—C10—C11 | −178.6 (2) |
| C1—C2—C3—C4 | 0.8 (3) | C7—C6—C11—C10 | 1.3 (3) |
| O1—C3—C4—C5 | 177.90 (19) | N1—C6—C11—C10 | −178.65 (18) |
| C2—C3—C4—C5 | −2.3 (3) | C9—C10—C11—C6 | 0.2 (3) |
| C3—C4—C5—N1 | 2.0 (3) | C2—C1—C12—C13 | −84.4 (2) |
| C1—N1—C5—C4 | 0.1 (3) | N1—C1—C12—C13 | 92.7 (2) |
| C6—N1—C5—C4 | −176.39 (18) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| O3—H15···N2$^{i}$ | 0.85 | 2.17 | 2.987 (3) | 160 |
| O3—H14···O1$^{ii}$ | 0.85 | 2.01 | 2.857 (2) | 174 |
| O2—H2···O1 | 0.82 | 2.34 | 2.7630 (19) | 113 |
| O2—H2···O1$^{iii}$ | 0.82 | 2.01 | 2.662 (2) | 136 |
| N2—H2B···O3 | 0.92 (3) | 2.15 (3) | 3.053 (3) | 170 (2) |
| C11—H11···O1$^{iv}$ | 0.93 | 2.45 | 3.324 (3) | 157 |

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

**Fig. 1**

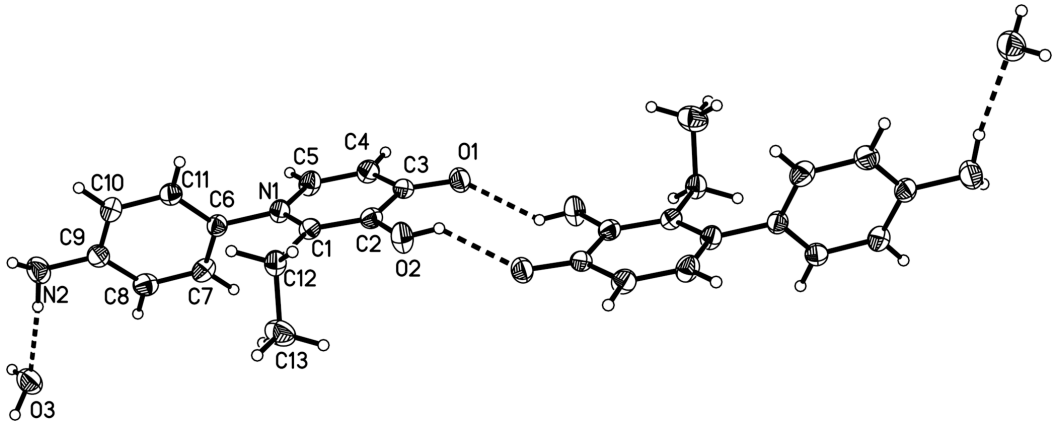


**Fig. 2**