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# N-[3-(Dimethylamino)propyl]-N'-(2-hydroxy-5-methylphenyl)oxamide

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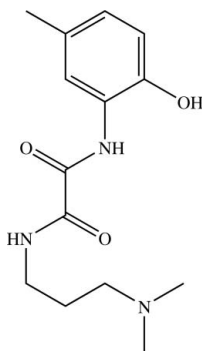
Received 19 February 2012; accepted 22 February 2012

 Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.053;  $wR$  factor = 0.151; data-to-parameter ratio = 15.8.

In the title compound,  $\text{C}_{14}\text{H}_{21}\text{N}_3\text{O}_3$ , the oxamide group has a *transoid* conformation. In the crystal, the molecules are connected by  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bonds into a double chain running along the  $b$  axis.

## Related literature

For the use of  $N,N'$ -bis(substituted)oxamides in the synthesis of nuclear complexes, see: Ojima & Nonoyama (1988); Ruiz *et al.* (1999). For related compounds, see: Han *et al.* (2007); Martinez *et al.* (1998); Yue *et al.* (2012).



## Experimental

## Crystal data

$\text{C}_{14}\text{H}_{21}\text{N}_3\text{O}_3$   
 $M_r = 279.34$   
 Monoclinic,  $P2_1/c$

$a = 11.542$  (2) Å  
 $b = 10.304$  (2) Å  
 $c = 13.860$  (3) Å

$\beta = 109.16$  (3)°  
 $V = 1557.2$  (5) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.09$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.27 \times 0.24 \times 0.17$  mm

## Data collection

Bruker APEX diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.977$ ,  $T_{\max} = 0.986$   
 7686 measured reflections

3099 independent reflections  
 2093 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$   
 Standard reflections: 0

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$   
 $wR(F^2) = 0.151$   
 $S = 1.02$   
 3099 reflections  
 196 parameters  
 1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.26$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                                     | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{N2}-\text{H2}\cdots\text{O2}^i$            | 0.92 (3) | 2.13 (3)    | 2.945 (2)   | 147 (2)       |
| $\text{O1}-\text{H1A}\cdots\text{N3}^{\text{ii}}$ | 0.90 (2) | 1.76 (2)    | 2.654 (2)   | 168 (3)       |

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: XP (Siemens, 1994); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5821).

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 Yue, X.-T., Li, X.-W. & Wu, Z.-Y. (2012). *Acta Cryst.* **E68**, o8.

## supporting information

*Acta Cryst.* (2012). E68, o895 [doi:10.1107/S1600536812007957]

***N*-[3-(Dimethylamino)propyl]-*N'*-(2-hydroxy-5-methylphenyl)oxamide****Yong-Jun Zheng, Kang Zheng, Zhi-Yong Wu and Yantuan Li****S1. Comment**

*N,N'*-bis(substituted)oxamides as multiatom bridging ligands has played an important role in designing and synthesizing polymetallic systems (Ojima & Nonoyama, 1988; Ruiz *et al.*, 1999). Due to the difficulties of synthesis only few dissymmetrical bis-substituted-oxamide ligand has been reported. In order to provide more examples of such ligand, quite recently, we reported the structure of (3-{[*N*-(5-Chloro-2-hydroxyphenyl)oxamoyl]amino}propyl)dimethylazanium perchlorate (Yue *et al.*, 2012). In continuation of our earlier work, the title compound was synthesized and its structure is reported here.

As shown in Fig. 1, the title compound has a *trans*-conformation of the oxamide group. The whole compound looks like the alphabet 'L'. The benzene ring is almost coplanar to the oxamide group [11.44 (9)°], just like that in the compound of *N,N'*-bis(2-Hydroxyphenyl)oxamide (Martinez *et al.*, 1998). However, the plane through the other substituent group, aminopropyl, is perpendicular to the oxamide plane [83.49 (12)°]. The torsion angle of C9—N2—C10—C11 is 106.7 (2)° (Table 1). And the conformation for the C10—C11 bond is *gauche*. While in the compound 2-(*N'*-[3-(Dimethylammonio)propyl]oxamido]benzoate (Han *et al.*, 2007), the corresponding angle is 151.3 (3)° and the conformation is *anti*.

A centrosymmetric dimer of a pair of the compounds is formed *via* the hydrogen bonds of the oxamide groups (Fig. 2, Table 2). These dimers are further assembled to a chain parallel to the *b*-axis through the hydrogen bonds between the phenolic hydroxyl groups and the tertiary amino groups.

**S2. Experimental**

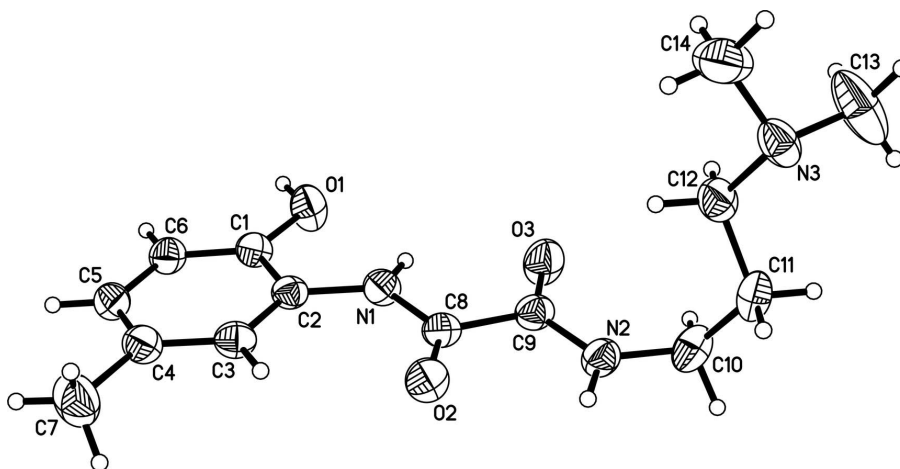
All reagents were of AR grade and obtained commercially without further purification. The title compound was prepared according to the method proposed by Han *et al.* (2007). A tetrahydrofuran (THF) solution (8 ml) of ethyl oxalyl chloride (1.11 ml, 10 mmol) was added dropwise into a THF solution (50 ml) of 2-amino-4-methylphenol (1.23 g, 10 mmol) with continuous stirring. The mixture was stirred quickly for 1 h and became clear. Then 20 ml ethanol was further added and the mixture was added dropwise into the solution (10 ml) of 3-dimethylamino-propylamine (1.02 g, 10 mmol) with stirring and kept the temperature at 273 K for 9 h. The title compound was precipitated as a white powder and washed with ethanol for several times and dried under vacuum. Yield: 1.83 g (66%). Colourless crystals of compound with the suitable size for X-ray analysis were obtained from an ethanol/water (1:1) mixture by slow evaporation for one week at room temperature.

Anal. Calcd. for C<sub>14</sub>H<sub>21</sub>N<sub>3</sub>O<sub>3</sub> (%): C, 60.20; H, 7.58; N, 15.04. Found: C, 60.33; H, 7.65; N, 15.00.

**S3. Refinement**

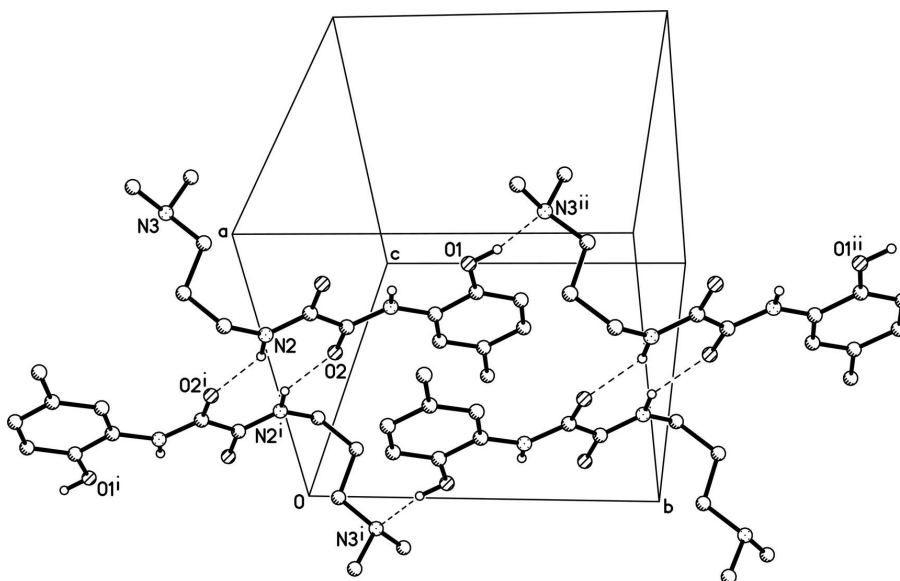
All H atoms were found in a difference Fourier map. Those bonded to N and O were freely refined with the O1—H1A bond restrained to a length of 0.86 (2) Å. Other H atoms were placed in calculated positions, with C—H = 0.93

(aromatic), 0.97 (methylene) and 0.96 (methyl), and refined using a riding model with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}\text{C}$  or  $1.5U_{\text{eq}}\text{C}(\text{methyl})$ .



**Figure 1**

The molecular structure of the title compound. The displacement ellipsoids are drawn at the 30% probability levels and H atoms are shown as small spheres of arbitrary radii.



**Figure 2**

A view of a hydrogen bonding chain extending along the  $b$ -axis. [Symmetry codes:  $i = -x + 1, -y, -z$ ;  $ii = x, y + 1, z$ .]

### *N*-[3-(Dimethylamino)propyl]-*N'*-(2-hydroxy-5-methylphenyl)ethanediamide

#### Crystal data

$\text{C}_{14}\text{H}_{21}\text{N}_3\text{O}_3$

$M_r = 279.34$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 11.542(2)\ \text{\AA}$

$b = 10.304(2)\ \text{\AA}$

$c = 13.860(3)\ \text{\AA}$

$\beta = 109.16(3)^\circ$

$V = 1557.2(5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 600$

$D_x = 1.191\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2107 reflections

$\theta = 2.5\text{--}23.3^\circ$   
 $\mu = 0.09\text{ mm}^{-1}$   
 $T = 296\text{ K}$

Block, colourless  
 $0.27 \times 0.24 \times 0.17\text{ mm}$

*Data collection*

Bruker APEX  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.977$ ,  $T_{\max} = 0.986$

7686 measured reflections  
 3099 independent reflections  
 2093 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$   
 $\theta_{\max} = 26.1^\circ$ ,  $\theta_{\min} = 2.5^\circ$   
 $h = -6 \rightarrow 14$   
 $k = -12 \rightarrow 12$   
 $l = -17 \rightarrow 12$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.053$   
 $wR(F^2) = 0.151$   
 $S = 1.02$   
 3099 reflections  
 196 parameters  
 1 restraint  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.3954P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.30\text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.26\text{ e \AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|---------------|----------------------------------|
| O1  | 0.78563 (15) | 0.54467 (14)  | 0.12601 (15)  | 0.0756 (5)                       |
| O2  | 0.52711 (14) | 0.16017 (13)  | 0.05547 (12)  | 0.0675 (4)                       |
| O3  | 0.81264 (15) | 0.18892 (16)  | 0.01920 (14)  | 0.0812 (5)                       |
| N1  | 0.66500 (17) | 0.32727 (16)  | 0.08912 (13)  | 0.0551 (4)                       |
| N2  | 0.67544 (19) | 0.02499 (17)  | -0.02835 (13) | 0.0606 (5)                       |
| N3  | 0.92876 (16) | -0.24706 (17) | 0.16828 (17)  | 0.0720 (6)                       |
| C1  | 0.67333 (18) | 0.54785 (18)  | 0.13783 (15)  | 0.0535 (5)                       |
| C2  | 0.60784 (17) | 0.43098 (17)  | 0.12124 (14)  | 0.0491 (5)                       |
| C3  | 0.49425 (19) | 0.4244 (2)    | 0.13394 (15)  | 0.0574 (5)                       |
| H3A | 0.4521       | 0.3459        | 0.1235        | 0.069*                           |
| C4  | 0.4419 (2)   | 0.5333 (2)    | 0.16214 (16)  | 0.0623 (5)                       |
| C5  | 0.5060 (2)   | 0.6490 (2)    | 0.17547 (15)  | 0.0617 (6)                       |

|      |              |              |               |             |
|------|--------------|--------------|---------------|-------------|
| H5   | 0.4715       | 0.7234       | 0.1927        | 0.074*      |
| C6   | 0.62038 (19) | 0.65640 (19) | 0.16368 (15)  | 0.0587 (5)  |
| H6   | 0.6618       | 0.7353       | 0.1733        | 0.070*      |
| C7   | 0.3173 (2)   | 0.5254 (3)   | 0.1756 (3)    | 0.0982 (9)  |
| H7A  | 0.3224       | 0.4715       | 0.2333        | 0.147*      |
| H7B  | 0.2593       | 0.4890       | 0.1152        | 0.147*      |
| H7C  | 0.2912       | 0.6109       | 0.1867        | 0.147*      |
| C8   | 0.62327 (18) | 0.20947 (18) | 0.05512 (14)  | 0.0522 (5)  |
| C9   | 0.7136 (2)   | 0.1393 (2)   | 0.01338 (15)  | 0.0568 (5)  |
| C10  | 0.7491 (3)   | -0.0559 (2)  | -0.07135 (18) | 0.0756 (7)  |
| H10A | 0.8154       | -0.0044      | -0.0798       | 0.091*      |
| H10B | 0.6987       | -0.0857      | -0.1384       | 0.091*      |
| C11  | 0.8022 (2)   | -0.1719 (2)  | -0.00525 (19) | 0.0739 (7)  |
| H11A | 0.8523       | -0.2210      | -0.0365       | 0.089*      |
| H11B | 0.7358       | -0.2277      | -0.0022       | 0.089*      |
| C12  | 0.8800 (2)   | -0.1347 (2)  | 0.10290 (19)  | 0.0692 (6)  |
| H12A | 0.9478       | -0.0812      | 0.0999        | 0.083*      |
| H12B | 0.8306       | -0.0833      | 0.1333        | 0.083*      |
| C13  | 1.0343 (3)   | -0.3023 (3)  | 0.1486 (4)    | 0.1352 (16) |
| H13A | 1.0639       | -0.3754      | 0.1927        | 0.203*      |
| H13B | 1.0979       | -0.2381      | 0.1613        | 0.203*      |
| H13C | 1.0112       | -0.3299      | 0.0787        | 0.203*      |
| C14  | 0.9619 (3)   | -0.2082 (3)  | 0.2779 (2)    | 0.1081 (10) |
| H14A | 1.0234       | -0.1416      | 0.2926        | 0.162*      |
| H14B | 0.9933       | -0.2822      | 0.3206        | 0.162*      |
| H14C | 0.8904       | -0.1759      | 0.2908        | 0.162*      |
| H1   | 0.732 (2)    | 0.343 (2)    | 0.0811 (16)   | 0.062 (7)*  |
| H2   | 0.599 (2)    | -0.003 (2)   | -0.0308 (18)  | 0.078 (8)*  |
| H1A  | 0.824 (2)    | 0.622 (2)    | 0.139 (2)     | 0.109 (10)* |

Atomic displacement parameters ( $\text{\AA}^2$ )

|    | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| O1 | 0.0618 (10) | 0.0484 (9)  | 0.1234 (14) | -0.0114 (7)  | 0.0397 (10) | -0.0126 (9)  |
| O2 | 0.0639 (9)  | 0.0513 (8)  | 0.0878 (11) | -0.0137 (7)  | 0.0256 (8)  | -0.0038 (7)  |
| O3 | 0.0699 (11) | 0.0702 (10) | 0.1088 (13) | -0.0180 (9)  | 0.0365 (10) | -0.0231 (9)  |
| N1 | 0.0503 (10) | 0.0458 (9)  | 0.0663 (11) | -0.0055 (8)  | 0.0153 (9)  | -0.0007 (8)  |
| N2 | 0.0644 (12) | 0.0522 (10) | 0.0612 (11) | -0.0056 (9)  | 0.0152 (9)  | -0.0059 (8)  |
| N3 | 0.0500 (10) | 0.0522 (10) | 0.1121 (16) | 0.0014 (8)   | 0.0245 (10) | 0.0097 (10)  |
| C1 | 0.0525 (11) | 0.0476 (11) | 0.0571 (11) | -0.0021 (9)  | 0.0134 (9)  | 0.0028 (9)   |
| C2 | 0.0503 (11) | 0.0444 (10) | 0.0477 (10) | -0.0003 (8)  | 0.0094 (8)  | 0.0034 (8)   |
| C3 | 0.0544 (12) | 0.0572 (12) | 0.0576 (12) | -0.0071 (9)  | 0.0142 (10) | 0.0030 (9)   |
| C4 | 0.0556 (12) | 0.0699 (14) | 0.0602 (12) | 0.0021 (11)  | 0.0176 (10) | 0.0001 (10)  |
| C5 | 0.0642 (13) | 0.0608 (13) | 0.0562 (12) | 0.0106 (11)  | 0.0144 (10) | -0.0013 (10) |
| C6 | 0.0639 (13) | 0.0459 (11) | 0.0620 (12) | -0.0015 (9)  | 0.0147 (10) | -0.0016 (9)  |
| C7 | 0.0717 (17) | 0.106 (2)   | 0.128 (2)   | -0.0018 (16) | 0.0483 (17) | -0.0125 (19) |
| C8 | 0.0538 (12) | 0.0433 (10) | 0.0524 (11) | -0.0039 (9)  | 0.0078 (9)  | 0.0045 (8)   |
| C9 | 0.0580 (12) | 0.0503 (11) | 0.0562 (11) | -0.0054 (10) | 0.0109 (10) | 0.0010 (9)   |

|     |             |             |             |              |              |              |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C10 | 0.1010 (19) | 0.0682 (14) | 0.0629 (14) | -0.0081 (13) | 0.0340 (13)  | -0.0143 (11) |
| C11 | 0.0895 (17) | 0.0542 (13) | 0.0863 (16) | -0.0011 (12) | 0.0402 (14)  | -0.0182 (12) |
| C12 | 0.0598 (13) | 0.0476 (11) | 0.0948 (17) | -0.0030 (10) | 0.0182 (12)  | -0.0023 (11) |
| C13 | 0.0772 (19) | 0.096 (2)   | 0.253 (5)   | 0.0301 (17)  | 0.083 (3)    | 0.056 (3)    |
| C14 | 0.097 (2)   | 0.096 (2)   | 0.100 (2)   | 0.0002 (17)  | -0.0096 (17) | 0.0116 (17)  |

*Geometric parameters (Å, °)*

|            |             |               |             |
|------------|-------------|---------------|-------------|
| O1—C1      | 1.359 (3)   | C5—H5         | 0.9300      |
| O2—C8      | 1.222 (2)   | C6—H6         | 0.9300      |
| O3—C9      | 1.231 (2)   | C7—H7A        | 0.9600      |
| N1—C2      | 1.403 (3)   | C7—H7B        | 0.9600      |
| N1—C8      | 1.334 (2)   | C7—H7C        | 0.9600      |
| N2—C9      | 1.322 (3)   | C8—C9         | 1.530 (3)   |
| O1—H1A     | 0.904 (17)  | C10—C11       | 1.509 (3)   |
| N1—H1      | 0.83 (2)    | C10—H10A      | 0.9700      |
| N2—C10     | 1.451 (3)   | C10—H10B      | 0.9700      |
| N2—H2      | 0.92 (3)    | C11—C12       | 1.522 (3)   |
| N3—C13     | 1.450 (3)   | C11—H11A      | 0.9700      |
| N3—C12     | 1.464 (3)   | C11—H11B      | 0.9700      |
| N3—C14     | 1.494 (4)   | C12—H12A      | 0.9700      |
| C1—C6      | 1.377 (3)   | C12—H12B      | 0.9700      |
| C1—C2      | 1.400 (3)   | C13—H13A      | 0.9600      |
| C2—C3      | 1.381 (3)   | C13—H13B      | 0.9600      |
| C3—C4      | 1.390 (3)   | C13—H13C      | 0.9600      |
| C3—H3A     | 0.9300      | C14—H14A      | 0.9600      |
| C4—C5      | 1.383 (3)   | C14—H14B      | 0.9600      |
| C4—C7      | 1.511 (3)   | C14—H14C      | 0.9600      |
| C5—C6      | 1.385 (3)   |               |             |
| C1—O1—H1A  | 112.6 (18)  | O2—C8—C9      | 122.52 (18) |
| C8—N1—C2   | 130.73 (19) | N1—C8—C9      | 110.60 (18) |
| C8—N1—H1   | 112.0 (15)  | O3—C9—N2      | 124.6 (2)   |
| C2—N1—H1   | 116.6 (15)  | O3—C9—C8      | 120.83 (19) |
| C9—N2—C10  | 122.4 (2)   | N2—C9—C8      | 114.58 (19) |
| C9—N2—H2   | 118.2 (16)  | N2—C10—C11    | 112.48 (19) |
| C10—N2—H2  | 119.4 (16)  | N2—C10—H10A   | 109.1       |
| C13—N3—C12 | 111.7 (2)   | C11—C10—H10A  | 109.1       |
| C13—N3—C14 | 110.2 (3)   | N2—C10—H10B   | 109.1       |
| C12—N3—C14 | 109.6 (2)   | C11—C10—H10B  | 109.1       |
| O1—C1—C6   | 124.96 (18) | H10A—C10—H10B | 107.8       |
| O1—C1—C2   | 116.38 (17) | C10—C11—C12   | 112.92 (18) |
| C6—C1—C2   | 118.66 (19) | C10—C11—H11A  | 109.0       |
| C3—C2—C1   | 120.35 (18) | C12—C11—H11A  | 109.0       |
| C3—C2—N1   | 124.63 (17) | C10—C11—H11B  | 109.0       |
| C1—C2—N1   | 114.99 (18) | C12—C11—H11B  | 109.0       |
| C2—C3—C4   | 120.99 (19) | H11A—C11—H11B | 107.8       |
| C2—C3—H3A  | 119.5       | N3—C12—C11    | 113.16 (18) |

|                |              |                |              |
|----------------|--------------|----------------|--------------|
| C4—C3—H3A      | 119.5        | N3—C12—H12A    | 108.9        |
| C5—C4—C3       | 118.1 (2)    | C11—C12—H12A   | 108.9        |
| C5—C4—C7       | 121.2 (2)    | N3—C12—H12B    | 108.9        |
| C3—C4—C7       | 120.7 (2)    | C11—C12—H12B   | 108.9        |
| C4—C5—C6       | 121.34 (19)  | H12A—C12—H12B  | 107.8        |
| C4—C5—H5       | 119.3        | N3—C13—H13A    | 109.5        |
| C6—C5—H5       | 119.3        | N3—C13—H13B    | 109.5        |
| C1—C6—C5       | 120.51 (19)  | H13A—C13—H13B  | 109.5        |
| C1—C6—H6       | 119.7        | N3—C13—H13C    | 109.5        |
| C5—C6—H6       | 119.7        | H13A—C13—H13C  | 109.5        |
| C4—C7—H7A      | 109.5        | H13B—C13—H13C  | 109.5        |
| C4—C7—H7B      | 109.5        | N3—C14—H14A    | 109.5        |
| H7A—C7—H7B     | 109.5        | N3—C14—H14B    | 109.5        |
| C4—C7—H7C      | 109.5        | H14A—C14—H14B  | 109.5        |
| H7A—C7—H7C     | 109.5        | N3—C14—H14C    | 109.5        |
| H7B—C7—H7C     | 109.5        | H14A—C14—H14C  | 109.5        |
| O2—C8—N1       | 126.9 (2)    | H14B—C14—H14C  | 109.5        |
|                |              |                |              |
| C8—N1—C2—C3    | 7.4 (3)      | O1—C1—C6—C5    | -178.97 (19) |
| C8—N1—C2—C1    | -170.86 (19) | C2—C1—C6—C5    | 1.6 (3)      |
| C9—N2—C10—C11  | 106.7 (2)    | C4—C5—C6—C1    | 0.2 (3)      |
| N2—C10—C11—C12 | -57.1 (3)    | C2—N1—C8—O2    | -9.1 (3)     |
| O1—C1—C2—C3    | 178.43 (18)  | C2—N1—C8—C9    | 170.82 (19)  |
| C6—C1—C2—C3    | -2.1 (3)     | C10—N2—C9—O3   | 0.8 (3)      |
| O1—C1—C2—N1    | -3.2 (3)     | C10—N2—C9—C8   | -179.59 (18) |
| C6—C1—C2—N1    | 176.24 (17)  | O2—C8—C9—O3    | -176.04 (19) |
| C1—C2—C3—C4    | 0.8 (3)      | N1—C8—C9—O3    | 4.0 (3)      |
| N1—C2—C3—C4    | -177.40 (19) | O2—C8—C9—N2    | 4.3 (3)      |
| C2—C3—C4—C5    | 1.1 (3)      | N1—C8—C9—N2    | -175.62 (17) |
| C2—C3—C4—C7    | 179.8 (2)    | C13—N3—C12—C11 | 78.6 (3)     |
| C3—C4—C5—C6    | -1.6 (3)     | C14—N3—C12—C11 | -158.9 (2)   |
| C7—C4—C5—C6    | 179.7 (2)    | C10—C11—C12—N3 | 178.2 (2)    |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i>   | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H2...O2 <sup>i</sup>   | 0.92 (3)    | 2.13 (3)      | 2.945 (2)             | 147 (2)                 |
| O1—H1A...N3 <sup>ii</sup> | 0.90 (2)    | 1.76 (2)      | 2.654 (2)             | 168 (3)                 |

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