



Crystal structure of 4,10-dimethoxy-13-methyl-6*H*,12*H*-6,12-epiminodibenzo[*b*,*f*][1,5]dioxocine

Katerina V. Kasyanova,^a Vladimir N. Kozozay,^a Elena A. Buvaylo,^a Olga Yu. Vassilyeva^{a*} and Brian W. Skelton^b

^aDepartment of Chemistry, Taras Shevchenko National University of Kyiv, 64/13 Volodymyrska Street, Kyiv 01601, Ukraine, and ^bSchool of Molecular Sciences, M310, University of Western Australia, Perth, WA 6009, Australia.

*Correspondence e-mail: vassilyeva@univ.kiev.ua

Received 10 February 2017

Accepted 11 February 2017

Edited by P. C. Healy, Griffith University, Australia

Keywords: crystal structure; *o*-vanillin; methylamine hydrochloride; Schiff base; self-condensation; [1,5]dioxocin ring; twisted-boat conformation.

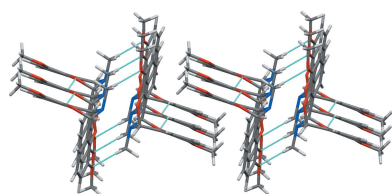
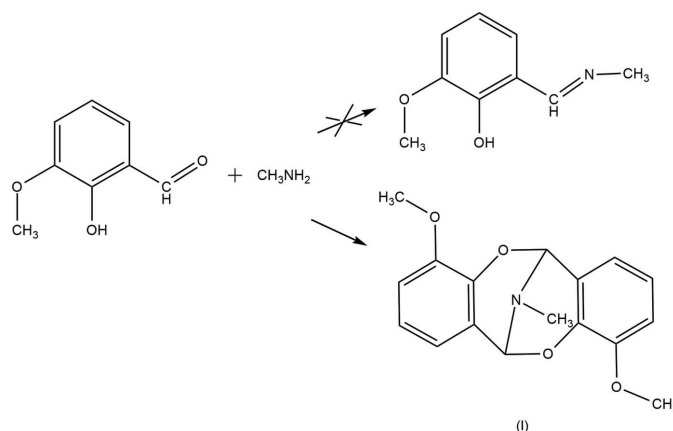
CCDC reference: 1532218

Supporting information: this article has supporting information at journals.iucr.org/e

The title compound, C₁₇H₁₇NO₄, lacks crystallographic symmetry with one molecule per asymmetric unit. The molecule exists in a folded butterfly-like conformation; the benzene rings form a dihedral angle of 84.72 (7)°. The central eight-membered imino-bridged dioxocin ring adopts a twisted-boat conformation. In the crystal, inversion-related molecules are linked by pairs of weak C—H···O hydrogen bonds, forming double-stranded chains parallel to the *a* axis.

1. Chemical context

Tröger's base and its structural analogues are characterized by two flat, usually aromatic and identical, pincers interlocked in an almost perpendicular fashion (Dolenský *et al.*, 2012). Both the chirality and the conformational rigidity of their central diazocine, dioxocin or dithiocin skeletons are the reasons why these cleft-shaped molecules have been of interest in molecular recognition (Hardouin-Lerouge *et al.*, 2011), as chiral solvating agents (Wilén *et al.*, 1991), and in the field of asymmetric synthesis (Minder *et al.*, 1995).



Over the last few years, we have been exploring the chemistry of transition metal complexes of Schiff base ligands with the aim of preparing heterometallic polynuclear compounds with diverse potential advantages (Chygorin *et al.*, 2012; Nesterova *et al.*, 2013). The Schiff base ligand 2-methoxy-6-iminomethylphenol (HL) with various connectivity modes has been successfully used as a multidentate linker between several metal centres by our group and others (Meally *et al.*, 2010; Sydoruk *et al.*, 2013). The HL ligand is usually obtained by the standard method of condensation of

the substituted salicylaldehyde with an aqueous solution of methylamine in methanol (Meally *et al.*, 2010). In the present work, we used a mixture of 2-hydroxy-3-methoxy-benzaldehyde and methylamine hydrochloride to react with a zinc salt in an attempt to synthesize a Zn complex with the HL ligand (see Scheme). The resulting Schiff base apparently underwent self-condensation to form the substituted dibenzoimino[1,5]-dioxocin, 4,10-dimethoxy-13-methyl-6*H*,12*H*-6,12-epimino-dibenzo[*b,f*][1,5]dioxocine, (I), the crystal structure of which is presented here. A close analogue of the title compound was reported to result from 2-(*N*-methyliminomethyl)phenol, a liquid product of a similar condensation of salicylaldehyde and methylamine, after a few months storage in mild conditions (Filarowski *et al.*, 1998). A tentative mechanism for the formation of the [1,5]iminodioxocin ring in the reaction between an aromatic aldehyde and a primary amine was given by Mandal *et al.* (2006).

2. Structural commentary

The title compound is composed of four fused rings including two benzene (C11–C16 and C21–C26) and two six-membered heterocyclic rings (O11/C11/C12/C121/N1/C221 and O21/C21/C22/C221/N1/C121) (Fig. 1). The organic molecule has two chiral centres and lacks crystallographic symmetry; the crystal is racemic. The molecule exists in a folded butterfly-like conformation with a dihedral (folding) angle between the two benzene rings of 84.72 (7)°. The eight-membered imino-bridged dioxocin ring adopts a twisted-boat conformation, as judged from the eight torsion angles observed within this ring (τ_1 – τ_8) (Mandal *et al.*, 2006). The bond lengths and angles are unexceptional and are closely related to those of *N*-methyl-2,6,-dioxo-9-aza-(*c.g*)dibenzo(3.3.1)nonane (CSD refcode

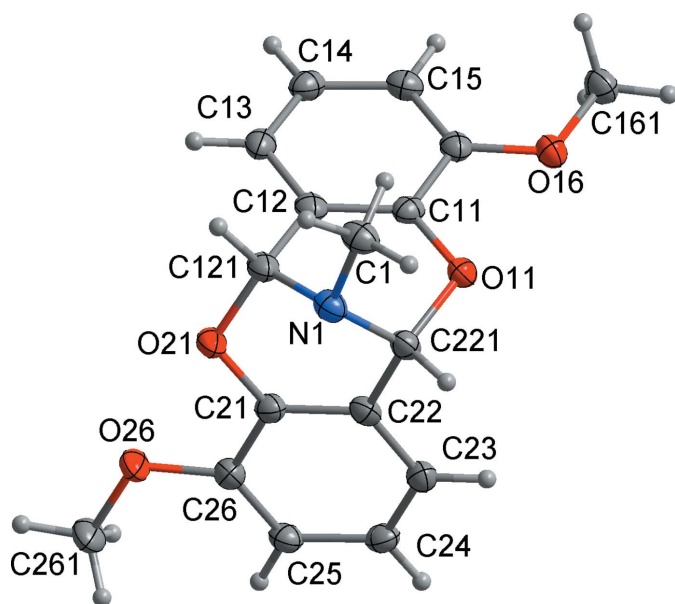


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Non-H atoms are shown with displacement ellipsoids at the 50% probability level.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13–H13 \cdots O11 ⁱ	0.95	2.69	3.5616 (19)	152
C1–H1B \cdots O26 ⁱⁱ	0.98	2.53	3.508 (2)	176

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

UCERIE; Filarowski *et al.*, 1998); the dihedral angle of 84.72 (7)° is larger than that in the unsubstituted imino-dioxocin molecule (80.95°).

3. Supramolecular features

In the crystal, double-stranded chains of inversion-related molecules linked by pairs of weak C–H \cdots O hydrogen bonds (Table 1) propagate in the *a*-axis direction (Fig. 2). Adjacent hydrogen-bonded chains are arranged in a parallel fashion to ensure efficient crystal packing of the clefts. Surprisingly, neither π – π stacking [the shortest centroid–centroid distance (offset) = 3.96 Å] nor C–H \cdots π interactions (the shortest H \cdots centroid distance = 3.34 Å) play a significant role in formation of the crystal structure of (I).

4. Database survey

More than 1000 crystal structures of molecules featuring eight-membered heterocine rings with two oxygen atoms in a 1,2-, 1,3-, 1,4- and 1,5-relationship, both uncondensed and fused to five-, six-, and seven-membered carbocycles or heterocycles, are found in the Cambridge Structural Database (CSD Version 5.37 plus one update; Groom *et al.*, 2016) with bridged dioxocines constituting the majority of the compounds reported. Of these, only five molecules contain the same central imino-bridged [1,5]dioxocin core as in compound (I) (refcodes GAQNUJ, QAYTIU, TECMAP, UCERIE, XESBON). Clearly, substituents on the aromatic rings and on the bridging imino N atom in the five compounds determine the differences in their folding angles, which fall in the range 78.49–96.84°. However, no obvious correlation between the

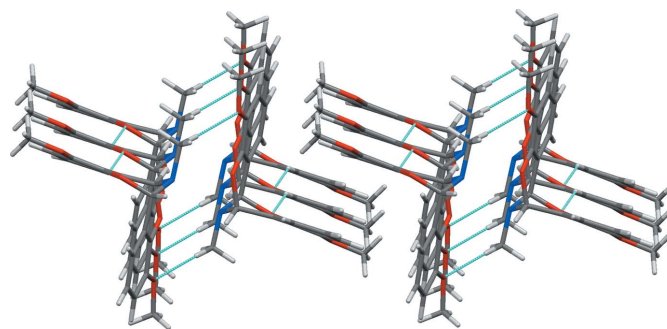


Figure 2
Crystal packing of (I), showing the parallel arrangement of double-stranded hydrogen-bonded chains of the dibenzoimino[1,5]dioxocin molecules along the *a*-axis direction. Intermolecular hydrogen bonds are shown as blue dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₇ H ₁₇ NO ₄
<i>M</i> _r	299.31
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.9956 (5), 8.8589 (6), 12.0938 (9)
α , β , γ (°)	93.980 (6), 106.603 (7), 102.133 (6)
<i>V</i> (Å ³)	695.46 (9)
<i>Z</i>	2
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	0.84
Crystal size (mm)	0.18 × 0.06 × 0.04
Data collection	
Diffractometer	Oxford Diffraction Gemini
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.818, 1
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	5235, 2456, 2147
<i>R</i> _{int}	0.027
(sin θ/λ) _{max} (Å ⁻¹)	0.598
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.041, 0.121, 1.07
No. of reflections	2456
No. of parameters	202
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.22, -0.25

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *DIAMOND* (Brandenburg, 1999), *Mercury* (Macrae *et al.*, 2006) and *WinGX* (Farrugia, 1999).

nature/size/position of the substituents and widening of the folding angle can be established due to the small number of compounds involved. While an example of [1,5]iminodioxocin bridgehead N-atom coordination to a metal atom (copper) has been reported (refcode XESBON; Mandal *et al.*, 2006), the Zn atom did not demonstrate the ability to coordinate the ligand (I) in the present study.

5. Synthesis and crystallization

2-Hydroxy-3-methoxy-benzaldehyde (0.23 g, 1.5 mmol) and methylamine hydrochloride (0.10 g, 1.5 mmol) were added to methanol (5 ml) and stirred magnetically for 10 min. Zn(CH₃COO)₂·2H₂O (0.11 g, 0.5 mmol) dissolved in 5 ml dimethylformamide was added to the yellow solution of the Schiff base formed *in situ*, and the resulting deep-yellow solution was stirred at room temperature for an hour. The addition of N(Et)₃ (1 ml) produced a light precipitate which was filtered off. The solution, which was kept cold (283–285 K), changed colour from yellow to brown. It was diluted twice with methanol (4 ml) since it was thickening. Brown plate-like crystals of the title compound formed over two months after successive addition of PrⁱOH (4 ml) in two portions. They were collected by filter-suction, washed with dry PrⁱOH and finally dried in air (yield: 23%). Analysis calculated for C₁₇H₁₇NO₄ (299.31): C, 68.21; H, 5.72; N, 4.68%. Found: C, 68.55; H, 5.49; N, 4.87%. ¹H NMR (400 MHz,

DMSO-*d*₆, *s*, singlet; *m*, multiplet): δ (ppm) 6.89–6.79, *m* (6H, benzene rings); 5.69, *s* (2H, dioxocin ring); 3.71, *s* (6H, OCH₃); 2.51, *s* (3H, NCH₃). The IR spectrum of powdered (I) in the range 4000–400 cm⁻¹ shows all characteristic functional groups peaks: ν(CH) due to aromatic =C–H and alkyl –C–H stretching above and below 3000, respectively, the aromatic rings vibrations in the 1600–1400 region, ν(CO) and ν(CN) at 1300–1000 and aromatic CH bending in the 900–600 cm⁻¹ region (see Supporting information).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms bound to carbon were included in calculated positions and refined using a riding model with isotropic displacement parameters based on those of the parent atom (C–H = 0.95 Å, *U*_{iso}(H) = 1.2*U*_{eq}C for CH, C–H = 0.98 Å, *U*_{iso}(H) = 1.5*U*_{eq}C for CH₃). Anisotropic displacement parameters were employed for the non-hydrogen atoms.

References

- Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Chygorin, E. N., Nesterova, O. V., Rusanova, J. A., Kokozay, V. N., Bon, V. V., Boča, R. & Ozarowski, A. (2012). *Inorg. Chem.* **51**, 386–396.
- Dolenský, B., Havlík, M. & Král, V. (2012). *Chem. Soc. Rev.* **41**, 3839–3858.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Filarowski, A., Koll, A., Glowiak, T., Majewski, E. & Dziembowska, T. (1998). *Berichte der Bunsengesellschaft für physikalische Chemie*, **102**, 393–402.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Hardouin-Lerouge, M., Hudhomme, P. & Sallé, M. (2011). *Chem. Soc. Rev.* **40**, 30–43.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Mandal, D., Wu, A. Q., Guo, G. C. & Ray, D. (2006). *Inorg. Chem.* **45**, 8826–8828.
- Meally, S. T., McDonald, C., Karotsis, G., Papaefstathiou, G. S., Brechin, E. K., Dunne, P. W., McArdle, P., Power, N. P. & Jones, L. F. (2010). *Dalton Trans.* **39**, 4809–4816.
- Minder, B., Schürch, M., Mallat, T. & Baiker, A. (1995). *Catal. Lett.* **31**, 143–151.
- Nesterova, O. V., Chygorin, E. N., Kokozay, V. N., Bon, V. V., Omelchenko, I. V., Shishkin, O. V., Titiš, J., Boča, R., Pombeiro, A. J. & Ozarowski, A. (2013). *Dalton Trans.* **42**, 16909–16919.
- Rigaku OD (2015). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, England.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Sydooruk, T. V., Buvaylo, E. A., Kokozay, V. N., Vassilyeva, O. Y. & Skelton, B. W. (2013). *Acta Cryst.* **E69**, m551–m552.
- Wilens, S. H., Qi, J. Z. & Williard, P. G. (1991). *J. Org. Chem.* **56**, 485–487.

supporting information

Acta Cryst. (2017). E73, 410-412 [https://doi.org/10.1107/S2056989017002328]

Crystal structure of 4,10-dimethoxy-13-methyl-6*H*,12*H*-6,12-epimino-dibenzo[*b,f*][1,5]dioxocine

Katerina V. Kasyanova, Vladimir N. Kokozay, Elena A. Buvaylo, Olga Yu. Vassilyeva and Brian W. Skelton

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg, 1999) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

4,10-Dimethoxy-13-methyl-6*H*,12*H*-6,12-epiminodibenzo[*b,f*][1,5]dioxocine

Crystal data

$C_{17}H_{17}NO_4$	$Z = 2$
$M_r = 299.31$	$F(000) = 316$
Triclinic, $P\bar{1}$	$D_x = 1.429 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
$a = 6.9956 (5) \text{ \AA}$	Cell parameters from 2735 reflections
$b = 8.8589 (6) \text{ \AA}$	$\theta = 3.9\text{--}67.1^\circ$
$c = 12.0938 (9) \text{ \AA}$	$\mu = 0.84 \text{ mm}^{-1}$
$\alpha = 93.980 (6)^\circ$	$T = 100 \text{ K}$
$\beta = 106.603 (7)^\circ$	Plate, brown
$\gamma = 102.133 (6)^\circ$	$0.18 \times 0.06 \times 0.04 \text{ mm}$
$V = 695.46 (9) \text{ \AA}^3$	

Data collection

Oxford Diffraction Gemini diffractometer	5235 measured reflections
Radiation source: sealed X-ray tube	2456 independent reflections
Mirror monochromator	2147 reflections with $I > 2\sigma(I)$
Detector resolution: $10.4738 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.027$
ω scans	$\theta_{\text{max}} = 67.2^\circ$, $\theta_{\text{min}} = 3.9^\circ$
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)	$h = -8 \rightarrow 8$
$T_{\text{min}} = 0.818$, $T_{\text{max}} = 1$	$k = -9 \rightarrow 10$
	$l = -12 \rightarrow 14$

Refinement

Refinement on F^2	202 parameters
Least-squares matrix: full	0 restraints
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.121$	H-atom parameters constrained
$S = 1.07$	
2456 reflections	

$$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.1321P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8680 (3)	0.77719 (18)	0.96792 (13)	0.0272 (4)
H1A	1.0173	0.8034	1.0043	0.041*
H1B	0.802	0.8059	1.0249	0.041*
H1C	0.8334	0.8345	0.9014	0.041*
N1	0.7947 (2)	0.60859 (15)	0.92762 (11)	0.0239 (3)
C11	0.6973 (2)	0.64283 (16)	0.69544 (13)	0.0208 (3)
O11	0.89314 (15)	0.63442 (12)	0.75392 (9)	0.0216 (3)
C12	0.5341 (2)	0.60187 (17)	0.74052 (13)	0.0220 (3)
C121	0.5761 (2)	0.56411 (17)	0.86455 (13)	0.0234 (3)
H121	0.5042	0.624	0.9056	0.028*
C13	0.3363 (2)	0.60620 (17)	0.67238 (14)	0.0248 (3)
H13	0.2232	0.5779	0.7016	0.03*
C14	0.3059 (2)	0.65148 (18)	0.56296 (14)	0.0260 (4)
H14	0.1707	0.6494	0.5159	0.031*
C15	0.4714 (2)	0.70032 (17)	0.52068 (13)	0.0245 (3)
H15	0.4492	0.7351	0.4465	0.029*
C16	0.6678 (2)	0.69812 (17)	0.58676 (13)	0.0217 (3)
O16	0.84270 (16)	0.74649 (12)	0.55663 (9)	0.0252 (3)
C161	0.8176 (3)	0.80636 (19)	0.44818 (13)	0.0280 (4)
H16A	0.7297	0.7244	0.3846	0.042*
H16B	0.9522	0.8411	0.4367	0.042*
H16C	0.7536	0.8946	0.4488	0.042*
C21	0.6140 (2)	0.31265 (18)	0.82770 (12)	0.0214 (3)
O21	0.50055 (16)	0.40035 (12)	0.86700 (9)	0.0242 (3)
C22	0.8031 (2)	0.37774 (17)	0.81425 (12)	0.0215 (3)
C221	0.8989 (2)	0.55005 (17)	0.85422 (13)	0.0218 (3)
H221	1.0459	0.563	0.9011	0.026*
C23	0.9039 (2)	0.28166 (18)	0.76738 (13)	0.0232 (3)
H23	1.033	0.3251	0.7573	0.028*
C24	0.8164 (2)	0.12416 (18)	0.73579 (13)	0.0241 (3)
H24	0.8838	0.0603	0.7017	0.029*
C25	0.6289 (2)	0.05727 (18)	0.75355 (13)	0.0235 (3)
H25	0.5711	-0.0517	0.7332	0.028*
C26	0.5290 (2)	0.15091 (18)	0.80075 (13)	0.0225 (3)
O26	0.34536 (17)	0.10158 (12)	0.82185 (10)	0.0275 (3)
C261	0.2625 (3)	-0.06296 (19)	0.80651 (16)	0.0326 (4)

H26A	0.2318	-0.1056	0.7247	0.049*
H26B	0.1359	-0.0843	0.8284	0.049*
H26C	0.3628	-0.1118	0.8559	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0350 (9)	0.0211 (8)	0.0248 (8)	0.0057 (7)	0.0094 (7)	0.0021 (6)
N1	0.0294 (7)	0.0195 (7)	0.0237 (6)	0.0051 (5)	0.0100 (5)	0.0035 (5)
C11	0.0217 (7)	0.0147 (7)	0.0253 (7)	0.0047 (6)	0.0061 (6)	0.0013 (5)
O11	0.0214 (5)	0.0201 (5)	0.0240 (6)	0.0041 (4)	0.0077 (4)	0.0064 (4)
C12	0.0261 (8)	0.0138 (7)	0.0270 (8)	0.0045 (6)	0.0103 (6)	0.0020 (5)
C121	0.0282 (8)	0.0166 (7)	0.0290 (8)	0.0064 (6)	0.0134 (6)	0.0044 (6)
C13	0.0236 (8)	0.0165 (7)	0.0362 (8)	0.0042 (6)	0.0129 (6)	0.0023 (6)
C14	0.0239 (8)	0.0197 (8)	0.0331 (8)	0.0070 (6)	0.0057 (6)	0.0027 (6)
C15	0.0294 (8)	0.0199 (8)	0.0242 (7)	0.0073 (6)	0.0070 (6)	0.0043 (6)
C16	0.0250 (7)	0.0158 (7)	0.0253 (7)	0.0043 (6)	0.0099 (6)	0.0017 (6)
O16	0.0257 (6)	0.0272 (6)	0.0250 (6)	0.0060 (4)	0.0108 (4)	0.0084 (4)
C161	0.0327 (8)	0.0287 (9)	0.0257 (8)	0.0077 (7)	0.0125 (7)	0.0091 (6)
C21	0.0246 (7)	0.0202 (8)	0.0222 (7)	0.0080 (6)	0.0094 (6)	0.0046 (6)
O21	0.0282 (6)	0.0167 (6)	0.0330 (6)	0.0060 (4)	0.0169 (5)	0.0052 (4)
C22	0.0240 (7)	0.0201 (8)	0.0203 (7)	0.0054 (6)	0.0060 (6)	0.0063 (6)
C221	0.0239 (7)	0.0205 (8)	0.0218 (7)	0.0048 (6)	0.0075 (6)	0.0072 (6)
C23	0.0221 (7)	0.0261 (8)	0.0240 (7)	0.0073 (6)	0.0092 (6)	0.0075 (6)
C24	0.0281 (8)	0.0236 (8)	0.0251 (7)	0.0125 (6)	0.0101 (6)	0.0052 (6)
C25	0.0280 (8)	0.0176 (8)	0.0258 (7)	0.0063 (6)	0.0089 (6)	0.0046 (6)
C26	0.0227 (7)	0.0218 (8)	0.0249 (7)	0.0060 (6)	0.0091 (6)	0.0065 (6)
O26	0.0277 (6)	0.0177 (6)	0.0415 (6)	0.0035 (4)	0.0183 (5)	0.0060 (4)
C261	0.0319 (9)	0.0183 (8)	0.0505 (10)	0.0024 (7)	0.0196 (8)	0.0064 (7)

Geometric parameters (Å, °)

C1—N1	1.4716 (19)	O16—C161	1.4271 (18)
C1—H1A	0.98	C161—H16A	0.98
C1—H1B	0.98	C161—H16B	0.98
C1—H1C	0.98	C161—H16C	0.98
N1—C221	1.433 (2)	C21—O21	1.3723 (18)
N1—C121	1.454 (2)	C21—C22	1.387 (2)
C11—O11	1.3707 (18)	C21—C26	1.407 (2)
C11—C12	1.394 (2)	C22—C23	1.399 (2)
C11—C16	1.410 (2)	C22—C221	1.515 (2)
O11—C221	1.4636 (17)	C221—H221	1
C12—C13	1.403 (2)	C23—C24	1.379 (2)
C12—C121	1.519 (2)	C23—H23	0.95
C121—O21	1.4413 (18)	C24—C25	1.403 (2)
C121—H121	1	C24—H24	0.95
C13—C14	1.381 (2)	C25—C26	1.381 (2)
C13—H13	0.95	C25—H25	0.95

C14—C15	1.395 (2)	C26—O26	1.3690 (19)
C14—H14	0.95	O26—C261	1.4286 (18)
C15—C16	1.382 (2)	C261—H26A	0.98
C15—H15	0.95	C261—H26B	0.98
C16—O16	1.3675 (19)	C261—H26C	0.98
N1—C1—H1A	109.5	H16A—C161—H16B	109.5
N1—C1—H1B	109.5	O16—C161—H16C	109.5
H1A—C1—H1B	109.5	H16A—C161—H16C	109.5
N1—C1—H1C	109.5	H16B—C161—H16C	109.5
H1A—C1—H1C	109.5	O21—C21—C22	122.61 (14)
H1B—C1—H1C	109.5	O21—C21—C26	116.68 (13)
C221—N1—C121	107.32 (12)	C22—C21—C26	120.72 (14)
C221—N1—C1	114.33 (12)	C21—O21—C121	111.48 (11)
C121—N1—C1	112.09 (12)	C21—C22—C23	119.15 (14)
O11—C11—C12	122.53 (13)	C21—C22—C221	119.33 (13)
O11—C11—C16	116.70 (13)	C23—C22—C221	121.48 (13)
C12—C11—C16	120.76 (13)	N1—C221—O11	111.99 (12)
C11—O11—C221	111.80 (11)	N1—C221—C22	109.13 (12)
C11—C12—C13	118.88 (14)	O11—C221—C22	110.50 (11)
C11—C12—C121	119.41 (13)	N1—C221—H221	108.4
C13—C12—C121	121.58 (13)	O11—C221—H221	108.4
O21—C121—N1	108.62 (12)	C22—C221—H221	108.4
O21—C121—C12	111.51 (12)	C24—C23—C22	120.23 (14)
N1—C121—C12	111.31 (12)	C24—C23—H23	119.9
O21—C121—H121	108.4	C22—C23—H23	119.9
N1—C121—H121	108.4	C23—C24—C25	120.62 (14)
C12—C121—H121	108.4	C23—C24—H24	119.7
C14—C13—C12	120.10 (14)	C25—C24—H24	119.7
C14—C13—H13	120	C26—C25—C24	119.59 (14)
C12—C13—H13	120	C26—C25—H25	120.2
C13—C14—C15	120.80 (14)	C24—C25—H25	120.2
C13—C14—H14	119.6	O26—C26—C25	125.72 (14)
C15—C14—H14	119.6	O26—C26—C21	114.69 (13)
C16—C15—C14	119.99 (14)	C25—C26—C21	119.56 (14)
C16—C15—H15	120	C26—O26—C261	116.82 (12)
C14—C15—H15	120	O26—C261—H26A	109.5
O16—C16—C15	125.65 (14)	O26—C261—H26B	109.5
O16—C16—C11	115.10 (13)	H26A—C261—H26B	109.5
C15—C16—C11	119.24 (14)	O26—C261—H26C	109.5
C16—O16—C161	116.22 (11)	H26A—C261—H26C	109.5
O16—C161—H16A	109.5	H26B—C261—H26C	109.5
O16—C161—H16B	109.5		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C13—H13 \cdots O11 ⁱ	0.95	2.69	3.5616 (19)	152

C1—H1B···O26 ⁱⁱ	0.98	2.53	3.508 (2)	176
----------------------------	------	------	-----------	-----

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