

Methyl 4-amino-3-methoxyisoxazole-5-carboxylate

Mohd Abdul Fatah Abdul Manan,^{a*} David B. Cordes^b and Alexandra M. Z. Slawin^b

^aFaculty of Applied Sciences, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia, and ^bEaStCHEM School of Chemistry, University of St Andrews, St Andrews, Fife KY16 9ST, United Kingdom. *Correspondence e-mail: abdfatah@uitm.edu.my

Received 14 July 2023

Accepted 16 July 2023

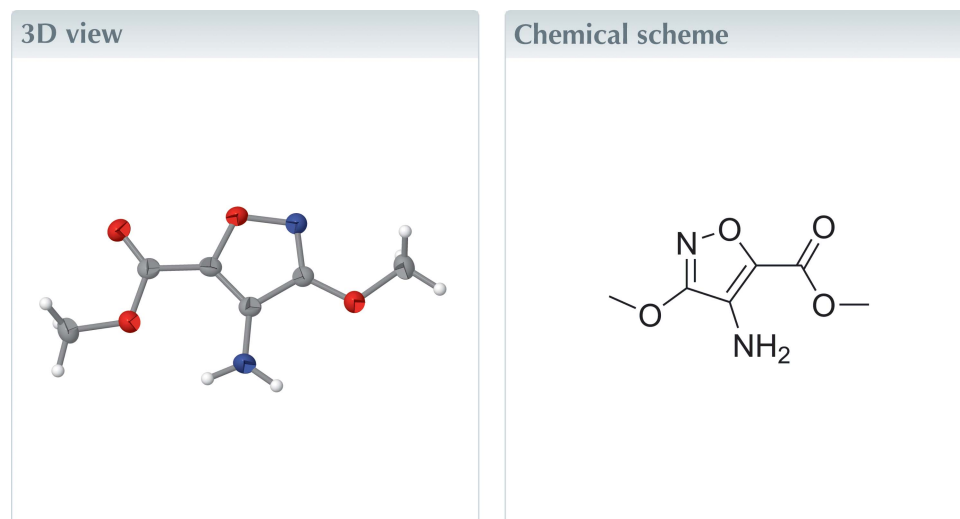
Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

Keywords: crystal structure; isoxazole; hydrogen-bonded chain.

CCDC reference: 2281787

Structural data: full structural data are available from iucrdata.iucr.org

The title compound, C₆H₈N₂O₄, a new derivative of isoxazole, has been synthesized and structurally characterized. The crystal structure shows the molecule to be almost planar (r.m.s. deviation for the non-hydrogen atoms = 0.029 Å), this conformation being supported by an intramolecular N—H···O hydrogen bond. In the extended structure, the molecules are linked by N—H···O hydrogen bonds into chains propagating along [010].



Structure description

Isoxazoles, five-membered heterocyclic compounds containing adjacent nitrogen and oxygen atoms, have many applications including in photochromic components (Pu *et al.*, 2011), liquid crystals (Kauhanka *et al.*, 2006), solar cells (Yoon *et al.*, 2022), high energy materials (Lal *et al.*, 2023), pesticides and insecticides (Wang *et al.*, 2022) and pharmaceuticals (Zhu *et al.*, 2018). In a continuation of our previous work on isoxazole derivatives (Abdul Manan *et al.*, 2023), we now present the synthesis and structure of the title compound.

The title compound, C₆H₈N₂O₄, crystallizes in space group *P2*₁/*c* with one molecule in the asymmetric unit (Fig. 1). All of the non-hydrogen atoms lie almost in the same plane, with an r.m.s. deviation of 0.029 Å and a maximum deviation of 0.060 (1) Å for C8. An intramolecular N—H···O_e (e = ester) hydrogen bond (Table 1) helps to ensure the near co-planarity of the isoxazole and ester moieties. This whole-molecule planarity, assisted by an intramolecular hydrogen bond, is similar to what was observed in the related compounds ethyl 5-amino-3-methylisoxazole-4-carboxylate (Sony *et al.*, 2005), ethyl 5-amino-3-(difluoromethyl)isoxazole-4-carboxylate (Schmitt *et al.*, 2015) and 5-amino-3-methylisoxazole-4-carbohydrazide (Regiec *et al.*, 2018). The relative orientation of the ester and amine groups, allowing the formation of the intramolecular hydrogen bond to the ester oxygen atom rather than the carbonyl oxygen atom, is, however, different to

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N4-H4A\cdots O6^i$	0.89 (1)	2.30 (2)	2.9765 (16)	133 (1)
$N4-H4B\cdots O1^i$	0.91 (1)	2.31 (2)	3.0233 (15)	136 (1)
$N4-H4B\cdots O7$	0.91 (1)	2.30 (2)	2.8734 (16)	121 (1)

Symmetry code: (i) $-x, y - \frac{1}{2}, -z + \frac{3}{2}$.

what is seen in ethyl 5-amino-3-methylisoxazole-4-carboxylate (Sony *et al.*, 2005), ethyl 5-amino-3-(difluoromethyl)isoxazole-4-carboxylate (Schmitt *et al.*, 2015), ethyl 5-amino-3-[fluoro-(trifluoromethoxy)methyl]isoxazole-4-carboxylate (Schmitt *et al.*, 2017) and 1-(cyclohexylcarbamoyl)cyclohexyl 5-amino-3-methylisoxazole-4-carboxylate (Baçhor *et al.*, 2019).

In the crystal of the title compound, adjacent molecules are linked by $N-H\cdots O_c$ (c = carbonyl) and $N-H\cdots O_i$ (i = isoxazole) hydrogen bonds, forming an $R_2^2(7)$ loop, which generates chains of molecules running along the crystallographic b -axis direction (Fig. 2). No additional directional interactions exist between chains. This combination of hydrogen bonds leading to chain formation is not seen in related isoxazole compounds as a result of the different relative position of the amine group on the isoxazole ring. While the combination of two inter- and one intramolecular hydrogen bond has been seen previously in related isoxazoles (Sony *et al.*, 2005; Regiec *et al.*, 2018; Baçhor *et al.*, 2019), the

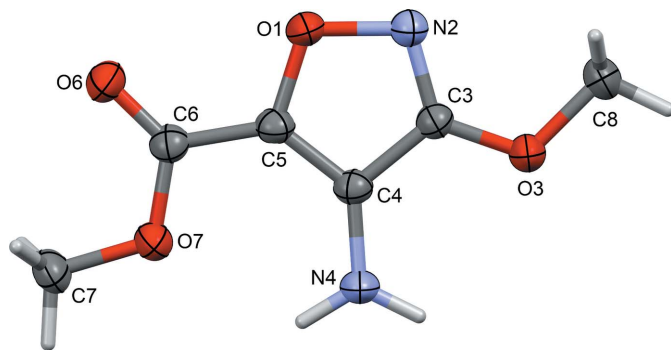


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

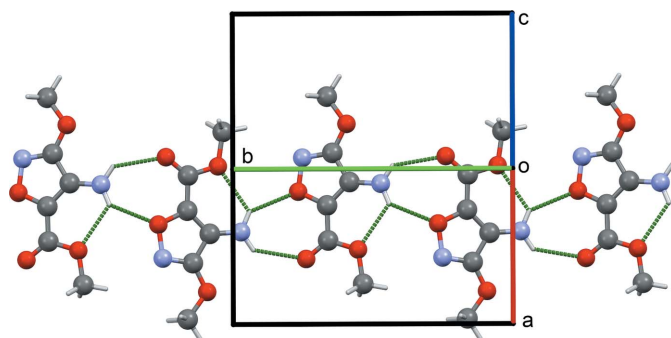


Figure 2
View down the [101] axis of the [010] chain formed by $N-H\cdots O$ hydrogen bonds, which are shown as dashed lines.

pattern of hydrogen bonds is either different or has additional hydrogen bonds contributing to it, and the resulting supramolecular motifs differ as well. One-dimensional chain motifs have been seen in two of the related isoxazoles (Schmitt *et al.*, 2015, 2017), although the pattern of hydrogen bonds that leads to the chains is different.

Synthesis and crystallization

Synthesis of the methyl 3-methoxy-4-nitroisoxazole-5-carboxylate precursor

The starting material, methyl 3-methoxyisoxazole-5-carboxylate, was prepared according to the previously described literature procedure with minor modifications (Melikian *et al.*, 1992). K_2CO_3 (2.9 g, 21.0 mmol, 1.5 eq) and CH_3I (1.3 ml, 21.0 mmol, 1.5 eq) were added to a solution of methyl 3-hydroxyisoxazole-5-carboxylate (2.0 g, 13.9 mmol, 1.0 eq) in dimethylformamide (DMF) (10 ml) at 0°C. After 14 h stirring at room temperature, the mixture was poured into an ice-cold aqueous solution of HCl (0.5 M, 100 ml) and extracted into Et_2O (5×80 ml). The combined organic layers were washed with a saturated aqueous solution of Na_2CO_3 (80 ml), dried over $MgSO_4$, filtered and concentrated under reduced pressure to afford a light yellow crystalline solid, which was purified by silica gel column chromatography (petroleum ether/ Et_2O , 80:20), affording methyl 3-methoxyisoxazole-5-carboxylate (1.45 g, 66%) as a colourless crystalline solid.

Triflic anhydride (5.9 g, 21.0 mmol, 3.0 eq) was added to a solution of tetramethylammonium nitrate (2.9 g, 21.0 mmol, 3.0 eq) in DCM (3 ml) at room temperature. The suspension was stirred for 2 h, then a solution of methyl 3-methoxyisoxazole-5-carboxylate (1.1 g, 7.0 mmol, 1.0 eq) in dichloromethane (DCM) (10 ml) was added. After 48 h stirring under reflux, the mixture was cooled to room temperature and partitioned between water (30 ml) and DCM (40 ml). The organic layer was separated and washed with water (50 ml). The aqueous layer was extracted with DCM (3×50 ml). The combined organic layers were washed with brine (50 ml), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The resulting yellow residue was purified by silica gel column chromatography (petroleum ether/DCM, 50:50) to afford methyl 3-methoxy-4-nitroisoxazole-5-carboxylate (0.9 g, 70%) as yellowish oil: $R_f = 0.41$ (petroleum ether/ Et_2O , 80:20, UV/ $KMnO_4$); 1H (500 MHz, $CDCl_3$), δ : (p.p.m): 4.14 (3H, s), 4.02 (3H, s); ^{13}C (125 MHz, $CDCl_3$), δ : (p.p.m): 164.0, 157.4, 155.0, 127.7, 58.9, 54.2; HRMS m/z (APCI⁺), found: $[M + H]^+$ 203.0295, $C_6H_7N_2O_6$ requires $[M + H]^+$ 203.0299.

Synthesis of methyl 4-amino-3-methoxyisoxazole-5-carboxylate

Iron powder (267 mg, 4.86 mmol, 5.0 eq) was added to a solution of methyl 3-methoxy-4-nitroisoxazole-5-carboxylate (196 mg, 0.97 mmol, 1.0 eq) in $AcOH/H_2O$ ($AcOH$ = acetic acid) (3:1 v/v mixture, 12 ml). After stirring at 50°C for 2 h, the solution was cooled to room temperature and the solvent was removed under reduced pressure. The residue was partitioned between water (20 ml) and ethyl acetate ($EtOAc$) (20 ml). The

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₆ H ₈ N ₂ O ₄
<i>M_r</i>	172.14
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.0425 (18), 11.555 (3), 9.654 (2)
β (°)	106.629 (6)
<i>V</i> (Å ³)	752.7 (3)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.13
Crystal size (mm)	0.27 × 0.06 × 0.06
Data collection	
Diffractometer	Rigaku XtaLAB P200
Absorption correction	Multi-scan (<i>CrystalClear</i> ; Rigaku, 2014)
<i>T</i> _{min} , <i>T</i> _{max}	0.695, 0.992
No. of measured, independent and observed [<i>F</i> ² > 2.0 σ (<i>F</i> ²)] reflections	9048, 1388, 1223
<i>R</i> _{int}	0.051
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.604
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.030, 0.083, 1.09
No. of reflections	1388
No. of parameters	119
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.17, -0.20

Computer programs: *CrystalClear* and *CrystalStructure* (Rigaku, 2014), *SIR2011* (Burla *et al.*, 2012), *SHELXL2018/3* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

mixture was basified with a saturated aqueous solution of Na₂CO₃ and further extracted with EtOAc (3 × 20 ml). The combined organic layers were washed with brine (20 ml), dried over MgSO₄, filtered and concentrated under reduced pressure to afford a pale-yellow solid, which was purified by silica gel column chromatography (DCM, 100), affording methyl 4-amino-3-methoxyisoxazole-5-carboxylate (139 mg, 83%) as a colourless crystalline solid: *R*_f = 0.74 (DCM/EtOAc, 90:10, UV/ninhydrin); m.p. 111–112°C; ¹H (500 MHz, CDCl₃), δ : (p.p.m): 4.15 (*br s*, 2H), 4.05 (*3H, s*), 3.92 (*3H, s*); ¹³C (125 MHz, CDCl₃), δ : (p.p.m): 164.5, 159.1, 138.4, 125.6, 57.5, 51.9; HRMS *m/z* (ESI⁺), found: [*M* + Na]⁺ 195.0373, C₆H₈N₂O₄Na requires [*M* + Na]⁺ 195.0382.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

The authors acknowledge Universiti Teknologi MARA for financial support.

References

- Abdul Manan, M. A. F., Cordes, D. B., Slawin, A. M. Z. & O'Hagan, D. (2023). *IUCrData*, **8**, x230300.
- Bąchor, U., Ryng, S., Mączyński, M., Artym, J., Kocięba, M., Zaczyńska, E., Kochanowska, I., Tykarska, E. & Zimecki, M. (2019). *Acta Pol. Pharm.* **76**, 251–263.
- Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Mallamo, M., Mazzzone, A., Polidori, G. & Spagna, R. (2012). *J. Appl. Cryst.* **45**, 357–361.
- Kauhanka, U. M. & Kauhanka, M. M. (2006). *Liq. Cryst.* **33**, 121–127.
- Lal, S., Staples, R. J. & Shreeve, J. M. (2023). *Dalton Trans.* **52**, 3449–3457.
- Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
- Melikian, A., Schlewer, G., Chambon, J. P. & Wermuth, C. G. (1992). *J. Med. Chem.* **35**, 4092–4097.
- Pu, S., Li, H., Liu, G., Liu, W., Cui, S. & Fan, C. (2011). *Tetrahedron*, **67**, 438–447.
- Regiec, A., Wojciechowski, P., Pietraszko, A. & Mączyński, M. (2018). *J. Mol. Struct.* **1161**, 320–338.
- Rigaku (2014). *CrystalStructure* and *CrystalClear*. Rigaku Americas Corporation, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
- Schmitt, E., Bouvet, S., Pégot, B., Panossian, A., Vors, J.-P., Pazenok, S., Magnier, E. & Leroux, F. R. (2017). *Org. Lett.* **19**, 4960–4963.
- Schmitt, E., Rugeri, B., Panossian, A., Vors, J.-P., Pazenok, S. & Leroux, F. R. (2015). *Org. Lett.* **17**, 4510–4513.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Sony, S. M. M., Charles, P., Ponnuswamy, M. N., Yathirajan, H. S. & Nethaji, M. (2005). *Acta Cryst.* **E61**, o198–o200.
- Wang, L., Huang, M., Wu, Z., Huang, M., Yan, Y., Song, B., Li, X. & Li, Q. X. (2022). *J. Agric. Food Chem.* **70**, 4871–4880.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Yoon, J., Liu, X. & Lee, E. C. (2022). *ACS Omega*, **7**, 34278–34285.
- Zhu, J., Mo, J., Lin, H. Z., Chen, Y. & Sun, H. P. (2018). *Bioorg. Med. Chem.* **26**, 3065–3075.

full crystallographic data

IUCrData (2023). **8**, x230623 [https://doi.org/10.1107/S2414314623006235]

Methyl 4-amino-3-methoxyisoxazole-5-carboxylate

Mohd Abdul Fatah Abdul Manan, David B. Cordes and Alexandra M. Z. Slawin

Methyl 4-amino-3-methoxyisoxazole-5-carboxylate

Crystal data

$C_6H_8N_2O_4$	$F(000) = 360.00$
$M_r = 172.14$	$D_x = 1.519 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71075 \text{ \AA}$
$a = 7.0425 (18) \text{ \AA}$	Cell parameters from 2384 reflections
$b = 11.555 (3) \text{ \AA}$	$\theta = 2.8\text{--}25.4^\circ$
$c = 9.654 (2) \text{ \AA}$	$\mu = 0.13 \text{ mm}^{-1}$
$\beta = 106.629 (6)^\circ$	$T = 173 \text{ K}$
$V = 752.7 (3) \text{ \AA}^3$	Prism, colorless
$Z = 4$	$0.27 \times 0.06 \times 0.06 \text{ mm}$

Data collection

Rigaku XtaLAB P200 diffractometer	$T_{\min} = 0.695$, $T_{\max} = 0.992$
Radiation source: Rotating Anode, Rigaku FR-X	9048 measured reflections
Rigaku Osmic Confocal Optical System monochromator	1388 independent reflections
Detector resolution: $11.628 \text{ pixels mm}^{-1}$	1223 reflections with $F^2 > 2.0\sigma(F^2)$
ω scans	$R_{\text{int}} = 0.051$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2014)	$\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 2.8^\circ$
	$h = -8 \rightarrow 8$
	$k = -13 \rightarrow 13$
	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.030$	Hydrogen site location: mixed
$wR(F^2) = 0.083$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.09$	$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.1362P]$
1388 reflections	where $P = (F_o^2 + 2F_c^2)/3$
119 parameters	$(\Delta/\sigma)_{\max} < 0.001$
2 restraints	$\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$

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.

Refinement. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2 . R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0 \sigma(F^2)$ is used only for calculating R-factor (gt).

Carbon-bound H atoms were included in calculated positions (C—H = 0.98 Å) and refined as riding atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The nitrogen-bound hydrogen atoms were located from difference Fourier maps and refined isotropically subject to a distance restraint.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	−0.02039 (14)	0.26697 (7)	0.62559 (10)	0.0332 (3)
O3	0.32430 (13)	0.09325 (8)	0.53449 (10)	0.0333 (3)
O6	−0.30018 (14)	0.23906 (8)	0.77949 (11)	0.0376 (3)
O7	−0.19751 (14)	0.05355 (8)	0.82206 (10)	0.0355 (3)
N2	0.12840 (17)	0.25137 (9)	0.55608 (12)	0.0321 (3)
N4	0.11557 (19)	−0.02825 (9)	0.71104 (14)	0.0380 (3)
H4A	0.201 (2)	−0.0715 (13)	0.6805 (17)	0.044 (4)*
H4B	0.039 (2)	−0.0582 (14)	0.7641 (17)	0.046 (4)*
C3	0.18521 (18)	0.14406 (10)	0.58185 (13)	0.0275 (3)
C4	0.08233 (18)	0.08329 (10)	0.66715 (13)	0.0271 (3)
C5	−0.04431 (18)	0.16376 (10)	0.69055 (14)	0.0289 (3)
C6	−0.19340 (18)	0.16034 (10)	0.76742 (14)	0.0297 (3)
C7	−0.3398 (2)	0.03562 (12)	0.90147 (17)	0.0396 (4)
H7A	−0.313327	0.089774	0.982892	0.048*
H7B	−0.329201	−0.043968	0.937968	0.048*
H7C	−0.473727	0.048846	0.837561	0.048*
C8	0.4105 (2)	0.16335 (12)	0.44464 (16)	0.0375 (3)
H8A	0.471816	0.232123	0.498567	0.045*
H8B	0.306859	0.187186	0.357868	0.045*
H8C	0.511435	0.118384	0.416398	0.045*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0415 (5)	0.0222 (4)	0.0413 (5)	0.0044 (4)	0.0205 (4)	0.0028 (4)
O3	0.0375 (5)	0.0275 (5)	0.0410 (6)	0.0027 (4)	0.0211 (4)	0.0030 (4)
O6	0.0383 (5)	0.0303 (5)	0.0495 (6)	0.0026 (4)	0.0209 (5)	−0.0031 (4)
O7	0.0399 (5)	0.0281 (5)	0.0451 (6)	−0.0013 (4)	0.0227 (4)	0.0007 (4)
N2	0.0385 (6)	0.0262 (6)	0.0360 (6)	0.0012 (4)	0.0178 (5)	0.0013 (4)
N4	0.0473 (7)	0.0223 (6)	0.0534 (8)	0.0053 (5)	0.0291 (6)	0.0077 (5)
C3	0.0299 (6)	0.0245 (6)	0.0291 (7)	−0.0004 (5)	0.0102 (5)	−0.0018 (5)
C4	0.0294 (7)	0.0231 (6)	0.0283 (6)	−0.0013 (5)	0.0077 (5)	−0.0013 (5)
C5	0.0340 (7)	0.0224 (6)	0.0309 (7)	−0.0018 (5)	0.0104 (6)	−0.0006 (5)
C6	0.0316 (7)	0.0260 (6)	0.0318 (7)	−0.0026 (5)	0.0098 (5)	−0.0042 (5)
C7	0.0421 (8)	0.0377 (8)	0.0469 (9)	−0.0057 (6)	0.0252 (7)	0.0002 (6)
C8	0.0398 (8)	0.0368 (7)	0.0427 (8)	0.0006 (6)	0.0227 (6)	0.0057 (6)

Geometric parameters (Å, °)

O1—C5	1.3801 (15)	N4—H4B	0.910 (14)
O1—N2	1.4083 (15)	C3—C4	1.4275 (18)
O3—C3	1.3302 (15)	C4—C5	1.3514 (17)
O3—C8	1.4417 (16)	C5—C6	1.4493 (19)
O6—C6	1.2070 (15)	C7—H7A	0.9800
O7—C6	1.3456 (16)	C7—H7B	0.9800
O7—C7	1.4403 (17)	C7—H7C	0.9800
N2—C3	1.3048 (16)	C8—H8A	0.9800
N4—C4	1.3560 (17)	C8—H8B	0.9800
N4—H4A	0.893 (13)	C8—H8C	0.9800
C5—O1—N2	108.07 (9)	O6—C6—O7	124.71 (12)
C3—O3—C8	115.92 (10)	O6—C6—C5	126.27 (12)
C6—O7—C7	115.91 (10)	O7—C6—C5	109.01 (10)
C3—N2—O1	105.08 (10)	O7—C7—H7A	109.5
C4—N4—H4A	120.1 (11)	O7—C7—H7B	109.5
C4—N4—H4B	117.4 (11)	H7A—C7—H7B	109.5
H4A—N4—H4B	122.2 (15)	O7—C7—H7C	109.5
N2—C3—O3	124.73 (11)	H7A—C7—H7C	109.5
N2—C3—C4	113.51 (11)	H7B—C7—H7C	109.5
O3—C3—C4	121.76 (11)	O3—C8—H8A	109.5
C5—C4—N4	131.68 (12)	O3—C8—H8B	109.5
C5—C4—C3	103.07 (11)	H8A—C8—H8B	109.5
N4—C4—C3	125.24 (11)	O3—C8—H8C	109.5
C4—C5—O1	110.27 (11)	H8A—C8—H8C	109.5
C4—C5—C6	132.56 (12)	H8B—C8—H8C	109.5
O1—C5—C6	117.18 (11)		
C5—O1—N2—C3	−0.22 (13)	N4—C4—C5—C6	−1.7 (2)
O1—N2—C3—O3	179.64 (11)	C3—C4—C5—C6	179.23 (13)
O1—N2—C3—C4	−0.08 (14)	N2—O1—C5—C4	0.46 (14)
C8—O3—C3—N2	−2.12 (18)	N2—O1—C5—C6	−179.30 (10)
C8—O3—C3—C4	177.58 (11)	C7—O7—C6—O6	−0.80 (19)
N2—C3—C4—C5	0.35 (14)	C7—O7—C6—C5	−179.66 (10)
O3—C3—C4—C5	−179.38 (11)	C4—C5—C6—O6	−179.16 (14)
N2—C3—C4—N4	−178.81 (12)	O1—C5—C6—O6	0.53 (19)
O3—C3—C4—N4	1.45 (19)	C4—C5—C6—O7	−0.32 (19)
N4—C4—C5—O1	178.61 (13)	O1—C5—C6—O7	179.37 (10)
C3—C4—C5—O1	−0.48 (13)		

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>
N4—H4A \cdots O6 ⁱ	0.89 (1)	2.30 (2)	2.9765 (16)	133 (1)

N4—H4B···O1 ⁱ	0.91 (1)	2.31 (2)	3.0233 (15)	136 (1)
N4—H4B···O7	0.91 (1)	2.30 (2)	2.8734 (16)	121 (1)

Symmetry code: (i) $-x, y-1/2, -z+3/2$.