



ISSN 2414-3146

2-Amino-4-methoxy-6-methylpyrimidinium hydrogen phthalate

Ramalingam Sangeetha,^a Bellarmin Edison,^a Kanagasabapathy Thanikasalam,^b Savaridasson Jose Kavitha^c and Kasthuri Balasubramani^{a*}

^aDepartment of Chemistry, Government Arts College (Autonomous), Thanthonimalai, Karur 639 005, Tamil Nadu, India, ^bDepartment of Chemistry, Government Arts College, Tiruchirappalli 620 022, Tamil Nadu, India, and ^cDepartment of Chemistry, Mother Teresa Women's University, Kodaikanal 624 102, Tamil Nadu, India. *Correspondence e-mail: manavaibala@gmail.com

Received 24 April 2016

Accepted 15 May 2016

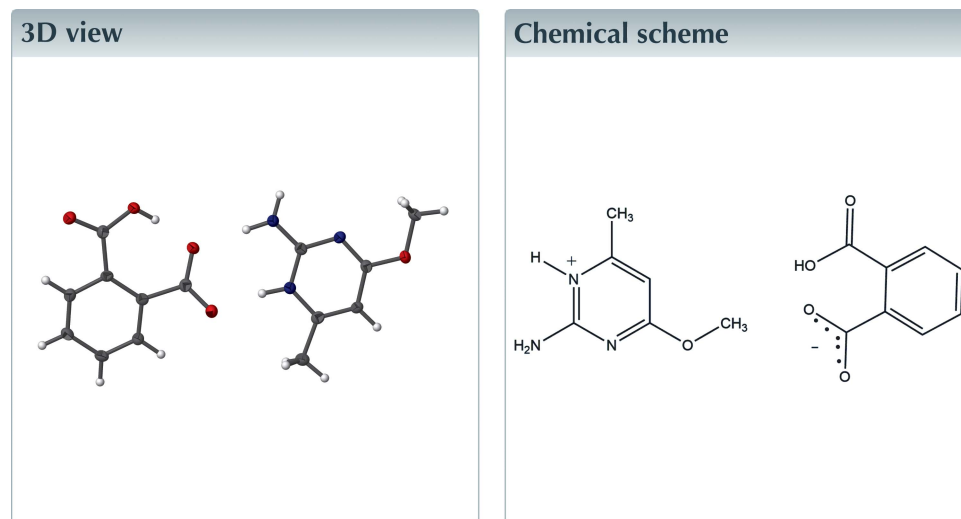
Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; hydrogen phthalate anion; pyrimidinium cation; N—H···O hydrogen bonding; π – π interactions.

CCDC reference: 1479857

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

In the hydrogen phthalate anion of the title molecular salt, $C_6H_{10}N_3O^+ \cdot C_8H_5O_4^-$, the dihedral angles formed by the benzene ring and the mean planes of the –COOH and –COO[–] groups are 16.1 (3) and 19.8 (3)°, respectively. There is an intramolecular O—H···O hydrogen bond in the anion generating an $S(7)$ ring motif. In the crystal, the protonated N atom of the pyrimidinium ring and the 2-amino group of the cation are hydrogen bonded to the carboxylate O atoms of the anion *via* a pair of N—H···O hydrogen bonds, forming an $R_2^2(8)$ ring motif. The ion pairs are further connected *via* N—H···O and C—H···O hydrogen bonds, forming ribbons parallel to the $[01\bar{1}]$ direction. The ribbons are linked by off-set π – π stacking interactions [intercentroid distances = 3.8279 (16) and 3.6074 (15) Å], forming a three-dimensional structure.



Structure description

Pyrimidine and aminopyrimidine derivatives are biologically very important compounds and they occur in nature as components of nucleic acids, such as cytosine, uracil and thymine. Pyrimidine derivatives have many applications in the areas of pesticide and pharmaceutical agents (Condon *et al.*, 1993). For example, imazosulfuron, ethirmol and mepanipyrim have been commercialized as agrochemicals (Maeno *et al.*, 1990). Pyrimidine derivatives have also been developed as antiviral agents, such as AZT, which is the most widely used anti-AIDS drug (Gilchrist, 1997). Phthalic acid forms hydrogen phthalate salts with various organic compounds. Hydrogen phthalates also form supra-molecular assemblies, such as extended chains, ribbons and three-dimensional networks (Dale *et al.*, 2004; Ballabh *et al.*, 2005). In order to study hydrogen-bonding interactions in

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H1N3 \cdots O2	0.94 (4)	1.80 (4)	2.738 (3)	172 (3)
N2—H1N2 \cdots O5 ⁱ	0.89 (4)	2.03 (4)	2.907 (3)	169 (3)
N2—H2N2 \cdots O3	0.94 (3)	1.91 (3)	2.846 (3)	177 (3)
O4—H1O4 \cdots O3	0.83 (3)	1.59 (3)	2.413 (3)	169 (3)
C6—H6A \cdots O5 ⁱⁱ	0.98	2.48	3.415 (4)	158

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

such molecular salts, we report herein on the synthesis and structure of the title molecular salt.

The molecular structure of the title molecular salt is illustrated in Fig. 1. In the hydrogen phthalate anion, there is a strong intramolecular O4—H1O4 \cdots O3 hydrogen bond enclosing an $S(7)$ ring (Fig. 1 and Table 1), which is a result of the negative charge-assisted effect described by Gilli *et al.* (1994). The proton transfers from the one of the carboxyl-group O atoms (O2) to atom N3 of form the 2-amino-4-methoxy-6-methylpyrimidinium cation, resulting in a widening of the C1—N3—C4 angle of the pyrimidinium ring to 121.2 (2)°, compared to the corresponding angle of 116.01 (18)° in neutral 2-amino-4-methoxy-6-methylpyrimidine (Glidewell *et al.*, 2003). The 2-amino-4-methoxy-6-methylpyrimidinium cation is essentially planar, with a maximum deviation of 0.006 (3) Å for atom C3. The carboxylate group of the hydrogen phthalate anion is slightly twisted from the attached ring, with the dihedral angle between the C7—C12 ring and the O2/O3/C14 plane being 19.8 (3)°.

In the crystal, the protonated N atom (N1) and the 2-amino group (N2) are hydrogen bonded to carboxylate O atoms (O2 and O3) via a pair of intermolecular N3—H1N3 \cdots O2 and N2—H2N2 \cdots O3 hydrogen bonds, forming an $R_2^2(8)$ ring motif (Fig. 2 and Table 1). Furthermore, these motifs are connected via N2—H1N2 \cdots O5ⁱ and weak C6—H6A \cdots O5ⁱⁱ hydrogen bonds (the symmetry codes are as in Table 1), forming ribbons parallel to the $[01\bar{1}]$ direction. The crystal structure is further stabilized by slipped parallel π – π interactions between inversion-related benzene rings of the anion [$Cg1\cdots Cg1^{iii} =$

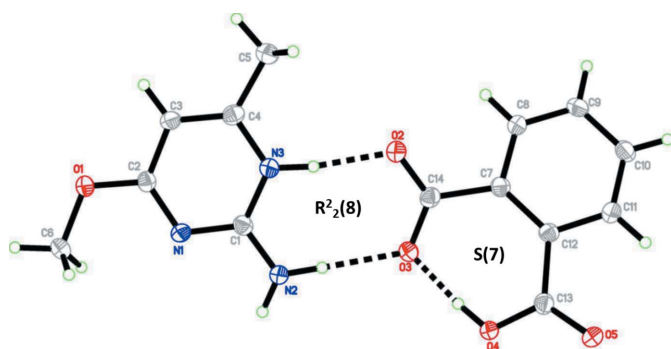


Figure 1
The molecular structure of the title molecular salt, showing the atom labelling and 50% probability displacement ellipsoids.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_6H_{10}N_3O^+ \cdot C_8H_5O_4^-$
M_r	305.29
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
a, b, c (Å)	7.3951 (4), 9.0021 (4), 10.5163 (4)
α, β, γ (°)	97.298 (3), 92.096 (3), 90.4322 (18)
V (Å ³)	693.90 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.11
Crystal size (mm)	0.65 × 0.24 × 0.06
Data collection	
Diffractometer	Bruker SMART APEXII CCD area-detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2009)
T_{\min}, T_{\max}	0.931, 0.994
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9218, 2391, 1802
R_{int}	0.050
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.058, 0.209, 1.12
No. of reflections	2391
No. of parameters	217
No. of restraints	1
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)	0.37, -0.38

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS97*, *SHELXTL* and *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

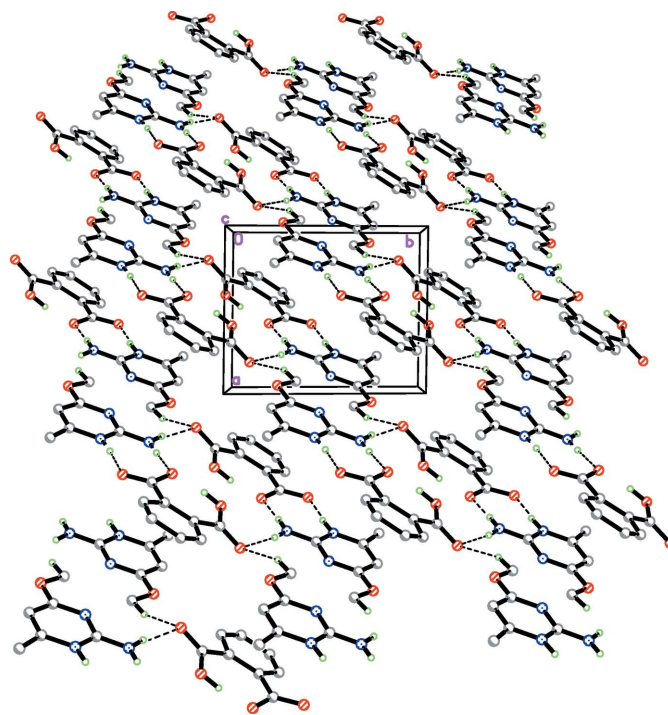


Figure 2
The crystal packing of the title compound, viewed along the c axis. H atoms not involved in the intermolecular interactions (dashed lines; see Table 1) have been omitted for clarity.

3.8279 (16) Å, interplanar distance = 3.465 (1) Å and slippage = 1.626 Å; Cg1 is the centroid of the C7–C12 ring; symmetry code: (iii) $-x + 1, -y + 2, -z + 2$] and between inversion-related pyrimidinium rings of the cation [$Cg2 \cdots Cg2^{iv}$ = 3.6074 (15) Å, interplanar distance = 3.275 (1) Å and slippage = 1.513 Å; Cg2 is the centroid of the N1/N3/C1–C4 ring; symmetry code: (iv) $-x, -y + 1, -z + 1$], forming a three-dimensional structure.

Synthesis and crystallization

A hot methanol solution (20 ml) of 2-amino-4-methoxy-6-methylpyrimidine (69 mg, Aldrich) and phthalic acid (41 mg, Merck) was mixed and warmed over a heating magnetic stirrer hotplate for a few minutes. The resulting solution was allowed to cool slowly to room temperature and crystals of the title salt appeared after a few days.

Refinement

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

Acknowledgements

RS thanks the Department of Science and Technology (DST), New Delhi, India, for financial support in the form of an INSPIRE fellowship (INSPIRE code No. IF131050).

References

- Ballabh, A., Trivedi, D. R. & Dastidar, P. (2005). *Cryst. Growth Des.* **5**, 1548–1553.
- Bruker (2009). *SADABS, APEX2 and SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Condon, M. E., Brady, T. E., Feist, D., Malefyt, T., Marc, P., Quakenbush, L. S., Rodaway, S. J., Shaner, D. L. & Tecle, B. (1993). *Brighton Crop Protection Conference on Weeds*, pp. 41–46. Alton, Hampshire, England: BCPC Publications.
- Dale, S. H., Elsegood, M. R. J., Hemmings, M. & Wilkinson, A. L. (2004). *CrystEngComm*, **6**, 207–214.
- Gilchrist, T. L. (1997). *Heterocyclic Chemistry*, 3rd ed., pp. 261–276. Singapore: Addison Wesley Longman.
- Gilli, P., Bertolasi, V., Ferretti, V. & Gilli, G. (1994). *J. Am. Chem. Soc.* **116**, 909–915.
- Glidewell, C., Low, J. N., Melguizo, M. & Quesada, A. (2003). *Acta Cryst.* **C59**, o9–o13.
- Maeno, S., Miura, I., Masuda, K. & Nagata, T. (1990). *Brighton Crop Protection Conference on Pests and Diseases*, pp. 415–422. Alton, Hampshire, England: BCPC Publications.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

full crystallographic data

IUCrData (2016). **1**, x160794 [doi:10.1107/S241431461600794X]

2-Amino-4-methoxy-6-methylpyrimidinium hydrogen phthalate

Ramalingam Sangeetha, Bellarmin Edison, Kanagasabapathy Thanikasalam, Savaridasson Jose Kavitha and Kasthuri Balasubramani

2-Amino-4-methoxy-6-methylpyrimidinium hydrogen phthalate

Crystal data

$C_6H_{10}N_3O^+ \cdot C_8H_5O_4^-$

$M_r = 305.29$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.3951(4) \text{ \AA}$

$b = 9.0021(4) \text{ \AA}$

$c = 10.5163(4) \text{ \AA}$

$\alpha = 97.298(3)^\circ$

$\beta = 92.096(3)^\circ$

$\gamma = 90.4322(18)^\circ$

$V = 693.90(5) \text{ \AA}^3$

$Z = 2$

$F(000) = 320$

$D_x = 1.461 \text{ Mg m}^{-3}$

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

Cell parameters from 3654 reflections

$\theta = 2.8\text{--}29.5^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Plate, colourless

$0.65 \times 0.24 \times 0.06 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.931$, $T_{\max} = 0.994$

9218 measured reflections

2391 independent reflections

1802 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -8 \rightarrow 8$

$k = -10 \rightarrow 10$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.058$

$wR(F^2) = 0.209$

$S = 1.12$

2391 reflections

217 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

$w = 1/[\sigma^2(F_o^2) + (0.1227P)^2 + 0.4813P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.37 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.38 \text{ e \AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.0588 (3)	0.2684 (2)	0.17366 (17)	0.0215 (5)
O2	0.4117 (3)	0.5765 (2)	0.70319 (17)	0.0240 (5)
O3	0.4156 (3)	0.7964 (2)	0.62996 (18)	0.0263 (5)
O4	0.5952 (3)	1.0238 (2)	0.66349 (18)	0.0252 (5)
O5	0.8160 (3)	1.1196 (2)	0.79417 (18)	0.0262 (5)
N1	0.0956 (3)	0.4729 (2)	0.2777 (2)	0.0184 (5)
N2	0.2541 (3)	0.6709 (3)	0.3901 (2)	0.0214 (6)
N3	0.2275 (3)	0.4535 (2)	0.4839 (2)	0.0192 (6)
C1	0.1924 (4)	0.5324 (3)	0.3839 (2)	0.0188 (6)
C2	0.0351 (4)	0.3341 (3)	0.2767 (2)	0.0179 (6)
C3	0.0649 (4)	0.2466 (3)	0.3776 (3)	0.0202 (6)
H3A	0.0172	0.1476	0.3729	0.024*
C4	0.1648 (4)	0.3099 (3)	0.4819 (3)	0.0204 (6)
C5	0.2110 (4)	0.2345 (3)	0.5968 (3)	0.0252 (7)
H5A	0.3427	0.2354	0.6115	0.038*
H5B	0.1662	0.1308	0.5825	0.038*
H5C	0.1546	0.2879	0.6720	0.038*
C6	-0.0780 (4)	0.3558 (3)	0.0676 (3)	0.0232 (7)
H6A	-0.1421	0.2960	-0.0049	0.035*
H6B	0.0421	0.3842	0.0416	0.035*
H6C	-0.1469	0.4462	0.0942	0.035*
C7	0.5555 (3)	0.7771 (3)	0.8393 (3)	0.0189 (6)
C8	0.5409 (4)	0.6919 (3)	0.9408 (3)	0.0202 (6)
H8A	0.4756	0.5996	0.9262	0.024*
C9	0.6175 (4)	0.7366 (3)	1.0616 (3)	0.0224 (6)
H9A	0.5996	0.6789	1.1298	0.027*
C10	0.7213 (4)	0.8674 (3)	1.0816 (3)	0.0226 (7)
H10A	0.7772	0.8987	1.1634	0.027*
C11	0.7428 (4)	0.9514 (3)	0.9827 (3)	0.0223 (6)
H11A	0.8174	1.0388	0.9971	0.027*
C12	0.6584 (3)	0.9126 (3)	0.8611 (3)	0.0178 (6)
C13	0.6944 (4)	1.0250 (3)	0.7689 (3)	0.0205 (6)
C14	0.4542 (4)	0.7115 (3)	0.7152 (3)	0.0198 (6)

H1N3	0.292 (5)	0.504 (4)	0.556 (4)	0.038 (10)*
H1N2	0.225 (5)	0.725 (4)	0.327 (4)	0.039 (10)*
H2N2	0.311 (5)	0.712 (4)	0.468 (3)	0.033 (9)*
H1O4	0.523 (4)	0.952 (3)	0.649 (4)	0.056 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0247 (11)	0.0213 (10)	0.0179 (10)	-0.0043 (8)	-0.0049 (8)	0.0024 (8)
O2	0.0271 (11)	0.0217 (11)	0.0227 (11)	-0.0045 (8)	-0.0059 (9)	0.0040 (8)
O3	0.0313 (12)	0.0238 (11)	0.0243 (11)	-0.0068 (9)	-0.0107 (9)	0.0086 (9)
O4	0.0298 (12)	0.0232 (11)	0.0233 (11)	-0.0097 (9)	-0.0102 (9)	0.0097 (9)
O5	0.0303 (12)	0.0229 (10)	0.0259 (11)	-0.0101 (9)	-0.0060 (9)	0.0079 (8)
N1	0.0183 (12)	0.0173 (11)	0.0197 (12)	-0.0032 (9)	0.0006 (9)	0.0038 (9)
N2	0.0260 (13)	0.0197 (12)	0.0187 (12)	-0.0053 (10)	-0.0039 (10)	0.0045 (10)
N3	0.0207 (12)	0.0190 (12)	0.0180 (12)	-0.0024 (9)	-0.0009 (10)	0.0032 (9)
C1	0.0165 (13)	0.0220 (14)	0.0183 (13)	-0.0007 (10)	-0.0002 (10)	0.0039 (11)
C2	0.0161 (13)	0.0205 (13)	0.0169 (13)	-0.0007 (10)	0.0008 (11)	0.0012 (11)
C3	0.0218 (14)	0.0173 (13)	0.0219 (14)	-0.0026 (11)	-0.0012 (12)	0.0048 (11)
C4	0.0179 (14)	0.0195 (14)	0.0241 (14)	-0.0004 (11)	0.0021 (11)	0.0040 (11)
C5	0.0279 (16)	0.0245 (15)	0.0241 (15)	-0.0021 (12)	-0.0026 (12)	0.0076 (11)
C6	0.0285 (15)	0.0244 (15)	0.0168 (13)	-0.0009 (12)	-0.0035 (11)	0.0047 (11)
C7	0.0140 (13)	0.0210 (14)	0.0221 (14)	0.0002 (10)	-0.0017 (11)	0.0055 (11)
C8	0.0203 (14)	0.0198 (13)	0.0205 (14)	-0.0031 (11)	-0.0015 (11)	0.0034 (11)
C9	0.0247 (15)	0.0234 (14)	0.0202 (14)	-0.0002 (11)	-0.0006 (12)	0.0080 (11)
C10	0.0259 (15)	0.0235 (14)	0.0183 (14)	0.0014 (12)	-0.0034 (11)	0.0037 (11)
C11	0.0221 (14)	0.0201 (14)	0.0243 (14)	-0.0034 (11)	-0.0040 (12)	0.0030 (11)
C12	0.0160 (13)	0.0165 (13)	0.0214 (14)	0.0028 (10)	-0.0007 (11)	0.0050 (10)
C13	0.0211 (14)	0.0191 (14)	0.0208 (14)	-0.0003 (11)	-0.0013 (11)	0.0010 (11)
C14	0.0172 (13)	0.0215 (14)	0.0205 (14)	-0.0029 (11)	0.0024 (11)	0.0022 (11)

Geometric parameters (Å, °)

O1—C2	1.335 (3)	C4—C5	1.490 (4)
O1—C6	1.448 (3)	C5—H5A	0.9800
O2—C14	1.243 (3)	C5—H5B	0.9800
O3—C14	1.276 (3)	C5—H5C	0.9800
O3—H1O4	1.588 (13)	C6—H6A	0.9800
O4—C13	1.306 (3)	C6—H6B	0.9800
O4—H1O4	0.834 (10)	C6—H6C	0.9800
O5—C13	1.232 (3)	C7—C8	1.399 (4)
N1—C2	1.323 (3)	C7—C12	1.421 (4)
N1—C1	1.353 (3)	C7—C14	1.529 (4)
N2—C1	1.318 (4)	C8—C9	1.382 (4)
N2—H1N2	0.90 (4)	C8—H8A	0.9500
N2—H2N2	0.94 (4)	C9—C10	1.390 (4)
N3—C1	1.360 (3)	C9—H9A	0.9500
N3—C4	1.367 (4)	C10—C11	1.374 (4)

N3—H1N3	0.95 (4)	C10—H10A	0.9500
C2—C3	1.412 (4)	C11—C12	1.405 (4)
C3—C4	1.360 (4)	C11—H11A	0.9500
C3—H3A	0.9500	C12—C13	1.516 (4)
C2—O1—C6	115.7 (2)	O1—C6—H6B	109.5
C14—O3—H1O4	114.1 (15)	H6A—C6—H6B	109.5
C13—O4—H1O4	114 (3)	O1—C6—H6C	109.5
C2—N1—C1	116.4 (2)	H6A—C6—H6C	109.5
C1—N2—H1N2	120 (2)	H6B—C6—H6C	109.5
C1—N2—H2N2	117 (2)	C8—C7—C12	118.2 (2)
H1N2—N2—H2N2	123 (3)	C8—C7—C14	114.5 (2)
C1—N3—C4	121.2 (2)	C12—C7—C14	127.3 (2)
C1—N3—H1N3	117 (2)	C9—C8—C7	122.6 (3)
C4—N3—H1N3	122 (2)	C9—C8—H8A	118.7
N2—C1—N1	118.7 (2)	C7—C8—H8A	118.7
N2—C1—N3	119.4 (2)	C8—C9—C10	118.9 (2)
N1—C1—N3	121.9 (2)	C8—C9—H9A	120.6
N1—C2—O1	119.2 (2)	C10—C9—H9A	120.6
N1—C2—C3	124.6 (2)	C11—C10—C9	119.9 (3)
O1—C2—C3	116.2 (2)	C11—C10—H10A	120.0
C4—C3—C2	117.1 (2)	C9—C10—H10A	120.0
C4—C3—H3A	121.4	C10—C11—C12	122.2 (2)
C2—C3—H3A	121.4	C10—C11—H11A	118.9
C3—C4—N3	118.7 (3)	C12—C11—H11A	118.9
C3—C4—C5	124.7 (3)	C11—C12—C7	118.0 (2)
N3—C4—C5	116.6 (2)	C11—C12—C13	113.1 (2)
C4—C5—H5A	109.5	C7—C12—C13	128.9 (2)
C4—C5—H5B	109.5	O5—C13—O4	119.6 (2)
H5A—C5—H5B	109.5	O5—C13—C12	120.1 (2)
C4—C5—H5C	109.5	O4—C13—C12	120.3 (2)
H5A—C5—H5C	109.5	O2—C14—O3	123.0 (2)
H5B—C5—H5C	109.5	O2—C14—C7	117.6 (2)
O1—C6—H6A	109.5	O3—C14—C7	119.3 (2)
C2—N1—C1—N2	-179.3 (2)	C8—C9—C10—C11	1.4 (4)
C2—N1—C1—N3	0.8 (4)	C9—C10—C11—C12	2.0 (4)
C4—N3—C1—N2	179.4 (2)	C10—C11—C12—C7	-3.4 (4)
C4—N3—C1—N1	-0.6 (4)	C10—C11—C12—C13	177.4 (2)
C1—N1—C2—O1	-178.9 (2)	C8—C7—C12—C11	1.3 (4)
C1—N1—C2—C3	0.0 (4)	C14—C7—C12—C11	-179.3 (2)
C6—O1—C2—N1	3.0 (3)	C8—C7—C12—C13	-179.6 (2)
C6—O1—C2—C3	-176.1 (2)	C14—C7—C12—C13	-0.1 (4)
N1—C2—C3—C4	-0.8 (4)	C11—C12—C13—O5	14.3 (4)
O1—C2—C3—C4	178.1 (2)	C7—C12—C13—O5	-164.8 (3)
C2—C3—C4—N3	0.9 (4)	C11—C12—C13—O4	-164.6 (2)
C2—C3—C4—C5	-179.6 (2)	C7—C12—C13—O4	16.2 (4)
C1—N3—C4—C3	-0.3 (4)	C8—C7—C14—O2	-18.8 (3)

C1—N3—C4—C5	-179.8 (2)	C12—C7—C14—O2	161.8 (3)
C12—C7—C8—C9	2.1 (4)	C8—C7—C14—O3	160.6 (2)
C14—C7—C8—C9	-177.4 (2)	C12—C7—C14—O3	-18.9 (4)
C7—C8—C9—C10	-3.5 (4)		

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>
N3—H1N3...O2	0.94 (4)	1.80 (4)	2.738 (3)	172 (3)
N2—H1N2...O5 ⁱ	0.89 (4)	2.03 (4)	2.907 (3)	169 (3)
N2—H2N2...O3	0.94 (3)	1.91 (3)	2.846 (3)	177 (3)
O4—H1O4...O3	0.83 (3)	1.59 (3)	2.413 (3)	169 (3)
C6—H6A...O5 ⁱⁱ	0.98	2.48	3.415 (4)	158

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