

Received 25 October 2015

Accepted 28 October 2015

Edited by P. C. Healy, Griffith University,
Australia**Keywords:** crystal structure; trichlorohydroxy-
ethyl; formamide; hydrogen bonding**CCDC reference:** 1009715**Supporting information:** this article has
supporting information at journals.iucr.org/e

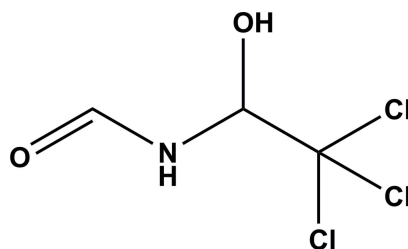
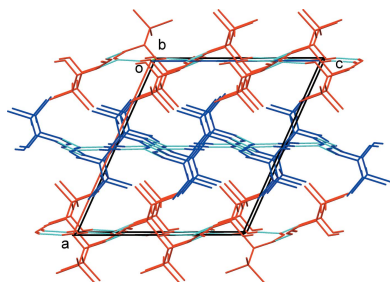
Crystal structure of *N*-(2,2,2-trichloro-1-hydroxyethyl)formamide

Mahimaidoss Baby Mariyatra^a and Helen Stoeckli-Evans^{b*}^aDepartment of Chemistry, St. Xavier's College, Palayamkottai 627 002, India, and ^bInstitute of Physics, University of Neuchâtel, rue Emile-Argand 11, CH-2000 Neuchâtel, Switzerland. *Correspondence e-mail: helen.stoeckli-evans@unine.ch, babymariyatra@gmail.com

The title compound, C₃H₄Cl₃NO₂, crystallized with two independent molecules (*A* and *B*) in the asymmetric unit. The two molecules have the same conformation; the molecular overlap gives weighted and unit-weight r.m.s. fits of 0.047 and 0.043 Å, respectively. The conformation of the *N*-(hydroxyethyl)formamide chains are very similar, as indicated by the C–N(H)–C=O and C–N(H)–C–O(H) torsion angles, which are, respectively, –1.8 (3) and –91.5 (2)° for molecule *A*, and –2.1 (3) and –95.7 (2)° for molecule *B*. In the crystal, individual molecules are linked by pairs of O–H···O hydrogen bonds, forming *A*–*A* and *B*–*B* inversion dimers with *R*₂²(12) ring motifs. The dimers are linked *via* N–H···O hydrogen bonds, forming alternating layers of *A* and *B* molecules parallel to the *bc* plane. Within the layers of *B* molecules, there are weak C–H···Cl hydrogen bonds present.

1. Chemical context

The skeletal structure of formamide is present in a number of medically important compounds. This has led to the use of formamides as key intermediates in numerous organic synthetic endeavours (Kobayashi *et al.*, 1995; Chen *et al.*, 2000; Jackson & Meth-Cohn, 1995). While formamides are useful formylating agents they have also found utility as easily accessible Lewis bases for promoting several organic transformations (Kobayashi & Nishio, 1994). Furthermore, in peptide synthesis the formyl group is a valued amino-protecting group (Martinez & Laur, 1982; Kraus, 1973). The title compound and related molecules have been found mentioned in several old patent literatures owing to their biocidal properties; both herbicidal (Schiewald *et al.*, 1974) and fungicidal (Summers & Carter, 1977) action is known. The title compound is easily obtained by the reaction of 2,2,2-trichloroacetaldehyde and formamide (Sethi, 2006) and we describe herein its crystal structure.



2. Structural commentary

The title compound, Fig. 1, crystallized with two independent molecules (*A* and *B*) in the asymmetric unit. The arbitrarily

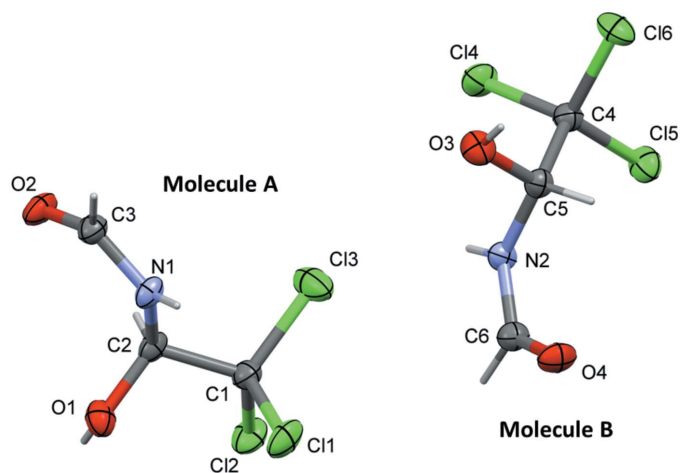


Figure 1
The molecular structure of the two independent molecules (*A* and *B*) of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The torsion angles C2–N1–C3–O2 and C3–N1–C2–O1 are $-1.8(3)$ and $-91.5(2)^\circ$, respectively, for molecule *A*, and C5–N2–C6–O4 and C6–N2–C5–O3 are $-2.1(3)$ and $-95.7(2)^\circ$, respectively, for molecule *B*.

chosen chirality of atoms C2 in molecule *A* and C5 in molecule *B* is the same. The backbones of the two molecules (O1/O3, C1/C4, C2/C5, N1/N2, C3/C6 and O2/O4) have almost identical conformations with weighted and unit-weight r.m.s. overlay fits of 0.047 and 0.043 Å, respectively, for the six atoms in each molecule (Fig. 2).

3. Supramolecular features

In the crystal, the individual molecules are linked by pairs of O–H···O hydrogen bonds, forming *A–A* and *B–B* inversion dimers with $R_2^2(12)$ ring motifs (Table 1 and Figs. 3 and 4). The

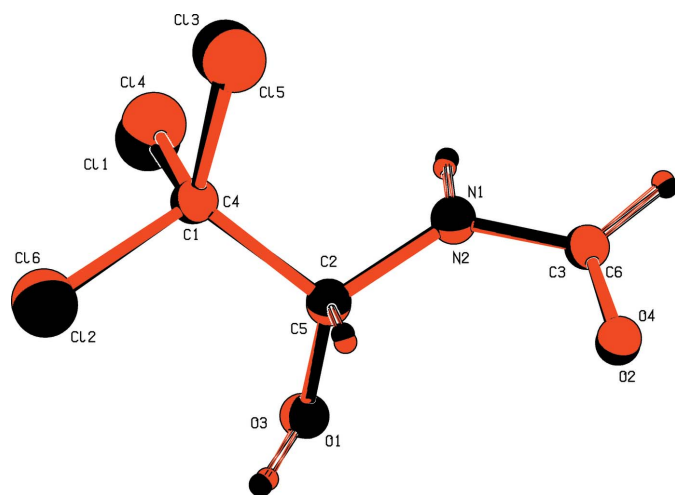


Figure 2
A view of the molecular fit of the six backbone atoms (O1/O3, C1/C4, C2/C5, N1/N2, C3/C6 and O2/O4) of the *A* (black) and *B* (red) molecules of the title compound, calculated using the MolFit routine in PLATON (Spek, 2009).

Table 1
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>
O1–H1O···O2 ⁱ	0.84 (3)	1.90 (4)	2.731 (2)	169 (3)
N1–H1N···O2 ⁱⁱ	0.85 (2)	2.08 (3)	2.893 (2)	159 (2)
O3–H3O···O4 ⁱⁱⁱ	0.76 (3)	1.97 (3)	2.721 (2)	174 (3)
N2–H2N···O4 ^{iv}	0.78 (3)	2.17 (3)	2.917 (2)	158 (2)
C6–H6···Cl4 ^v	1.00 (2)	2.91 (2)	3.586 (2)	125 (2)

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

dimers are linked *via* N–H···O hydrogen bonds, forming layers of *A* and *B* molecules parallel to the *bc* plane (Table 1 and Figs. 3 and 4). These latter hydrogen bonds lead to the formation of $R_6^4(20)$ ring motifs in each layer (Figs. 3 and 4). The layers stack alternately along the *a* axis, as shown in Fig. 5. Within the layers of *B* molecules there are weak C–H···Cl hydrogen bonds present (Table 1). There are no significant intermolecular interactions linking the layers.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.36, last update May 2015; Groom & Allen, 2014) for the acyclic substructure C(=O)–N(H)–C(OH), *viz.* *N*-(hydroxymethyl)formamide, yielded 25 hits. The majority

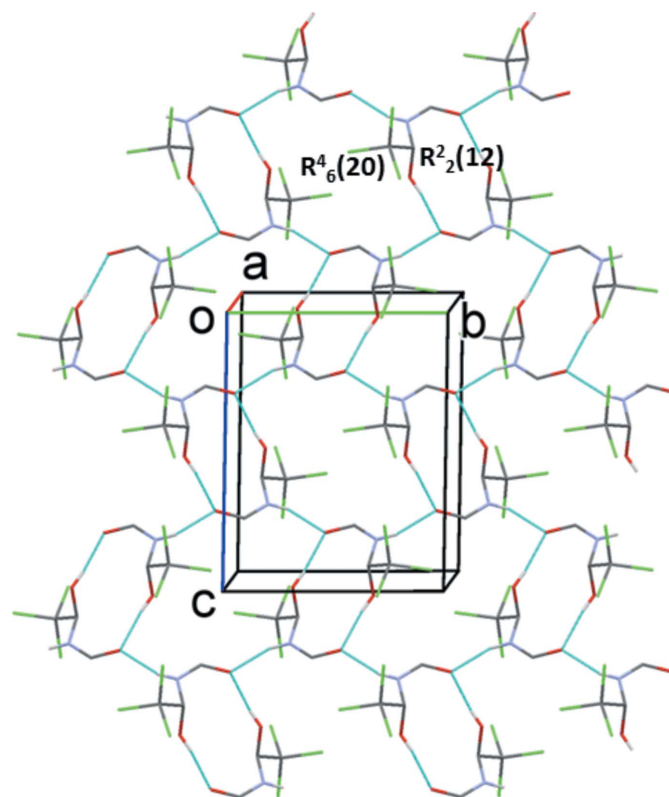


Figure 3
A view along the *a* axis of the hydrogen-bonded layer of *A* molecules of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1) and C-bound H atoms have been omitted for clarity.

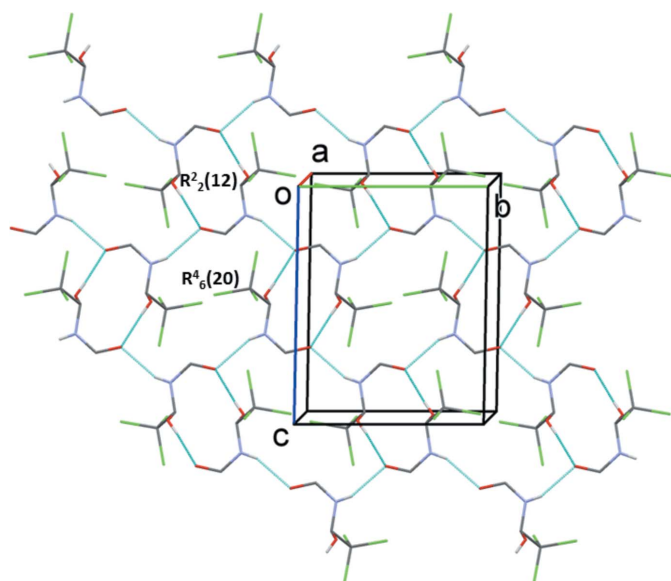


Figure 4
A view along the *a* axis of the hydrogen-bonded layer of *B* molecules of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1) and C-bound H atoms have been omitted for clarity.

concern metal complexes of the ligand *N*-(hydroxymethyl)nicotinamide. Only one compound, *N,N'*-(1,2-dihydroxyethylene)diformamide (OGEJUG; Taheri & Moosavi, 2008) resembles the title compound. In the solid state, the whole molecule of this compound is generated by inversion symmetry. The geometric parameters are similar to those observed for the title compound, for example the conformation of the *N*-(hydroxymethyl)formamide chain as indicated by the C–N(H)–C–O(H) and C–N(H)–C=O torsion angles: 1.6 (2) and -99.09 (14) $^\circ$ for the above mentioned compound compared to -1.8 (3) and -91.5 (2) $^\circ$ for molecule *A* and -2.1 (3) and -95.7 (2) $^\circ$ for molecule *B* of the title compound (see Fig. 1).

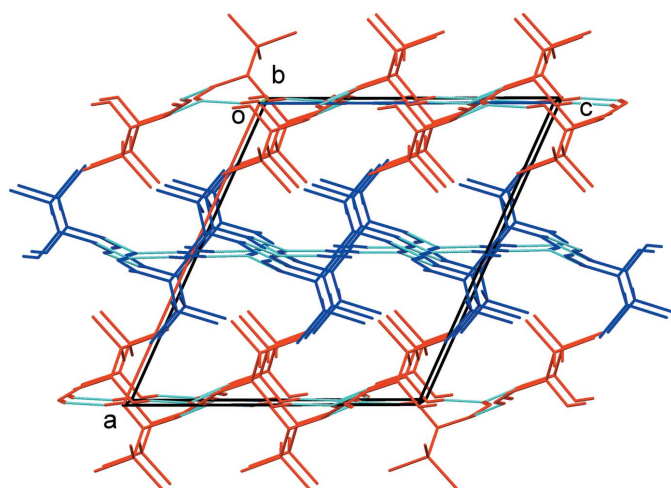


Figure 5
A view along the *b* axis of the crystal packing of the title compound, showing the alternating layers of hydrogen-bonded *A* (blue) and *B* (red) molecules. Hydrogen bonds are shown as dashed lines (see Table 1) and C-bound H atoms have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_3H_4Cl_3NO_2$
M_r	192.42
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	13.7964 (8), 9.0798 (7), 12.2453 (7)
β ($^\circ$)	114.413 (4)
<i>V</i> (Å ³)	1396.80 (16)
<i>Z</i>	8
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	1.24
Crystal size (mm)	0.45 × 0.43 × 0.40
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Multi-scan (<i>MULABS</i> in <i>PLATON</i> ; Spek, 2009)
T_{min} , T_{max}	0.579, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16347, 2645, 2468
R_{int}	0.056
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.610
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, <i>S</i>	0.030, 0.078, 1.08
No. of reflections	2645
No. of parameters	196
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	0.82, -0.55

Computer programs: *X-Area* and *X-RED32* (Stoe & Cie, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008).

5. Synthesis and crystallization

The title compound can be synthesized following a literature procedure (Sethi, 2006), by the reaction of 2,2,2-trichloroacetaldehyde and formamide. An old and discoloured sample of *N*-(2,2,2-trichloro-1-hydroxyethyl)formamide was dissolved in hot ethanol, followed by treatment with charcoal. The filtered solution was left to crystallize by slow evaporation, forming colourless block-like crystals (m.p. 393 K).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All of the H atoms were located from difference Fourier maps and freely refined.

Acknowledgements

MBM thanks the Department of Chemistry, St Xavier's College, for support of this work. HSE thanks the XRD Laboratory, CSEM, Neuchâtel, for access to the X-ray diffractometer.

References

- Chen, B.-C., Bednarz, M. S., Zhao, R., Sundeen, J. E., Chen, P., Shen, Z., Skoumbourdis, A. P. & Barrish, J. C. (2000). *Tetrahedron Lett.* **41**, 5453–5456.

- Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
- Jackson, A. & Meth-Cohn, O. (1995). *J. Chem. Soc. Chem. Commun.* pp. 1319.
- Kobayashi, K., Nagato, S., Kawakita, M., Morikawa, O. & Konishi, H. (1995). *Chem. Lett.* **24**, 575–576.
- Kobayashi, S. & Nishio, K. (1994). *J. Org. Chem.* **59**, 6620–6628.
- Kraus, N. A. (1973). *Synthesis*, pp. 361–362.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Martinez, J. & Laur, J. (1982). *Synthesis*, pp. 979–981.
- Schiewald, E., Naumann, K., Loettge, W., Kochmann, W., Pallas, M. & Hesse, B. (1974). Ger. Patent No. DD 109979.
- Sethi, A. (2006). *Systematic Lab Experiments in Organic Chemistry*, p. 514. New Age International (P) Ltd.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Stoe & Cie. (2009). *X-AREA and X-RED32*. Stoe & Cie GmbH, Darmstadt, Germany.
- Summers, L. A. & Carter, G. A. (1977). *Aust. J. Chem.* **30**, 663–668.
- Taheri, A. & Moosavi, S. M. (2008). *Acta Cryst.* **E64**, o2316.

supporting information

Acta Cryst. (2015). E71, 1501-1504 [https://doi.org/10.1107/S2056989015020459]

Crystal structure of *N*-(2,2,2-trichloro-1-hydroxyethyl)formamide

Mahimaidoss Baby Mariyatra and Helen Stoeckli-Evans

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2009); cell refinement: *X-AREA* (Stoe & Cie, 2009); data reduction: *X-RED32* (Stoe & Cie, 2009); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

N-(2,2,2-Trichloro-1-hydroxyethyl)formamide

Crystal data

$C_3H_4Cl_3NO_2$

$M_r = 192.42$

Monoclinic, $P2_1/c$

$a = 13.7964$ (8) Å

$b = 9.0798$ (7) Å

$c = 12.2453$ (7) Å

$\beta = 114.413$ (4)°

$V = 1396.80$ (16) Å³

$Z = 8$

$F(000) = 768$

$D_x = 1.830$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 22309 reflections

$\theta = 1.6$ – 26.2 °

$\mu = 1.24$ mm⁻¹

$T = 173$ K

Block, colourless

$0.45 \times 0.43 \times 0.40$ mm

Data collection

Stoe IPDS 2

diffractometer

Radiation source: fine-focus sealed tube

Plane graphite monochromator

$\varphi + \omega$ scans

Absorption correction: multi-scan

(MULABS in PLATON; Spek, 2009)

$T_{\min} = 0.579$, $T_{\max} = 1.000$

16347 measured reflections

2645 independent reflections

2468 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.056$

$\theta_{\max} = 25.7$ °, $\theta_{\min} = 1.6$ °

$h = -15 \rightarrow 16$

$k = -11 \rightarrow 11$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.078$

$S = 1.08$

2645 reflections

196 parameters

0 restraints

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0333P)^2 + 1.2964P]$

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

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

$\Delta\rho_{\max} = 0.82$ e Å⁻³

$\Delta\rho_{\min} = -0.55$ e Å⁻³

Extinction correction: SHELXL2014

(Sheldrick, 2015),

$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0089 (9)

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.13449 (5)	0.04880 (6)	0.08600 (5)	0.03428 (17)
C12	0.22308 (4)	0.28605 (6)	-0.00018 (5)	0.03123 (16)
C13	0.28102 (5)	0.25302 (8)	0.25345 (5)	0.04298 (19)
O1	-0.00275 (12)	0.31695 (18)	-0.01499 (14)	0.0284 (4)
H1O	0.005 (3)	0.366 (4)	-0.069 (3)	0.058 (10)*
O2	0.00144 (13)	0.53996 (16)	0.21058 (14)	0.0295 (4)
N1	0.04737 (14)	0.30393 (19)	0.18967 (16)	0.0213 (4)
H1N	0.0392 (18)	0.215 (3)	0.207 (2)	0.020 (6)*
C1	0.17653 (17)	0.2341 (2)	0.10963 (19)	0.0234 (4)
C2	0.08245 (16)	0.3364 (2)	0.09670 (18)	0.0217 (4)
H2	0.1107 (18)	0.434 (3)	0.1068 (19)	0.020 (6)*
C3	0.00885 (17)	0.4079 (2)	0.23751 (18)	0.0237 (4)
H3	-0.0156 (18)	0.374 (3)	0.299 (2)	0.026 (6)*
C14	0.64204 (5)	0.45558 (5)	0.52480 (5)	0.03177 (16)
C15	0.78172 (4)	0.22393 (6)	0.51229 (5)	0.02994 (15)
C16	0.71719 (4)	0.23240 (7)	0.70803 (5)	0.03280 (16)
O3	0.49315 (12)	0.20023 (18)	0.51142 (15)	0.0274 (3)
H3O	0.497 (2)	0.153 (3)	0.564 (3)	0.036 (8)*
O4	0.50422 (13)	-0.04575 (15)	0.29695 (13)	0.0281 (3)
N2	0.54593 (14)	0.19299 (19)	0.35510 (15)	0.0208 (4)
H2N	0.5415 (19)	0.274 (3)	0.332 (2)	0.023 (6)*
C4	0.67565 (16)	0.2671 (2)	0.55274 (18)	0.0216 (4)
C5	0.57819 (16)	0.1686 (2)	0.48142 (18)	0.0206 (4)
H5	0.6025 (16)	0.066 (2)	0.5015 (18)	0.012 (5)*
C6	0.51071 (16)	0.0854 (2)	0.27421 (18)	0.0230 (4)
H6	0.4911 (19)	0.119 (3)	0.190 (2)	0.028 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0493 (4)	0.0157 (3)	0.0449 (3)	0.0062 (2)	0.0265 (3)	0.0024 (2)
C12	0.0363 (3)	0.0317 (3)	0.0356 (3)	0.0029 (2)	0.0248 (2)	0.0016 (2)
C13	0.0250 (3)	0.0729 (5)	0.0282 (3)	0.0012 (3)	0.0081 (2)	-0.0095 (3)
O1	0.0292 (8)	0.0315 (9)	0.0259 (8)	0.0064 (6)	0.0127 (7)	0.0100 (7)
O2	0.0478 (10)	0.0170 (7)	0.0307 (8)	0.0078 (6)	0.0232 (7)	0.0031 (6)
N1	0.0268 (9)	0.0138 (8)	0.0279 (9)	0.0012 (7)	0.0159 (7)	0.0025 (7)
C1	0.0259 (10)	0.0227 (10)	0.0236 (10)	0.0014 (8)	0.0121 (9)	-0.0014 (8)
C2	0.0272 (10)	0.0144 (10)	0.0275 (10)	0.0016 (8)	0.0154 (8)	0.0010 (8)
C3	0.0288 (11)	0.0221 (10)	0.0229 (10)	0.0034 (8)	0.0133 (9)	0.0013 (8)

C14	0.0415 (3)	0.0152 (2)	0.0346 (3)	-0.0028 (2)	0.0117 (2)	-0.0021 (2)
C15	0.0247 (3)	0.0366 (3)	0.0304 (3)	0.0009 (2)	0.0133 (2)	0.0020 (2)
C16	0.0336 (3)	0.0425 (3)	0.0191 (3)	-0.0029 (2)	0.0077 (2)	0.0028 (2)
O3	0.0287 (8)	0.0303 (8)	0.0265 (8)	-0.0020 (6)	0.0150 (7)	0.0051 (7)
O4	0.0426 (9)	0.0170 (7)	0.0273 (8)	-0.0055 (6)	0.0171 (7)	-0.0019 (6)
N2	0.0271 (9)	0.0132 (8)	0.0202 (9)	-0.0017 (7)	0.0080 (7)	0.0025 (7)
C4	0.0253 (10)	0.0199 (10)	0.0199 (10)	-0.0008 (8)	0.0096 (8)	0.0008 (7)
C5	0.0253 (10)	0.0147 (10)	0.0216 (10)	-0.0018 (8)	0.0095 (8)	0.0016 (7)
C6	0.0270 (10)	0.0211 (10)	0.0217 (10)	-0.0021 (8)	0.0111 (8)	-0.0007 (8)

Geometric parameters (Å, °)

C11—C1	1.764 (2)	C14—C4	1.769 (2)
C12—C1	1.777 (2)	C15—C4	1.772 (2)
C13—C1	1.764 (2)	C16—C4	1.773 (2)
O1—C2	1.398 (3)	O3—C5	1.396 (3)
O1—H1O	0.84 (3)	O3—H3O	0.76 (3)
O2—C3	1.236 (3)	O4—C6	1.235 (3)
N1—C3	1.332 (3)	N2—C6	1.332 (3)
N1—C2	1.440 (3)	N2—C5	1.439 (3)
N1—H1N	0.85 (2)	N2—H2N	0.78 (3)
C1—C2	1.550 (3)	C4—C5	1.549 (3)
C2—H2	0.96 (2)	C5—H5	0.99 (2)
C3—H3	0.99 (2)	C6—H6	1.00 (2)
C2—O1—H1O	112 (2)	C5—O3—H3O	110 (2)
C3—N1—C2	121.88 (17)	C6—N2—C5	122.84 (17)
C3—N1—H1N	116.3 (16)	C6—N2—H2N	118.0 (18)
C2—N1—H1N	120.8 (16)	C5—N2—H2N	118.7 (18)
C2—C1—C13	110.37 (14)	C5—C4—C14	110.64 (14)
C2—C1—C11	110.49 (14)	C5—C4—C15	109.84 (14)
C13—C1—C11	109.52 (11)	C14—C4—C15	109.91 (11)
C2—C1—C12	108.24 (14)	C5—C4—C16	108.82 (14)
C13—C1—C12	109.15 (11)	C14—C4—C16	108.79 (11)
C11—C1—C12	109.04 (11)	C15—C4—C16	108.80 (11)
O1—C2—N1	109.04 (17)	O3—C5—N2	109.32 (16)
O1—C2—C1	110.74 (16)	O3—C5—C4	111.15 (16)
N1—C2—C1	109.73 (16)	N2—C5—C4	109.14 (16)
O1—C2—H2	112.1 (13)	O3—C5—H5	111.4 (12)
N1—C2—H2	109.8 (14)	N2—C5—H5	109.6 (12)
C1—C2—H2	105.3 (14)	C4—C5—H5	106.2 (12)
O2—C3—N1	125.03 (19)	O4—C6—N2	125.30 (19)
O2—C3—H3	119.1 (14)	O4—C6—H6	120.9 (14)
N1—C3—H3	115.9 (14)	N2—C6—H6	113.7 (14)
C3—N1—C2—O1	-91.5 (2)	C6—N2—C5—O3	-95.7 (2)
C3—N1—C2—C1	147.08 (19)	C6—N2—C5—C4	142.52 (19)
C13—C1—C2—O1	-177.16 (14)	C14—C4—C5—O3	-58.44 (19)

C11—C1—C2—O1	-55.9 (2)	C15—C4—C5—O3	-179.96 (13)
C12—C1—C2—O1	63.45 (19)	C16—C4—C5—O3	61.03 (19)
C13—C1—C2—N1	-56.7 (2)	C14—C4—C5—N2	62.20 (19)
C11—C1—C2—N1	64.54 (19)	C15—C4—C5—N2	-59.31 (19)
C12—C1—C2—N1	-176.13 (14)	C16—C4—C5—N2	-178.33 (14)
C2—N1—C3—O2	-1.8 (3)	C5—N2—C6—O4	-2.1 (3)

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>
O1—H1O...O2 ⁱ	0.84 (3)	1.90 (4)	2.731 (2)	169 (3)
N1—H1N...O2 ⁱⁱ	0.85 (2)	2.08 (3)	2.893 (2)	159 (2)
O3—H3O...O4 ⁱⁱⁱ	0.76 (3)	1.97 (3)	2.721 (2)	174 (3)
N2—H2N...O4 ^{iv}	0.78 (3)	2.17 (3)	2.917 (2)	158 (2)
C6—H6...Cl4 ^v	1.00 (2)	2.91 (2)	3.586 (2)	125 (2)

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