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Crystal structure of (5-chloro-2-hydroxyphenyl)(3-methylisoxazolo[5,4-*b*]pyridin-5-yl)methanone

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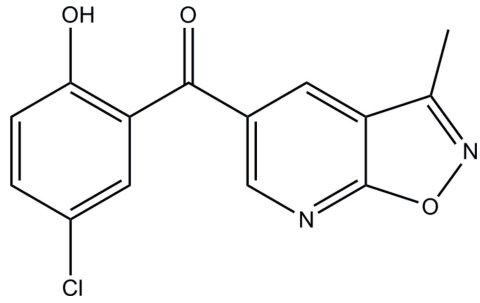
In the title compound, $C_{14}H_9ClN_2O_3$, the fused pyridine and isoxazole rings are approximately planar, making a dihedral angle of 1.14 (16)°. The molecule is twisted with the benzene ring and the mean plane through the fused pyridine-isoxazole ring system being inclined to one another by 47.03 (13)°. There is an intramolecular O—H···O hydrogen bond forming an *S*(6) ring motif. In the crystal, molecules are linked by C—H···N hydrogen bonds, forming chains propagating along [001]. The chains are linked by slipped parallel π–π interactions, involving inversion-related benzene rings, forming slabs lying parallel to the *bc* plane {inter-centroid distance = 3.770 (2) Å}.

Keywords: crystal structure; polyfunctional pyridines; isoxazole; O—H···O hydrogen bonds; C—H···N hydrogen bonds.

CCDC reference: 1431889

1. Related literature

For various applications of polyfunctional pyridines, see: Knyazhanskii *et al.* (1996); Kürfurst *et al.* (1989); Enyedy *et al.* (2003); Arora & Knaus (1999); Kim *et al.* 2004); Pillai *et al.*(2003).



2. Experimental

2.1. Crystal data

$C_{14}H_9ClN_2O_3$
M_r = 288.68
Monoclinic, $P2_1/c$
a = 11.0317 (10) Å
b = 11.8701 (10) Å
c = 11.1220 (9) Å
β = 118.675 (2)°
V = 1277.78 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 293 K
0.35 × 0.30 × 0.25 mm

2.2. Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.900, T_{max} = 0.927
17705 measured reflections
2250 independent reflections
1763 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.110
S = 1.13
2250 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82	1.84	2.561 (4)	145
C12—H12···N2^{i}	0.93	2.40	3.315 (4)	168

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Acknowledgements

The authors thank the Department of Chemistry, IIT, Chennai, India, for the data collection.

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5220).

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Crystal structure of (5-chloro-2-hydroxyphenyl)(3-methylisoxazolo[5,4-*b*]pyridin-5-yl)methanone

Rajamani Raja, Nataraj Poomathi, Paramasivam T. Perumal and A. SubbiahPandi

S1. Chemical context

Poly-functional pyridines are an interesting class of compounds due to their optical properties (Knyazhanskii *et al.*, 1996; Kürfurst *et al.*, 1989), and their biological activities (Enyedy *et al.*, 2003), such as anticonvulsants (Arora *et al.*, 1999), antihistaminic reagents (Kim *et al.*, 2004), and cardivascular disorder treatments (Pillai *et al.*, 2003). In view of such facts we herein report on the synthesis and crystal structure of the new title poly-functional pyridine compound.

S2. Structural commentary

In the title compound, Fig. 1, the fused pyridine ring (N1/C8—C12) and isoxazole ring (O3/N2/C13/C11/C10) are almost coplanar being inclined to one another by 1.14 (16) °. The molecule is twisted with the benzene ring (C1—C6) and the mean plane through the fused pyridine-isoxazole ring system being inclined to one another by 47.03 (13) °. The molecular conformation is partly determined by the intramolecular O—H···O hydrogen bond which forms an S(6) ring motif.

In the crystal, molecules are linked by C—H···N hydrogen bond to form chains propagating along the c-axis direction (Table 1 and Fig. 2). The chains are linked by slipped parallel π—π interactions, involving inversion related 5-chloro-2-hydroxyphenyl rings, forming slabs parallel to the *bc*-plane; see Fig. 2 [Cg3—Cg3^{i} = 3.770 (2) Å, inter-planar distance = 3.4094 (14) Å, slippage = 1.609 Å; Cg3 is the centroid of ring (C1—C6); symmetry code: (i) -x, -y, 2-z].

S3. Synthesis and crystallization

To a mixture of 6-chloro-3-formylchromone (1 mmol) and 3-methylisoxazol-5-amine (1 mmol) in ethanol (3 ml) was added a catalytic amount (0.050 mmol) of $In(OTf)_3$ and the mixture was refluxed for about 20 min. The precipitated solid was filtered and dried under vacuum to afford the pure product in 87% yield. The purified compound was recrystallised from ethanol and DMSO-D_6 by slow evaporation giving colourless block-like crystals.

S4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The OH and C-bound H atoms were positioned geometrically (O—H = 0.82 Å, C–H = 0.93–0.96 Å) and allowed to ride on their parent atoms, with $U_{iso}(H) = 1.5U_{eq}(O,C)$ for hydroxyl and methyl H atoms and $1.2U_{eq}(C)$ for other H atoms.

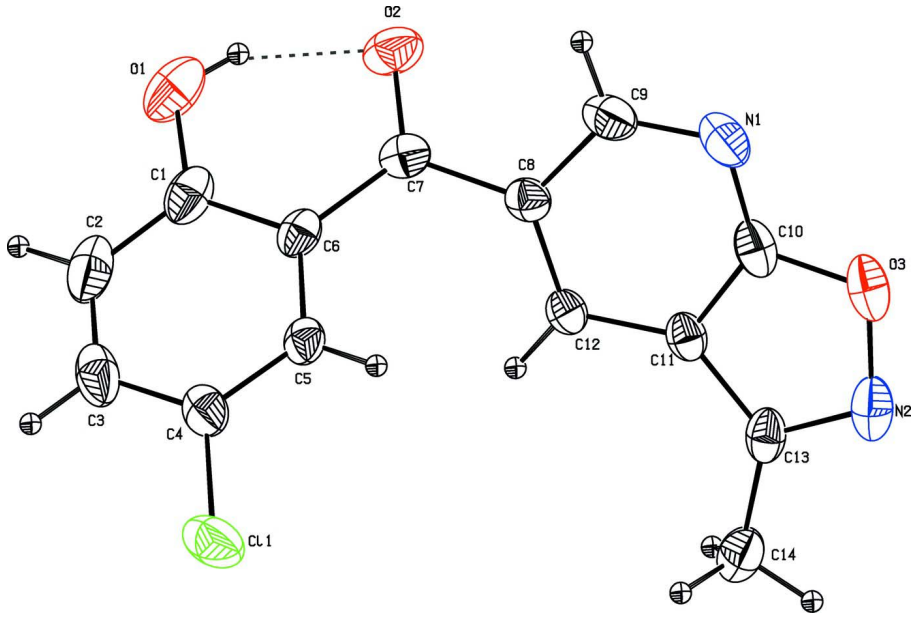

Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

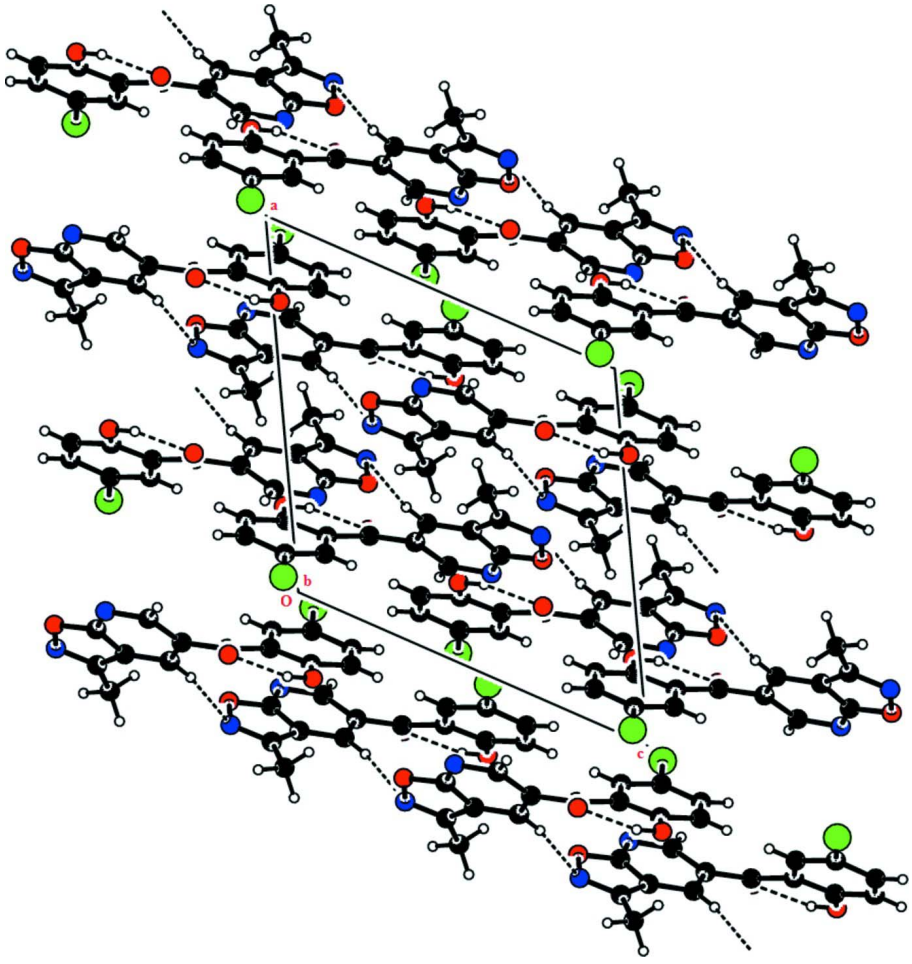

Figure 2

A view along the *b*-axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1 for details).

(5-Chloro-2-hydroxyphenyl)(3-methylisoxazolo[5,4-*b*]pyridin-5-yl)methanone

Crystal data

$C_{14}H_9ClN_2O_3$
$M_r = 288.68$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
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$b = 11.8701\ (10)$ Å
$c = 11.1220\ (9)$ Å
$\beta = 118.675\ (2)°$
$V = 1277.78\ (19)$ Å^3
$Z = 4$

$F(000) = 592$
$D_x = 1.501$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1763 reflections
$\theta = 2.1–25.0°$
$\mu = 0.31$ mm^{-1}
$T = 293$ K
Block, colourless
0.35 × 0.30 × 0.25 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator

ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.900$, $T_{max} = 0.927$

17705 measured reflections
2250 independent reflections
1763 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.110$
$S = 1.13$
2250 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 1.3834P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-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 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.1833 (3)	−0.0368 (3)	0.9816 (3)	0.0592 (8)
C2	0.1378 (3)	0.0264 (4)	0.8629 (3)	0.0738 (10)
H2	0.1386	−0.0051	0.7868	0.089*
C3	0.0917 (3)	0.1343 (3)	0.8560 (3)	0.0711 (10)
H3	0.0614	0.1755	0.7755	0.085*
C4	0.0900 (3)	0.1827 (3)	0.9696 (3)	0.0541 (7)
C5	0.1386 (2)	0.1234 (2)	1.0890 (2)	0.0432 (6)
H5	0.1384	0.1564	1.1648	0.052*
C6	0.1887 (2)	0.0133 (2)	1.0988 (3)	0.0436 (6)
C7	0.2418 (2)	−0.0531 (2)	1.2263 (3)	0.0455 (6)
C8	0.2796 (2)	−0.0001 (2)	1.3606 (3)	0.0424 (6)
C9	0.2544 (3)	−0.0637 (2)	1.4535 (3)	0.0578 (7)
H9	0.2125	−0.1336	1.4243	0.069*
C10	0.3469 (3)	0.0675 (3)	1.6116 (3)	0.0528 (7)
C11	0.3811 (2)	0.1373 (2)	1.5332 (2)	0.0414 (6)
C12	0.3455 (2)	0.1025 (2)	1.4011 (2)	0.0388 (6)
H12	0.3652	0.1460	1.3430	0.047*
C13	0.4447 (3)	0.2330 (2)	1.6185 (2)	0.0471 (6)
C14	0.5010 (3)	0.3350 (3)	1.5876 (3)	0.0607 (8)
H14A	0.5386	0.3840	1.6659	0.091*
H14B	0.4286	0.3733	1.5107	0.091*

H14C	0.5726	0.3140	1.5662	0.091*
N1	0.2861 (3)	−0.0312 (2)	1.5798 (3)	0.0656 (7)
N2	0.4480 (3)	0.2213 (2)	1.7365 (2)	0.0640 (7)
O1	0.2205 (3)	−0.1444 (2)	0.9793 (3)	0.0850 (8)
H1	0.2460	−0.1732	1.0547	0.127*
O2	0.2567 (2)	−0.15626 (16)	1.2243 (2)	0.0661 (6)
O3	0.3854 (2)	0.1149 (2)	1.73518 (19)	0.0709 (6)
Cl1	0.02472 (9)	0.31717 (7)	0.95930 (9)	0.0802 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0495 (17)	0.074 (2)	0.0569 (18)	−0.0167 (15)	0.0281 (14)	−0.0221 (16)
C2	0.071 (2)	0.106 (3)	0.0519 (19)	−0.033 (2)	0.0359 (17)	−0.0268 (19)
C3	0.061 (2)	0.102 (3)	0.0392 (16)	−0.0353 (19)	0.0150 (14)	0.0022 (17)
C4	0.0383 (14)	0.0663 (18)	0.0424 (15)	−0.0144 (13)	0.0070 (12)	0.0058 (13)
C5	0.0346 (13)	0.0518 (15)	0.0355 (13)	−0.0093 (11)	0.0106 (11)	−0.0045 (11)
C6	0.0340 (13)	0.0525 (15)	0.0442 (14)	−0.0113 (11)	0.0188 (11)	−0.0120 (12)
C7	0.0335 (13)	0.0425 (15)	0.0556 (16)	0.0001 (11)	0.0175 (12)	−0.0020 (12)
C8	0.0371 (13)	0.0408 (14)	0.0444 (14)	0.0072 (11)	0.0156 (11)	0.0055 (11)
C9	0.0558 (17)	0.0484 (16)	0.0618 (18)	0.0011 (13)	0.0222 (15)	0.0138 (14)
C10	0.0456 (16)	0.073 (2)	0.0380 (14)	0.0126 (14)	0.0183 (12)	0.0119 (14)
C11	0.0364 (13)	0.0506 (15)	0.0355 (13)	0.0095 (11)	0.0159 (11)	0.0066 (11)
C12	0.0336 (12)	0.0447 (14)	0.0365 (12)	0.0059 (11)	0.0154 (10)	0.0061 (11)
C13	0.0381 (14)	0.0623 (17)	0.0339 (13)	0.0125 (12)	0.0116 (11)	−0.0022 (12)
C14	0.0591 (18)	0.0647 (19)	0.0491 (16)	−0.0026 (15)	0.0185 (14)	−0.0124 (14)
N1	0.0707 (17)	0.0709 (18)	0.0530 (15)	0.0019 (14)	0.0277 (13)	0.0197 (13)
N2	0.0630 (16)	0.088 (2)	0.0388 (13)	0.0124 (14)	0.0224 (12)	−0.0035 (13)
O1	0.0909 (17)	0.0841 (17)	0.0906 (17)	−0.0042 (14)	0.0521 (15)	−0.0375 (14)
O2	0.0642 (13)	0.0443 (12)	0.0777 (15)	0.0077 (10)	0.0243 (11)	−0.0065 (10)
O3	0.0781 (15)	0.0984 (18)	0.0404 (11)	0.0073 (13)	0.0317 (11)	0.0092 (11)
Cl1	0.0684 (5)	0.0677 (5)	0.0732 (6)	−0.0036 (4)	0.0090 (4)	0.0258 (4)

Geometric parameters (Å, º)

C1—O1	1.345 (4)	C9—N1	1.331 (4)
C1—C2	1.386 (5)	C9—H9	0.9300
C1—C6	1.408 (4)	C10—N1	1.312 (4)
C2—C3	1.366 (5)	C10—O3	1.351 (3)
C2—H2	0.9300	C10—C11	1.380 (4)
C3—C4	1.396 (4)	C11—C12	1.390 (3)
C3—H3	0.9300	C11—C13	1.430 (4)
C4—C5	1.365 (4)	C12—H12	0.9300
C4—Cl1	1.733 (3)	C13—N2	1.302 (3)
C5—C6	1.403 (4)	C13—C14	1.474 (4)
C5—H5	0.9300	C14—H14A	0.9600
C6—C7	1.476 (4)	C14—H14B	0.9600
C7—O2	1.237 (3)	C14—H14C	0.9600

C7—C8	1.485 (4)	N2—O3	1.436 (3)
C8—C12	1.378 (3)	O1—H1	0.8200
C8—C9	1.411 (4)		

O1—C1—C2	118.0 (3)	N1—C9—H9	117.5
O1—C1—C6	122.8 (3)	C8—C9—H9	117.5
C2—C1—C6	119.2 (3)	N1—C10—O3	121.1 (3)
C3—C2—C1	121.1 (3)	N1—C10—C11	128.7 (3)
C3—C2—H2	119.5	O3—C10—C11	110.2 (3)
C1—C2—H2	119.5	C10—C11—C12	117.7 (3)
C2—C3—C4	120.1 (3)	C10—C11—C13	104.7 (2)
C2—C3—H3	120.0	C12—C11—C13	137.5 (2)
C4—C3—H3	120.0	C8—C12—C11	116.4 (2)
C5—C4—C3	120.0 (3)	C8—C12—H12	121.8
C5—C4—Cl1	119.7 (2)	C11—C12—H12	121.8
C3—C4—Cl1	120.4 (2)	N2—C13—C11	110.5 (3)
C4—C5—C6	120.8 (3)	N2—C13—C14	120.7 (3)
C4—C5—H5	119.6	C11—C13—C14	128.7 (2)
C6—C5—H5	119.6	C13—C14—H14A	109.5
C5—C6—C1	118.8 (3)	C13—C14—H14B	109.5
C5—C6—C7	122.2 (2)	H14A—C14—H14B	109.5
C1—C6—C7	119.0 (3)	C13—C14—H14C	109.5
O2—C7—C6	120.4 (2)	H14A—C14—H14C	109.5
O2—C7—C8	117.6 (2)	H14B—C14—H14C	109.5
C6—C7—C8	122.0 (2)	C10—N1—C9	112.6 (2)
C12—C8—C9	119.5 (2)	C13—N2—O3	107.7 (2)
C12—C8—C7	123.5 (2)	C1—O1—H1	109.5
C9—C8—C7	116.8 (2)	C10—O3—N2	106.9 (2)
N1—C9—C8	125.0 (3)		

O1—C1—C2—C3	176.6 (3)	C7—C8—C9—N1	177.5 (3)
C6—C1—C2—C3	−3.4 (5)	N1—C10—C11—C12	1.4 (4)
C1—C2—C3—C4	0.1 (5)	O3—C10—C11—C12	−178.4 (2)
C2—C3—C4—C5	2.1 (4)	N1—C10—C11—C13	179.9 (3)
C2—C3—C4—Cl1	−177.2 (2)	O3—C10—C11—C13	0.1 (3)
C3—C4—C5—C6	−0.9 (4)	C9—C8—C12—C11	−1.1 (3)
Cl1—C4—C5—C6	178.34 (18)	C7—C8—C12—C11	−176.2 (2)
C4—C5—C6—C1	−2.4 (4)	C10—C11—C12—C8	−0.4 (3)
C4—C5—C6—C7	−180.0 (2)	C13—C11—C12—C8	−178.3 (3)
O1—C1—C6—C5	−175.5 (2)	C10—C11—C13—N2	0.0 (3)
C2—C1—C6—C5	4.4 (4)	C12—C11—C13—N2	178.0 (3)
O1—C1—C6—C7	2.2 (4)	C10—C11—C13—C14	−179.6 (3)
C2—C1—C6—C7	−177.9 (3)	C12—C11—C13—C14	−1.6 (5)
C5—C6—C7—O2	164.1 (2)	O3—C10—N1—C9	179.3 (3)
C1—C6—C7—O2	−13.5 (4)	C11—C10—N1—C9	−0.6 (4)
C5—C6—C7—C8	−16.1 (4)	C8—C9—N1—C10	−1.2 (4)
C1—C6—C7—C8	166.3 (2)	C11—C13—N2—O3	0.0 (3)
O2—C7—C8—C12	140.6 (3)	C14—C13—N2—O3	179.6 (2)

C6—C7—C8—C12	−39.3 (4)	N1—C10—O3—N2	−179.9 (2)
O2—C7—C8—C9	−34.7 (3)	C11—C10—O3—N2	−0.1 (3)
C6—C7—C8—C9	145.5 (2)	C13—N2—O3—C10	0.0 (3)
C12—C8—C9—N1	2.1 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.84	2.561 (4)	145
C12—H12···N2^{i}	0.93	2.40	3.315 (4)	168

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