Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of (1*S*,2*S*,2′*R*,3a′*S*,5*R*)-2′-[(5-bromo-1*H*-indol-3-yl)methyl]-2-isopropyl-5,5′-dimethyldihydro-2′*H*-spiro[cyclohexane-1,6′-imidazo[1,5-*b*]isoxazol]-4′(5′*H*)-one

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Received 8 June 2016
Accepted 5 July 2016

Edited by G. Smith, Queensland University of Technology, Australia

Keywords: crystal structure; isoxazolidines; 1,3-dipolar cycloaddition; chiral nitrone; hydrogen bonding.

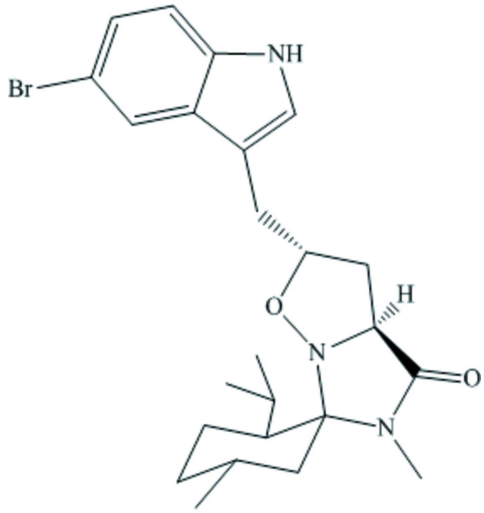
CCDC reference: 1490701

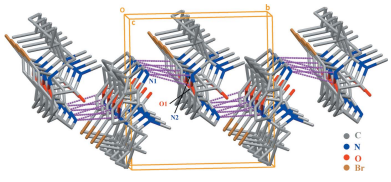
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compound, $C_{24}H_{32}BrN_3O_2$, the six-membered cyclohexane ring adopts a chair conformation and the isoxasolidine ring adopts a twisted conformation. The molecule has five chiral centres and the absolute configuration has been determined in this analysis. The molecular structure is stabilized by weak intramolecular C—H···O and C—H···N contacts. In the crystal, molecules are linked by N—H···N and C—H···O hydrogen bonds, forming undulating sheets parallel to the *bc* plane.

1. Chemical context

1,3-Dipolar cycloadditions of alkenes with nitrones produce substituted isoxazolidines. Nitrone cycloadducts offer a general route to natural and unnatural amino acids (Aouadi *et al.*, 2006, 2007) through opening of the isoxazolidine ring, usually by reductive cleavage of the weak N—O bond. Consequently, isoxazolidines have been used as key intermediates for the synthesis of various natural products, anti-fungals (Kumar *et al.*, 2003), anti-tuberculosis (Kumar *et al.*, 2010) and antiviral agents (Loh *et al.*, 2010). We present herein the synthesis, the molecular structure and the spectroscopic data of the title compound, $C_{24}H_{32}BrN_3O_2$, (I).





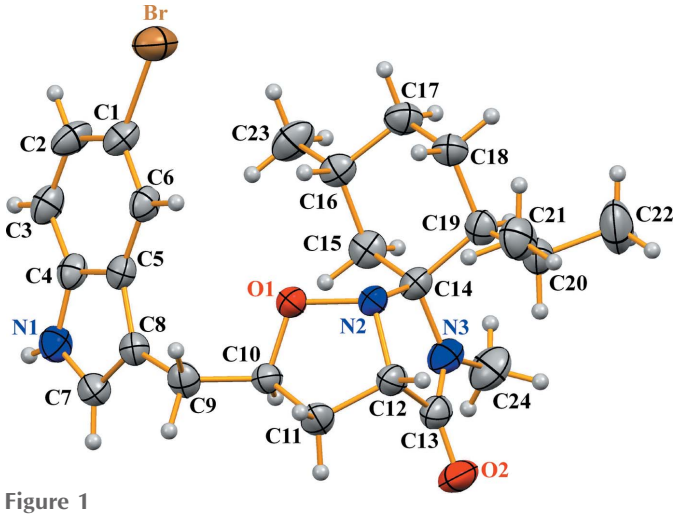


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 40% probability level.

2. Structural commentary

In the title compound (I) (Fig. 1), the five-membered isoxazolidine ring has a twist conformation. The O1—N2 bond length in the isoxazolidine ring is 1.475 (6) Å which is close to the values in related compounds (Lee *et al.*, 2010; Molander & Cavalcanti, 2013). The cyclohexane ring adopts a chair conformation. The dihedral angle between the mean planes of the isoxazolidine and imidazolidinone rings is 73.1 (3)° while the C8—C9—C10—O1 torsion angle is 74.7 (7)°. In the molecule there are some short C—H···O and C—H···N contacts present (Table 1). The absolute configuration of (I) has been confirmed as C10(*R*),C12(*S*),C14(*S*),C16(*R*),C19(*S*) for the five arbitrarily numbered chiral centres in the molecule.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H*N*1···N2^{i}	0.89	2.34	3.087 (8)	141
C3—H3···O2^{ii}	0.93	2.42	3.292 (9)	156
C16—H16···O1	0.98	2.55	3.091 (8)	115
C20—H20···N3	0.98	2.54	3.032 (10)	111
C21—H21*A*···N2	0.96	2.60	3.236 (9)	124

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

3. Supramolecular features

In the crystal packing of (I), the molecules are linked through an intermolecular N1—H*N*1···N2^{i} hydrogen bond (Table 1) and a weak N1—H*N*1···O1^{i} interaction [3.053 (8) Å], forming undulating sheets parallel to the *bc* plane (Fig. 2). Within the chains, the molecules are stabilized by a weak intermolecular C3—H3···O2^{ii} hydrogen bond (Table 1). Also present in the crystal are 39.3 Å^3 solvent-accessible voids.

4. Synthesis and crystallization

To a solution of 3-allyl-5-bromo-1*H*-indole (1.40 mmol, 330 mg) in toluene (10 mL) was added 5(*R*),6(*S*),9(*R*)-6-isopropyl-1,9-dimethyl-1,4-diazoaspiro[4,5]-decan-1-ene-3-one-1-oxide (II) (1.19 mmol, 285 mg) and the mixture was stirred and heated at reflux at 383 K for 24 h under argon. TLC indicated the complete conversion of (II). The solution obtained was concentrated and the residue was purified by flash chromatography (petroleum ether–ethyl acetate 7:3) to afford the cycloadduct (I) as a white solid (507 mg, 90% yield) (Fig. 3). Colorless plate-shaped crystals of (I) were obtained by slow evaporation of a diethyl ether solution.

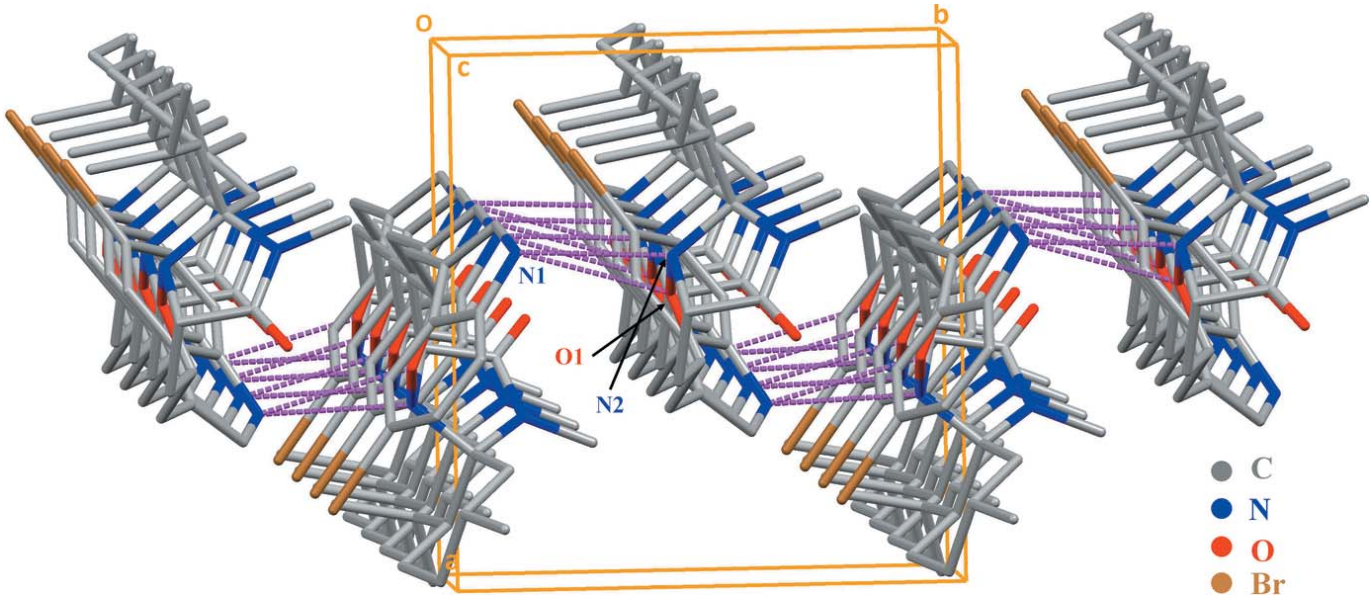


Figure 2
A view of the title structure, showing the molecules of the title compound arranged in zigzag parallel chains sustained by weak N—H···N and N—H···O hydrogen bonds.

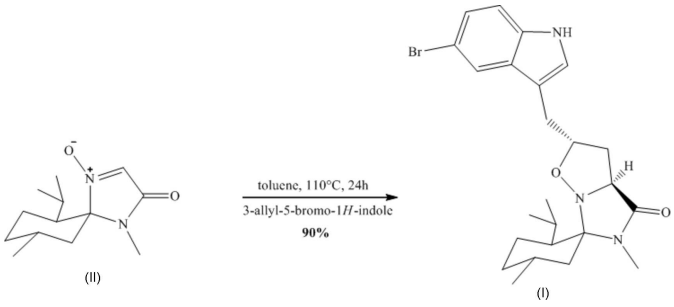


Figure 3
Reaction scheme for the synthesis of compound (I).

5. Spectroscopic investigations

NMR spectra were recorded on a Bruker Avance II 300 MHz spectrometer operating at 300 MHz for ^{1}H and 75.46 MHz for ^{13}C and were referenced to tetramethylsilane (δ = 0 p.p.m.). High-resolution (HR–ESI–QToF) mass spectra were recorded using a Bruker Micro ToF-Q II XL spectrometer.

The ^{1}H NMR spectrum of (I) shows the presence of an NH proton at 8.32 p.p.m. and the ^{13}C NMR spectrum confirms the existence of the C3 and C5 stereogenic centres at 66.4 p.p.m. and 78.0 p.p.m., respectively. The spectroscopic measurements are consistent with the crystal structure of (I). High-resolution mass spectrometry in the positive-ion mode exhibits an $[M+H]^+$ fragment of 474.1759 m/z which is very close to the calculated value of 474.1756 m/z.

R_f = 0.33 (PE–EtOAc 7:3). NMR ^{1}H (300 MHz, $CDCl_3$) δ(p.p.m.): 0.62 (*d*, 3H, *J* = 6.6 Hz), 0.83 (*d*, 3H, *J* = 6.6 Hz), 0.85 (*m*, 1H), 0.86 (*d*, 3H, *J* = 6.3 Hz), 1.11 (*t*, 1H, *J* = 12.3 Hz), 1.21–1.43 (*m*, 2H), 1.57–1.67 (*m*, 1H), 1.70–1.83 (*m*, 3H), 1.90–2.02 (*m*, 1H), 2.26 (*ddd*, 1H, *J* = 8.7 Hz, 10.2 Hz and 12 Hz), 2.69 (*s*, 3H, NCH_3), 2.67–2.72 (*m*, 1H), 2.93–2.97 (*m*, 2H), 3.88–3.97 (*m*, 1H), 4.01 (*brd*, 1H, *J* = 8.4 Hz), 7.02 (*brd*, 1H, *J* = 4.8 Hz), 7.21 (*m*, 2H), 7.74 (*brd*, 1H, *J* = 1.8 Hz), 8.32 (*brs*, 1H, NH). ^{13}C NMR ($CDCl_3$, 75.46 MHz) δ(p.p.m.): 18.3, 22.0, 22.2 (CH_2), 24.1, 24.3, 26.0, 28.1, 29.6, 34.5 (CH_2), 38.8 (CH_2), 40.3 (CH_2), 48.0, 66.4, 78.0, 90.0, 112.3, 112.4, 112.7, 121.6, 123.5, 124.7, 129.1, 134.6, 173.0 (C═O). [α] = + 43.7 (*c* = 1, CH_2Cl_2).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.98 Å (methine), 0.97 Å (methylene), 0.96 Å (methyl) and 0.93 Å (aromatic), with U_{iso}(H) = 1.2U_{eq}(C)(methine, methylene, aromatic) or 1.5U_{eq}C(methyl). The H atom on the nitrogen N1 of the indole ring was found in a difference-Fourier map but was subsequently refined with the coordinates and isotropic displacement parameter also riding with U_{iso} = 1.2 U_{eq}(N). The bond length N1—H*N*1 was restrained to ensure proper geometry using the DFIX instruction of *SHELXL2014/7* (Sheldrick, 2015). The absolute structure Flack parameter [−0.013 (13) for 1005 quotients (Parsons *et al.*, 2013)] confirmed the configuration of the molecule as C10(*R*),C12(*S*),C14(*S*),C16(*R*),C19(*S*) for the five arbitrarily numbered chiral centres in the molecule.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{32}BrN_3O_2$
M_r	474.43
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	293
a, *b*, *c* (Å)	10.2640 (5), 9.6480 (5), 12.0480 (5)
β (°)	96.204 (5)
V (Å^3)	1186.09 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.76
Crystal size (mm)	0.46 × 0.39 × 0.11
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Atlas Gemini Ultra CCD
Absorption correction	Multi-scan (*SCALEPACK*; Otwinowski *et al.*, 1997)
T_{min}, T_{max}	0.455, 0.802
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10653, 4337, 2924
R_{int}	0.104
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.053, 0.135, 0.97
No. of reflections	4337
No. of parameters	272
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.51
Absolute structure	Flack *x* determined using 1005 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.013 (13)

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SIR2011* (Burla *et al.*, 2012), *SHELXL2014* (Sheldrick, 2015), *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012).

Acknowledgements

The authors gratefully acknowledge financial support from the Ministry of Higher Education and Scientific Research of Tunisia.

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supporting information

Acta Cryst. (2016). E**72**, 1081-1084 [doi:10.1107/S2056989016010872]

Crystal structure of (1*S*,2*S*,2′*R*,3a′*S*,5*R*)-2′-[(5-bromo-1*H*-indol-3-yl)methyl]-2-isopropyl-5,5′-dimethyldihydro-2′*H*-spiro[cyclohexane-1,6′-imidazo[1,5-*b*]isoxazol]-4′(5′*H*)-one

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013); program(s) used to solve structure: *SIR2011* (Burla *et al.*, 2012); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

(1*S*,2*S*,2′*R*,3a′*S*,5*R*)-2′-[(5-Bromo-1*H*-indol-3-yl)methyl]-2-isopropyl-5,5′-dimethyldihydro-2′*H*-spiro[cyclohexane-1,6′-imidazo[1,5-*b*]isoxazol]-4′(5′*H*)-one

Crystal data

$C_{24}H_{32}BrN_3O_2$
$M_r = 474.43$
Monoclinic, $P2_1$
$a = 10.2640\ (5)$ Å
$b = 9.6480\ (5)$ Å
$c = 12.0480\ (5)$ Å
$\beta = 96.204\ (5)°$
$V = 1186.09\ (10)$ Å^3
$Z = 2$
$F(000) = 496$
$D_x = 1.328$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8154 reflections
$\theta = 1.0–27.9°$
$\mu = 1.76$ mm^{-1}
$T = 293$ K
Plate, colorless
0.46 × 0.39 × 0.11 mm

Data collection

Oxford Diffraction Xcalibur Atlas Gemini Ultra CCD diffractometer
Radiation source: Enhance (Mo) X-ray source)
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: multi-scan (SCALEPACK; Otwinowski *et al.*, 1997)
$T_{min} = 0.455$, $T_{max} = 0.802$
10653 measured reflections
4337 independent reflections
2924 reflections with $I > 2\sigma(I)$
$R_{int} = 0.104$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.7°$
$h = -12\rightarrow12$
$k = -11\rightarrow11$
$l = -13\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.135$
$S = 0.97$
4337 reflections

272 parameters
2 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}
Absolute structure: Flack *x* determined using 1005 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.013 (13)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.17985 (7)	0.23216 (8)	0.27079 (6)	0.0716 (3)
O1	0.4307 (4)	0.4046 (5)	0.6482 (4)	0.0455 (11)
N3	0.3297 (6)	0.6276 (6)	0.8208 (5)	0.0559 (16)
O2	0.5063 (6)	0.6393 (6)	0.9554 (4)	0.0728 (16)
N2	0.3578 (5)	0.4065 (6)	0.7471 (4)	0.0419 (12)
N1	0.6516 (6)	0.6022 (6)	0.3324 (5)	0.0521 (15)
C14	0.2644 (7)	0.5267 (7)	0.7427 (5)	0.0447 (15)
C6	0.4147 (7)	0.3475 (8)	0.3841 (6)	0.0472 (16)
H6	0.4007	0.2871	0.4418	0.057*
C9	0.6468 (6)	0.4016 (8)	0.5952 (5)	0.0478 (16)
H9A	0.7375	0.4153	0.6248	0.057*
H9B	0.6317	0.3025	0.5885	0.057*
C1	0.3301 (7)	0.3486 (8)	0.2869 (6)	0.0523 (18)
C11	0.5918 (7)	0.4126 (9)	0.7977 (6)	0.0513 (18)
H11A	0.6196	0.3164	0.8013	0.062*
H11B	0.6594	0.4698	0.8372	0.062*
C10	0.5591 (6)	0.4605 (7)	0.6774 (5)	0.0426 (15)
H10	0.5572	0.5619	0.6734	0.051*
C8	0.6248 (6)	0.4659 (7)	0.4806 (5)	0.0439 (15)
C13	0.4388 (7)	0.5769 (8)	0.8831 (6)	0.0523 (18)
C19	0.1319 (7)	0.4750 (8)	0.7834 (5)	0.0485 (17)
H19	0.0774	0.5582	0.7854	0.058*
C5	0.5213 (6)	0.4378 (7)	0.3948 (5)	0.0428 (15)
C12	0.4609 (6)	0.4315 (8)	0.8439 (5)	0.0471 (16)
H12	0.4518	0.3648	0.9039	0.057*
C16	0.1585 (6)	0.4977 (7)	0.5389 (5)	0.0472 (16)
H16	0.2112	0.4153	0.5275	0.057*
C15	0.2358 (7)	0.5914 (7)	0.6262 (6)	0.0500 (17)
H15A	0.3184	0.6159	0.5992	0.060*
H15B	0.1867	0.6764	0.6328	0.060*
C7	0.6991 (6)	0.5668 (8)	0.4386 (6)	0.0503 (17)
H7	0.7730	0.6064	0.4776	0.060*

C4	0.5407 (7)	0.5268 (7)	0.3039 (6)	0.0459 (16)
C18	0.0585 (7)	0.3816 (8)	0.6964 (6)	0.0544 (18)
H18A	−0.0241	0.3541	0.7220	0.065*
H18B	0.1096	0.2983	0.6884	0.065*
C3	0.4561 (8)	0.5248 (8)	0.2046 (6)	0.056 (2)
H3	0.4705	0.5828	0.1455	0.068*
C17	0.0316 (6)	0.4521 (9)	0.5829 (6)	0.0574 (19)
H17A	−0.0146	0.3883	0.5302	0.069*
H17B	−0.0241	0.5322	0.5896	0.069*
C21	0.1702 (8)	0.2603 (9)	0.9120 (6)	0.071 (2)
H21A	0.2474	0.2388	0.8770	0.107*
H21B	0.1833	0.2340	0.9892	0.107*
H21C	0.0968	0.2105	0.8754	0.107*
C2	0.3522 (8)	0.4357 (8)	0.1965 (6)	0.061 (2)
H2	0.2954	0.4324	0.1309	0.073*
C20	0.1432 (7)	0.4168 (9)	0.9030 (6)	0.0554 (18)
H20	0.2168	0.4641	0.9457	0.066*
C23	0.1317 (8)	0.5741 (9)	0.4278 (6)	0.068 (2)
H23A	0.2133	0.6015	0.4022	0.102*
H23B	0.0854	0.5139	0.3736	0.102*
H23C	0.0796	0.6549	0.4376	0.102*
C22	0.0205 (10)	0.4502 (13)	0.9589 (8)	0.097 (3)
H22A	0.0032	0.5479	0.9535	0.145*
H22B	−0.0527	0.4001	0.9222	0.145*
H22C	0.0338	0.4236	1.0361	0.145*
C24	0.2777 (9)	0.7638 (8)	0.8427 (8)	0.081 (3)
H24A	0.2015	0.7815	0.7912	0.121*
H24B	0.2542	0.7668	0.9177	0.121*
H24C	0.3431	0.8329	0.8338	0.121*
HN1	0.6758	0.6699	0.2885	0.07 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0695 (5)	0.0753 (5)	0.0667 (5)	−0.0179 (5)	−0.0081 (3)	0.0009 (5)
O1	0.042 (3)	0.058 (3)	0.036 (3)	−0.003 (2)	0.0033 (19)	0.000 (2)
N3	0.066 (4)	0.049 (4)	0.052 (4)	−0.006 (3)	0.004 (3)	−0.008 (3)
O2	0.078 (4)	0.092 (4)	0.047 (3)	−0.029 (3)	−0.001 (3)	−0.015 (3)
N2	0.042 (3)	0.053 (3)	0.031 (3)	−0.002 (3)	0.002 (2)	0.001 (2)
N1	0.059 (4)	0.049 (4)	0.049 (4)	−0.007 (3)	0.012 (3)	0.004 (3)
C14	0.051 (4)	0.046 (4)	0.037 (4)	0.000 (3)	0.002 (3)	−0.003 (3)
C6	0.058 (4)	0.048 (4)	0.037 (4)	−0.004 (3)	0.009 (3)	0.005 (3)
C9	0.043 (4)	0.056 (4)	0.045 (4)	0.012 (3)	0.006 (3)	0.011 (3)
C1	0.067 (5)	0.047 (4)	0.042 (4)	−0.001 (4)	−0.001 (3)	−0.007 (3)
C11	0.045 (4)	0.070 (5)	0.037 (4)	0.003 (4)	−0.002 (3)	0.007 (4)
C10	0.040 (3)	0.049 (4)	0.038 (4)	−0.006 (3)	0.003 (3)	0.000 (3)
C8	0.042 (3)	0.049 (4)	0.041 (4)	0.000 (3)	0.009 (3)	−0.001 (3)
C13	0.058 (4)	0.062 (5)	0.038 (4)	−0.013 (4)	0.008 (3)	−0.001 (3)

C19	0.048 (4)	0.052 (4)	0.046 (4)	0.009 (3)	0.006 (3)	0.002 (3)
C5	0.048 (4)	0.043 (4)	0.038 (4)	0.004 (3)	0.008 (3)	−0.003 (3)
C12	0.046 (4)	0.060 (5)	0.034 (3)	−0.009 (3)	0.003 (3)	0.003 (3)
C16	0.051 (4)	0.044 (4)	0.045 (4)	0.006 (3)	−0.003 (3)	0.004 (3)
C15	0.058 (4)	0.037 (4)	0.054 (5)	0.008 (3)	0.003 (3)	0.008 (3)
C7	0.047 (4)	0.054 (4)	0.051 (4)	−0.005 (4)	0.009 (3)	−0.004 (3)
C4	0.057 (4)	0.038 (4)	0.043 (4)	0.001 (3)	0.011 (3)	−0.003 (3)
C18	0.044 (4)	0.067 (5)	0.050 (4)	−0.002 (3)	0.000 (3)	−0.003 (4)
C3	0.075 (5)	0.055 (5)	0.039 (4)	0.003 (4)	0.009 (4)	0.003 (3)
C17	0.048 (4)	0.069 (5)	0.052 (4)	0.003 (4)	−0.007 (3)	−0.001 (4)
C21	0.073 (5)	0.085 (7)	0.057 (5)	−0.006 (5)	0.015 (4)	0.015 (4)
C2	0.082 (5)	0.061 (5)	0.036 (4)	−0.007 (4)	−0.008 (3)	0.000 (4)
C20	0.051 (4)	0.072 (5)	0.045 (4)	0.002 (4)	0.012 (3)	−0.002 (4)
C23	0.090 (6)	0.065 (5)	0.045 (4)	0.011 (5)	−0.011 (4)	0.006 (4)
C22	0.087 (6)	0.139 (9)	0.071 (6)	0.021 (7)	0.037 (5)	0.009 (6)
C24	0.104 (6)	0.063 (7)	0.075 (6)	0.002 (5)	0.009 (5)	−0.021 (5)

Geometric parameters (Å, º)

Br—C1	1.901 (7)	C5—C4	1.423 (9)
O1—C10	1.432 (7)	C12—H12	0.9800
O1—N2	1.475 (6)	C16—C17	1.523 (10)
N3—C13	1.369 (10)	C16—C23	1.526 (10)
N3—C24	1.453 (10)	C16—C15	1.540 (10)
N3—C14	1.465 (9)	C16—H16	0.9800
O2—C13	1.212 (9)	C15—H15A	0.9700
N2—C14	1.502 (9)	C15—H15B	0.9700
N2—C12	1.507 (8)	C7—H7	0.9300
N1—C7	1.362 (9)	C4—C3	1.401 (11)
N1—C4	1.362 (9)	C18—C17	1.526 (10)
N1—HN1	0.8929	C18—H18A	0.9700
C14—C15	1.535 (9)	C18—H18B	0.9700
C14—C19	1.576 (10)	C3—C2	1.365 (11)
C6—C1	1.381 (10)	C3—H3	0.9300
C6—C5	1.394 (10)	C17—H17A	0.9700
C6—H6	0.9300	C17—H17B	0.9700
C9—C8	1.508 (9)	C21—C20	1.537 (12)
C9—C10	1.519 (9)	C21—H21A	0.9600
C9—H9A	0.9700	C21—H21B	0.9600
C9—H9B	0.9700	C21—H21C	0.9600
C1—C2	1.413 (11)	C2—H2	0.9300
C11—C12	1.519 (9)	C20—C22	1.525 (10)
C11—C10	1.524 (9)	C20—H20	0.9800
C11—H11A	0.9700	C23—H23A	0.9600
C11—H11B	0.9700	C23—H23B	0.9600
C10—H10	0.9800	C23—H23C	0.9600
C8—C7	1.368 (9)	C22—H22A	0.9600
C8—C5	1.426 (9)	C22—H22B	0.9600

C13—C12	1.506 (11)	C22—H22C	0.9600
C19—C18	1.519 (10)	C24—H24A	0.9600
C19—C20	1.540 (10)	C24—H24B	0.9600
C19—H19	0.9800	C24—H24C	0.9600
C10—O1—N2	109.3 (4)	C17—C16—H16	108.6
C13—N3—C24	121.2 (7)	C23—C16—H16	108.6
C13—N3—C14	113.9 (6)	C15—C16—H16	108.6
C24—N3—C14	124.5 (7)	C14—C15—C16	114.8 (5)
O1—N2—C14	111.0 (5)	C14—C15—H15A	108.6
O1—N2—C12	104.6 (4)	C16—C15—H15A	108.6
C14—N2—C12	107.2 (5)	C14—C15—H15B	108.6
C7—N1—C4	107.9 (6)	C16—C15—H15B	108.6
C7—N1—HN1	130.2	H15A—C15—H15B	107.5
C4—N1—HN1	121.3	N1—C7—C8	111.6 (6)
N3—C14—N2	104.1 (5)	N1—C7—H7	124.2
N3—C14—C15	110.2 (5)	C8—C7—H7	124.2
N2—C14—C15	113.9 (5)	N1—C4—C3	130.3 (7)
N3—C14—C19	111.1 (5)	N1—C4—C5	108.1 (6)
N2—C14—C19	108.3 (5)	C3—C4—C5	121.6 (7)
C15—C14—C19	109.1 (5)	C19—C18—C17	112.5 (6)
C1—C6—C5	119.2 (6)	C19—C18—H18A	109.1
C1—C6—H6	120.4	C17—C18—H18A	109.1
C5—C6—H6	120.4	C19—C18—H18B	109.1
C8—C9—C10	113.7 (5)	C17—C18—H18B	109.1
C8—C9—H9A	108.8	H18A—C18—H18B	107.8
C10—C9—H9A	108.8	C2—C3—C4	118.5 (7)
C8—C9—H9B	108.8	C2—C3—H3	120.7
C10—C9—H9B	108.8	C4—C3—H3	120.7
H9A—C9—H9B	107.7	C16—C17—C18	111.2 (5)
C6—C1—C2	121.4 (7)	C16—C17—H17A	109.4
C6—C1—Br	120.7 (6)	C18—C17—H17A	109.4
C2—C1—Br	117.9 (6)	C16—C17—H17B	109.4
C12—C11—C10	101.5 (5)	C18—C17—H17B	109.4
C12—C11—H11A	111.5	H17A—C17—H17B	108.0
C10—C11—H11A	111.5	C20—C21—H21A	109.5
C12—C11—H11B	111.5	C20—C21—H21B	109.5
C10—C11—H11B	111.5	H21A—C21—H21B	109.5
H11A—C11—H11B	109.3	C20—C21—H21C	109.5
O1—C10—C9	107.0 (5)	H21A—C21—H21C	109.5
O1—C10—C11	102.8 (5)	H21B—C21—H21C	109.5
C9—C10—C11	114.9 (5)	C3—C2—C1	120.5 (7)
O1—C10—H10	110.6	C3—C2—H2	119.7
C9—C10—H10	110.6	C1—C2—H2	119.7
C11—C10—H10	110.6	C22—C20—C21	109.1 (8)
C7—C8—C5	105.6 (6)	C22—C20—C19	110.7 (7)
C7—C8—C9	126.7 (6)	C21—C20—C19	114.7 (6)
C5—C8—C9	127.7 (6)	C22—C20—H20	107.3

O2—C13—N3	125.9 (8)	C21—C20—H20	107.3
O2—C13—C12	126.4 (7)	C19—C20—H20	107.3
N3—C13—C12	107.6 (6)	C16—C23—H23A	109.5
C18—C19—C20	114.3 (6)	C16—C23—H23B	109.5
C18—C19—C14	110.7 (6)	H23A—C23—H23B	109.5
C20—C19—C14	115.3 (6)	C16—C23—H23C	109.5
C18—C19—H19	105.2	H23A—C23—H23C	109.5
C20—C19—H19	105.2	H23B—C23—H23C	109.5
C14—C19—H19	105.2	C20—C22—H22A	109.5
C6—C5—C4	118.7 (6)	C20—C22—H22B	109.5
C6—C5—C8	134.6 (6)	H22A—C22—H22B	109.5
C4—C5—C8	106.7 (6)	C20—C22—H22C	109.5
C13—C12—N2	105.9 (6)	H22A—C22—H22C	109.5
C13—C12—C11	113.3 (6)	H22B—C22—H22C	109.5
N2—C12—C11	105.8 (5)	N3—C24—H24A	109.5
C13—C12—H12	110.5	N3—C24—H24B	109.5
N2—C12—H12	110.5	H24A—C24—H24B	109.5
C11—C12—H12	110.5	N3—C24—H24C	109.5
C17—C16—C23	111.4 (6)	H24A—C24—H24C	109.5
C17—C16—C15	109.2 (6)	H24B—C24—H24C	109.5
C23—C16—C15	110.3 (6)		

C10—O1—N2—C12	−14.5 (6)	C8—C5—C6—C1	179.1 (7)
C10—O1—N2—C14	100.9 (5)	C4—C5—C8—C7	−0.1 (7)
N2—O1—C10—C9	155.8 (5)	C4—C5—C8—C9	178.3 (6)
N2—O1—C10—C11	34.4 (6)	C6—C5—C8—C7	179.7 (8)
C7—N1—C4—C3	−179.9 (8)	C6—C5—C8—C9	−1.9 (13)
C7—N1—C4—C5	−2.0 (8)	N1—C7—C8—C5	−1.2 (8)
C4—N1—C7—C8	2.0 (8)	N1—C7—C8—C9	−179.6 (6)
O1—N2—C12—C11	−11.6 (7)	C5—C8—C9—C10	−78.1 (9)
O1—N2—C12—C13	109.0 (5)	C7—C8—C9—C10	100.1 (8)
C14—N2—C12—C11	−129.6 (6)	C8—C9—C10—O1	74.7 (7)
C14—N2—C12—C13	−9.0 (6)	C8—C9—C10—C11	−171.8 (6)
O1—N2—C14—N3	−103.0 (5)	O1—C10—C11—C12	−39.7 (7)
O1—N2—C14—C15	17.2 (7)	C9—C10—C11—C12	−155.6 (6)
O1—N2—C14—C19	138.7 (5)	C10—C11—C12—N2	31.3 (7)
C12—N2—C14—N3	10.8 (6)	C10—C11—C12—C13	−84.4 (7)
C12—N2—C14—C15	130.9 (6)	N2—C12—C13—O2	−175.3 (7)
C12—N2—C14—C19	−107.6 (5)	N2—C12—C13—N3	3.5 (7)
C14—N3—C13—O2	−177.5 (7)	C11—C12—C13—O2	−59.8 (10)
C14—N3—C13—C12	3.6 (8)	C11—C12—C13—N3	119.1 (6)
C24—N3—C13—O2	−4.7 (12)	N2—C14—C15—C16	67.4 (8)
C24—N3—C13—C12	176.5 (6)	N3—C14—C15—C16	−176.0 (6)
C13—N3—C14—N2	−9.2 (7)	C19—C14—C15—C16	−53.7 (7)
C13—N3—C14—C15	−131.7 (6)	N2—C14—C19—C18	−71.8 (7)
C13—N3—C14—C19	107.2 (7)	N2—C14—C19—C20	59.8 (7)
C24—N3—C14—N2	178.3 (6)	N3—C14—C19—C18	174.4 (6)
C24—N3—C14—C15	55.8 (9)	N3—C14—C19—C20	−54.0 (8)

C24—N3—C14—C19	−65.3 (8)	C15—C14—C19—C18	52.6 (7)
Br—C1—C2—C3	177.9 (6)	C15—C14—C19—C20	−175.8 (6)
C6—C1—C2—C3	−2.2 (12)	C14—C15—C16—C17	55.4 (8)
Br—C1—C6—C5	−177.7 (5)	C14—C15—C16—C23	178.1 (6)
C2—C1—C6—C5	2.4 (11)	C15—C16—C17—C18	−55.5 (8)
C1—C2—C3—C4	0.6 (12)	C23—C16—C17—C18	−177.4 (6)
C2—C3—C4—N1	178.3 (7)	C16—C17—C18—C19	58.6 (8)
C2—C3—C4—C5	0.7 (11)	C17—C18—C19—C14	−56.6 (8)
N1—C4—C5—C6	−178.5 (6)	C17—C18—C19—C20	171.3 (6)
N1—C4—C5—C8	1.3 (8)	C14—C19—C20—C21	−89.9 (8)
C3—C4—C5—C6	−0.4 (10)	C14—C19—C20—C22	146.0 (7)
C3—C4—C5—C8	179.4 (7)	C18—C19—C20—C21	40.0 (9)
C4—C5—C6—C1	−1.1 (10)	C18—C19—C20—C22	−84.1 (9)

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the N2/C12/C13/N3/C14 five-membered imidazolidinone ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H*N*1···N2^{i}	0.89	2.34	3.087 (8)	141
C3—H3···O2^{ii}	0.93	2.42	3.292 (9)	156
C16—H16···O1	0.98	2.55	3.091 (8)	115
C20—H20···N3	0.98	2.54	3.032 (10)	111
C21—H21*A*···N2	0.96	2.60	3.236 (9)	124
N1—H*N*1···O1^{i}	0.89	2.66	3.053 (8)	108
C20—H20···*Cg*3	0.98	2.41	2.866 (8)	104

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1; (ii) *x*, *y*, *z*−1.