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Crystal structure of the nucleoside 2′-deoxyguanosine dimethyl sulfoxide disolvate

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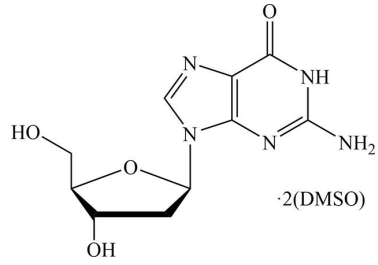
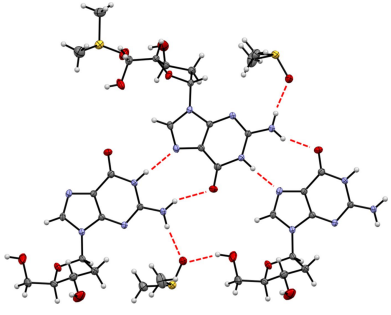
The title compound, $C_{10}H_{13}N_5O_4{\cdot}2C_2H_6OS$, which is of interest with respect to its biological activity, at 183 K has orthorhombic ($P2_12_12_1$) crystal symmetry. The structure displays a network of intermolecular N—H···N, N—H···O and O—H···O hydrogen bonds. 2′-Deoxyguanosine molecules are linked to each other and to the two dimethyl sulfoxide solvent molecules by hydrogen bonding.

1. Chemical context

Deoxynucleosides are the building blocks of DNA, the storage place for the genetic information in most organisms. Understanding the properties of DNA is crucial for our knowledge of its reactivity in cellular processes of replication and transcription to yield transfer RNA (Stryer, 1995). Furthermore, mutagenic reagents can irreversibly alter the structure and function of DNA (Wang *et al.*, 1998). In view of all this, it is of upmost importance to know the precise geometric parameters of all the nucleobases. These parameters are needed for techniques such as macromolecular X-ray crystallography in some cases and (NMR restrained) modelling of oligonucleotides (Clowney *et al.*, 1996; Gelbin *et al.*, 1996). Surprisingly, no high-quality crystal structure of unmodified 2′-deoxyguanosine has been published to date. In the course of studying the interaction of nucleobases with copper(II) (Santangelo *et al.*, 2007), we obtained single crystals of 2′-deoxyguanosine as a solvate with two molecules of dimethyl sulfoxide (DMSO), (**I**), and characterized it by X-ray diffraction.





2. Structural commentary

Nucleobase (**I**) crystallized in the orthorhombic Sohnke space group $P2_12_12_1$, with four formula units per unit cell and one per asymmetric unit (Fig. 1). The sugar conformation at the C3′ position (C13) is *endo*. The torsion angle χ (Alvarez *et al.*, 2019; Schabert *et al.*, 2021) of O14—C11—N9—C4 is −165.6 (1)° (Table 1). The freely refined H atoms of the exocyclic atom N2 were found to be in the plane of the latter and the adjacent six-membered aromatic ring, implying an sp^2 hybridization of N2. It is of interest to note that in the ligand

Table 1
Sugar conformations in 2′-deoxyguanosine, its supramolecular complexes and guanosine.

Compound	Space group	Sugar conformation	χ (O4′—C1′—N1—C6) (°)	Reference
2′-Deoxyguanosine·2(DMSO)	$P2_12_12_1$	Envelope, C3′-*endo*	−165.6 (1)	This work
(Actinomycin D)·2(2′-deoxyguanosine)·12H_2O	$P2_12_12_1$	Envelope, C3′-*endo*; Twisted, C1′-*exo*/C2′-*endo*	−86.5; −90.6	Jain & Sobell (1972)
(7-Bromoactinomycin D)·2(2′-deoxyguanosine)·11H_2O	$P2_12_12_1$	Twisted, C2′-*exo*/C3′-*endo*; Twisted, C1′-*exo*/C2′-*endo*	−86.5; −88.9	Jain & Sobell (1972)
(2′-Deoxyguanosine)·(5-bromo-2′-deoxycytidine)	$P2_12_12$	Envelope, C2′-*endo*	56.7	Haschemeyer *et al.* (1965)
(Guanosine)$_2$·4H_2O	$P2_1$	Envelope, C2′-*endo*; Twisted, C1′-*exo*/C2′-*endo*	−58.1; −137.2	Thewalt *et al.* (1970)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C23—H23B···O6^{i}	0.96 (3)	2.35 (3)	3.239 (3)	153 (2)
N1—H1···N7^{ii}	0.87 (2)	2.09 (2)	2.962 (2)	173 (2)
N2—H2A···O22	0.83 (2)	2.09 (2)	2.902 (2)	168 (2)
N2—H2B···O6^{ii}	0.83 (2)	2.24 (2)	3.012 (2)	155 (2)
O13—H13A···O21	0.87 (3)	1.88 (4)	2.738 (3)	169 (3)
O15—H15···O22^{iii}	0.89 (3)	1.91 (3)	2.752 (2)	159 (3)

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

database (Ligand Expo; Feng *et al.*, 2004) of the Protein Databank (Burley *et al.*, 2023) an incorrect Lewis structure of 2′-deoxyguanosine (identifier GNG) is present (Fig. S1 in the supporting information).

3. Supramolecular features

The hydrogen bonds are listed in Table 2. The hydrogen bonding among the guanine nucleobases is a reverse Hoogsteen pairing (Johnson *et al.*, 1992), generating an $R_2^2(9)$ graph set (Bernstein *et al.*, 1995) (Fig. 2). A very similar hydrogen-bonding motif was found for guanine monohydrate (Thewalt *et al.*, 1971) and guanosine dihydrate (Thewalt *et al.*, 1970). Atom O21 of one DMSO molecule is hydrogen bonded to the secondary alcohol group of 2′-deoxyguanosine, while atom O22 of the other DMSO molecule is hydrogen bonded both to the exocyclic amino group of one 2′-deoxyguanosine molecule and to the primary –OH group of another 2′-deoxyguanosine molecule. Analysis of the fingerprint plots of the Hirshfeld surface around the 2′-deoxyguanosine molecule calculated by *CrystalExplorer* (Spackman *et al.*, 2021) (Fig. 3) indicates that H···H contacts account for 38.3% of the surface contacts, O···H/H···O contacts for 28.4%, N···H/H···N for 16.5% and C···H/H···C for 9.9%.

4. Database survey

The search of the Cambridge Structural Database (CSD, Version 5.44, April 2023; Groom *et al.*, 2016) was made with *ConQuest* (Version 2023.1.0; Bruno *et al.*, 2002). The first structure containing 2′-deoxyguanosine (CSD refcode DGUBCY, a cocrystal with 5-bromo-2′-deoxycytidine) was published by Haschemeyer *et al.* (1965). However, in the corresponding CSD entry, the Lewis diagram of the 2′-deoxyguanosine is wrong (Fig. S2), showing a 2-aminopyrimidin-4-ol moiety, which should be redrawn as a 2-aminopyrimidin-4(3*H*)-one. The cocrystal structures of (actinomycin D)·2(2′-deoxyguanosine)·12H_2O (ACTDGU) and (7-bromoactinomycin)·2(2′-deoxyguanosine)·11H_2O (BRAXGU) at room temperature were also reported (Sobell *et al.*, 1971; Jain & Sobell, 1972). In addition, the X-ray structures of four metal complexes containing 2′-deoxyguanosine are known (WEWKEO, UKISEM, WUNXIM and EWOBIN; Shionoya *et al.*, 1994; Ito *et al.*, 2002; Aoki & Salam, 2002; Baruah *et al.*,

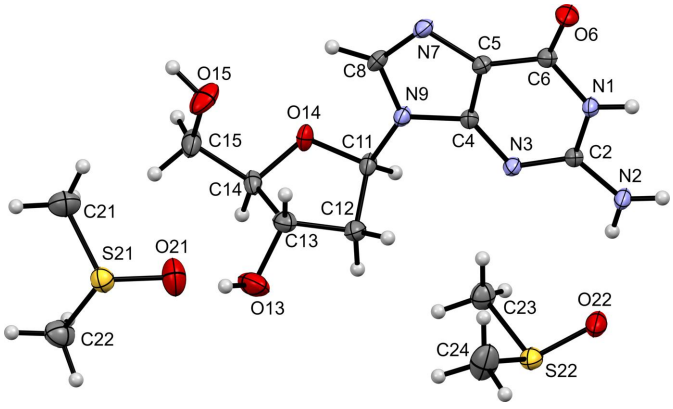


Figure 1
Displacement ellipsoid plot (50% probability) of (**I**).

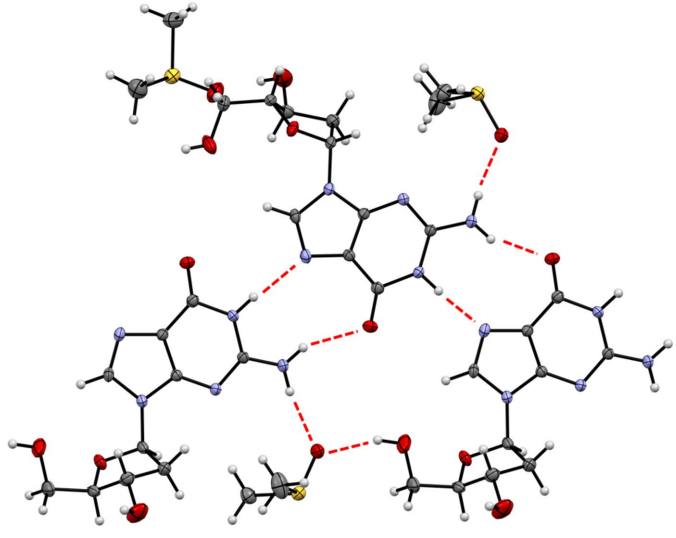


Figure 2
Hydrogen-bonding pattern of (**I**). Displacement ellipsoids are drawn at the 50% probability level.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{13}N_5O_4{\cdot}2C_2H_6OS$
M_r	423.51
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	183
a, b, c (Å)	9.7590 (1), 11.7951 (2), 16.7553 (2)
V (Å^3)	1928.68 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.32
Crystal size (mm)	0.44 × 0.24 × 0.17
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Ruby
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)
T_{min}, T_{max}	0.909, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19896, 5874, 5295
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.714
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.073, 1.01
No. of reflections	5874
No. of parameters	271
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.27
Absolute structure	Flack x determined using 2107 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.022 (14)

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2008), *CrysAlis RED* (Oxford Diffraction, 2008), *SIR97* (Altomare *et al.*, 1999), *SHELXL2018* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

2004). Only one of them was recorded at low temperature (Baruah *et al.*, 2004). Still, even for the latter structure, the average C—C bond distance was determined with a rather low precision of 0.009 Å. The present structure, (**I**), is the only purine nucleoside solvate in the CSD (Groom *et al.*, 2016) with two DMSO molecules per host molecule.

5. Synthesis and crystallization

Single crystals of (**I**) were obtained upon slow evaporation of 2′-deoxyguanosine (product number D0052, TCI) from DMSO.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The structure was solved by direct methods with the program *SIR97* (Altomare *et al.*, 1999). All C-bonded H atoms were placed in ideal positions, with C—H bond lengths of 0.95 Å for aromatic, 1.00 Å for methine, 0.99 Å for methylene and 0.98 Å for methyl C atoms, and refined as riding atoms, except those of methyl group C23H$_3$ of one DMSO molecule, which makes a relatively close contact to O6. The latter H atoms and those attached to non-C atoms were freely refined. The U_{iso}(H) values were set at 1.2 times (for CH, NH, NH$_2$ and CH$_2$ units) or 1.5 times (for methyl and OH groups) the U_{eq} value of the parent atom. The Flack parameter x was −0.00 (6) by classical fit to all intensities and 0.022 (14) by Parsons' method (Parsons *et al.*, 2013), from 2107 selected quotients.

7. Computational details

The sugar conformations (Table 1) were analysed with *PLATON* (Spek, 2020), using the published description of such conformations by Saenger (1984). For older structures, where the CSD does not contain H atoms, these were added

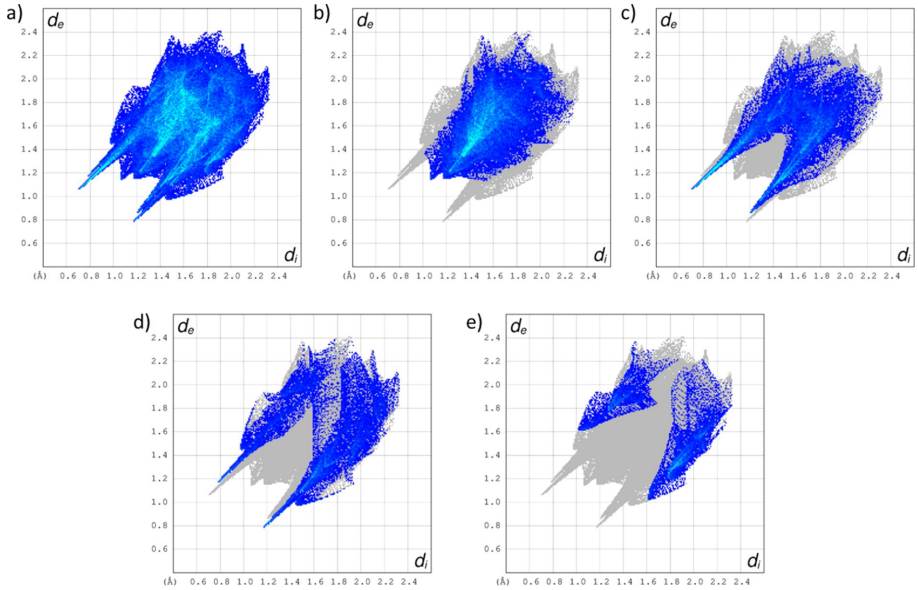


Figure 3
Fingerprint plots of (*a*) the entire Hirshfeld surface of 2′-deoxyguanosine within 2′-deoxyguanosine·2(DMSO), (*b*) H···H contacts, (*c*) O···H/H···O contacts, (*d*) N···H/H···N contacts and (*e*) C···H/H···C contacts.

using *OLEX2* (Dolomanov *et al.*, 2009) with default parameters.

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supporting information

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Crystal structure of the nucleoside 2′-deoxyguanosine dimethyl sulfoxide disolvate

Bernhard Spingler

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED* (Oxford Diffraction, 2008); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

2′-Deoxyguanosine dimethyl sulfoxide disolvate

Crystal data

$C_{10}H_{13}N_5O_4 \cdot 2C_2H_6OS$
$M_r = 423.51$
Orthorhombic, $P2_12_12_1$
$a = 9.7590$ (1) Å
$b = 11.7951$ (2) Å
$c = 16.7553$ (2) Å
$V = 1928.68$ (4) Å^3
$Z = 4$
$F(000) = 896$
$D_x = 1.459$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11856 reflections
$\theta = 2.4$–$32.7°$
$\mu = 0.32$ mm^{-1}
$T = 183$ K
Needle, colourless
0.44 × 0.24 × 0.17 mm

Data collection

Oxford Diffraction Xcalibur Ruby diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.4498 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)
$T_{min} = 0.909$, $T_{max} = 1.000$
19896 measured reflections
5874 independent reflections
5295 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 30.5°$, $\theta_{min} = 2.4°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 16$
$l = -22 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.073$
$S = 1.01$
5874 reflections
271 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}

$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Absolute structure: Flack *x* determined using 2107 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.022 (14)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C2	0.35188 (19)	0.60776 (14)	0.66640 (9)	0.0159 (3)
C4	0.31935 (18)	0.42499 (14)	0.64001 (9)	0.0157 (3)
C5	0.40248 (18)	0.38572 (15)	0.70072 (10)	0.0163 (3)
C6	0.47568 (18)	0.46616 (14)	0.74730 (10)	0.0183 (3)
C8	0.32641 (18)	0.23836 (15)	0.64098 (10)	0.0184 (3)
H8	0.308547	0.161923	0.626466	0.022*
C11	0.17747 (19)	0.33364 (15)	0.53323 (10)	0.0192 (3)
H11	0.101178	0.388127	0.543999	0.023*
C12	0.2513 (2)	0.36613 (16)	0.45639 (10)	0.0242 (4)
H12A	0.190773	0.410232	0.420577	0.029*
H12B	0.334890	0.410942	0.467733	0.029*
C13	0.2870 (2)	0.25191 (18)	0.42025 (10)	0.0243 (4)
H13	0.370902	0.219466	0.445824	0.029*
C14	0.16057 (19)	0.18386 (15)	0.44271 (11)	0.0212 (4)
H14	0.084806	0.204527	0.405249	0.025*
C15	0.1745 (2)	0.05766 (16)	0.44360 (13)	0.0296 (4)
H15A	0.088134	0.023128	0.462851	0.035*
H15B	0.191768	0.029894	0.388760	0.035*
C23	0.0397 (2)	0.63289 (18)	0.47584 (13)	0.0280 (4)
H23A	0.093 (3)	0.593 (2)	0.5123 (15)	0.042*
H23B	0.010 (3)	0.585 (2)	0.4328 (16)	0.042*
H23C	−0.038 (3)	0.668 (2)	0.4972 (15)	0.042*
C24	0.2803 (3)	0.6683 (2)	0.40425 (16)	0.0438 (6)
H24A	0.248599	0.617530	0.361775	0.066*
H24B	0.319500	0.623323	0.447802	0.066*
H24C	0.350235	0.719659	0.382971	0.066*
N1	0.44138 (16)	0.57763 (13)	0.72580 (8)	0.0173 (3)
H1	0.483 (2)	0.6326 (19)	0.7512 (13)	0.021*
N2	0.32939 (19)	0.71824 (13)	0.65567 (9)	0.0218 (3)
H2A	0.281 (3)	0.737 (2)	0.6175 (13)	0.026*
H2B	0.367 (3)	0.771 (2)	0.6808 (13)	0.026*
N3	0.28910 (16)	0.53300 (12)	0.61976 (8)	0.0169 (3)
N7	0.40557 (16)	0.26752 (12)	0.70054 (8)	0.0187 (3)
N9	0.27173 (15)	0.33028 (12)	0.60156 (8)	0.0168 (3)
O6	0.56048 (15)	0.44860 (12)	0.80073 (7)	0.0270 (3)

O13	0.3041 (2)	0.26086 (16)	0.33678 (8)	0.0432 (4)
H13A	0.363 (4)	0.215 (3)	0.3158 (18)	0.065*
O14	0.12431 (14)	0.22319 (10)	0.52196 (7)	0.0219 (3)
O15	0.28382 (17)	0.02499 (13)	0.49400 (10)	0.0385 (4)
H15	0.275 (3)	−0.048 (3)	0.5060 (16)	0.058*
O21	0.50822 (19)	0.11673 (15)	0.28913 (10)	0.0440 (4)
O22	0.19120 (17)	0.80671 (12)	0.51554 (8)	0.0318 (3)
S22	0.13986 (5)	0.74863 (4)	0.44083 (3)	0.02532 (11)
S21	0.53507 (5)	0.01236 (4)	0.24068 (3)	0.02556 (11)
C22	0.4129 (2)	0.0115 (2)	0.16179 (12)	0.0362 (5)
H22A	0.431974	0.074434	0.125214	0.054*
H22B	0.320604	0.020153	0.183990	0.054*
H22C	0.418952	−0.060392	0.132742	0.054*
C21	0.4665 (3)	−0.1037 (2)	0.29503 (13)	0.0396 (5)
H21A	0.371508	−0.087110	0.309943	0.059*
H21B	0.521108	−0.116028	0.343349	0.059*
H21C	0.469085	−0.172052	0.261807	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0194 (8)	0.0140 (8)	0.0144 (7)	0.0000 (6)	0.0000 (6)	−0.0005 (6)
C4	0.0175 (8)	0.0146 (8)	0.0150 (7)	−0.0013 (6)	0.0000 (6)	−0.0009 (6)
C5	0.0184 (8)	0.0146 (8)	0.0158 (7)	0.0009 (6)	−0.0004 (6)	0.0015 (6)
C6	0.0216 (8)	0.0172 (8)	0.0160 (7)	0.0014 (7)	0.0003 (7)	0.0009 (6)
C8	0.0208 (8)	0.0115 (8)	0.0231 (8)	−0.0016 (7)	0.0007 (6)	0.0026 (6)
C11	0.0211 (9)	0.0129 (8)	0.0235 (8)	0.0001 (6)	−0.0063 (7)	−0.0033 (6)
C12	0.0327 (10)	0.0191 (9)	0.0207 (8)	−0.0027 (8)	−0.0048 (7)	0.0022 (7)
C13	0.0301 (9)	0.0238 (9)	0.0189 (7)	0.0038 (8)	−0.0012 (7)	0.0018 (7)
C14	0.0247 (9)	0.0165 (9)	0.0224 (8)	0.0042 (7)	−0.0097 (7)	−0.0051 (7)
C15	0.0368 (11)	0.0169 (9)	0.0350 (10)	0.0027 (8)	−0.0101 (9)	−0.0059 (8)
C23	0.0255 (10)	0.0275 (11)	0.0310 (10)	−0.0040 (9)	0.0012 (9)	−0.0017 (8)
C24	0.0400 (13)	0.0350 (13)	0.0564 (14)	−0.0071 (11)	0.0217 (11)	−0.0083 (11)
N1	0.0228 (8)	0.0124 (7)	0.0166 (6)	−0.0014 (6)	−0.0055 (6)	−0.0015 (5)
N2	0.0317 (9)	0.0118 (7)	0.0218 (7)	−0.0004 (6)	−0.0091 (7)	−0.0011 (6)
N3	0.0208 (7)	0.0127 (7)	0.0171 (6)	0.0010 (6)	−0.0035 (5)	−0.0001 (5)
N7	0.0223 (7)	0.0125 (7)	0.0212 (7)	0.0005 (6)	0.0001 (6)	0.0032 (5)
N9	0.0191 (7)	0.0120 (7)	0.0195 (6)	−0.0018 (6)	−0.0031 (5)	−0.0003 (5)
O6	0.0339 (8)	0.0236 (7)	0.0234 (6)	0.0003 (6)	−0.0129 (6)	0.0019 (5)
O13	0.0663 (12)	0.0431 (10)	0.0201 (7)	0.0143 (9)	0.0066 (7)	0.0027 (7)
O14	0.0225 (6)	0.0156 (6)	0.0275 (6)	−0.0048 (5)	−0.0010 (5)	−0.0059 (5)
O15	0.0365 (8)	0.0198 (8)	0.0592 (10)	0.0036 (7)	−0.0145 (7)	0.0086 (7)
O21	0.0483 (11)	0.0361 (9)	0.0477 (10)	0.0013 (8)	−0.0043 (8)	−0.0179 (7)
O22	0.0463 (9)	0.0188 (7)	0.0302 (7)	−0.0004 (7)	−0.0144 (7)	−0.0014 (5)
S22	0.0316 (2)	0.0205 (2)	0.0239 (2)	−0.0008 (2)	−0.00666 (18)	0.00218 (18)
S21	0.0255 (2)	0.0264 (3)	0.0248 (2)	−0.00201 (19)	−0.00031 (19)	−0.00260 (17)
C22	0.0353 (11)	0.0424 (13)	0.0310 (10)	−0.0071 (10)	−0.0079 (9)	0.0039 (9)
C21	0.0464 (14)	0.0379 (13)	0.0345 (11)	−0.0042 (11)	0.0051 (11)	0.0076 (9)

Geometric parameters (Å, º)

C2—N1	1.371 (2)	C15—H15A	0.9900
C2—N2	1.334 (2)	C15—H15B	0.9900
C2—N3	1.328 (2)	C15—O15	1.414 (2)
C4—C5	1.381 (2)	C23—H23A	0.93 (3)
C4—N3	1.351 (2)	C23—H23B	0.96 (3)
C4—N9	1.371 (2)	C23—H23C	0.94 (3)
C5—C6	1.421 (2)	C23—S22	1.778 (2)
C5—N7	1.395 (2)	C24—H24A	0.9800
C6—N1	1.404 (2)	C24—H24B	0.9800
C6—O6	1.237 (2)	C24—H24C	0.9800
C8—H8	0.9500	C24—S22	1.775 (2)
C8—N7	1.308 (2)	N1—H1	0.87 (2)
C8—N9	1.377 (2)	N2—H2A	0.83 (2)
C11—H11	1.0000	N2—H2B	0.83 (2)
C11—C12	1.525 (2)	O13—H13A	0.87 (3)
C11—N9	1.469 (2)	O15—H15	0.89 (3)
C11—O14	1.415 (2)	O21—S21	1.4978 (17)
C12—H12A	0.9900	O22—S22	1.5124 (14)
C12—H12B	0.9900	S21—C22	1.780 (2)
C12—C13	1.518 (3)	S21—C21	1.775 (2)
C13—H13	1.0000	C22—H22A	0.9800
C13—C14	1.520 (3)	C22—H22B	0.9800
C13—O13	1.412 (2)	C22—H22C	0.9800
C14—H14	1.0000	C21—H21A	0.9800
C14—C15	1.495 (3)	C21—H21B	0.9800
C14—O14	1.450 (2)	C21—H21C	0.9800
N2—C2—N1	117.13 (15)	H23A—C23—H23B	111 (2)
N3—C2—N1	123.30 (15)	H23A—C23—H23C	115 (2)
N3—C2—N2	119.57 (16)	H23B—C23—H23C	108 (2)
N3—C4—C5	129.01 (15)	S22—C23—H23A	107.2 (17)
N3—C4—N9	125.20 (15)	S22—C23—H23B	111.7 (15)
N9—C4—C5	105.78 (15)	S22—C23—H23C	103.7 (16)
C4—C5—C6	118.42 (16)	H24A—C24—H24B	109.5
C4—C5—N7	110.26 (15)	H24A—C24—H24C	109.5
N7—C5—C6	131.13 (16)	H24B—C24—H24C	109.5
N1—C6—C5	111.38 (14)	S22—C24—H24A	109.5
O6—C6—C5	128.47 (16)	S22—C24—H24B	109.5
O6—C6—N1	120.15 (15)	S22—C24—H24C	109.5
N7—C8—H8	123.6	C2—N1—C6	125.52 (14)
N7—C8—N9	112.82 (15)	C2—N1—H1	117.2 (14)
N9—C8—H8	123.6	C6—N1—H1	117.3 (15)
C12—C11—H11	110.1	C2—N2—H2A	117.4 (17)
N9—C11—H11	110.1	C2—N2—H2B	125.9 (17)
N9—C11—C12	111.64 (15)	H2A—N2—H2B	116 (2)
O14—C11—H11	110.1	C2—N3—C4	112.18 (14)

O14—C11—C12	106.99 (14)	C8—N7—C5	104.58 (15)
O14—C11—N9	107.98 (14)	C4—N9—C8	106.55 (13)
C11—C12—H12A	111.2	C4—N9—C11	123.81 (14)
C11—C12—H12B	111.2	C8—N9—C11	129.61 (14)
H12A—C12—H12B	109.1	C13—O13—H13A	116 (2)
C13—C12—C11	102.84 (15)	C11—O14—C14	109.10 (14)
C13—C12—H12A	111.2	C15—O15—H15	109 (2)
C13—C12—H12B	111.2	C24—S22—C23	97.37 (11)
C12—C13—H13	110.9	O22—S22—C23	104.88 (9)
C12—C13—C14	100.59 (15)	O22—S22—C24	105.77 (12)
C14—C13—H13	110.9	O21—S21—C22	106.85 (11)
O13—C13—C12	110.84 (16)	O21—S21—C21	106.85 (11)
O13—C13—H13	110.9	C21—S21—C22	97.15 (12)
O13—C13—C14	112.36 (17)	S21—C22—H22A	109.5
C13—C14—H14	108.4	S21—C22—H22B	109.5
C15—C14—C13	117.04 (17)	S21—C22—H22C	109.5
C15—C14—H14	108.4	H22A—C22—H22B	109.5
O14—C14—C13	104.83 (14)	H22A—C22—H22C	109.5
O14—C14—H14	108.4	H22B—C22—H22C	109.5
O14—C14—C15	109.36 (16)	S21—C21—H21A	109.5
C14—C15—H15A	109.6	S21—C21—H21B	109.5
C14—C15—H15B	109.6	S21—C21—H21C	109.5
H15A—C15—H15B	108.1	H21A—C21—H21B	109.5
O15—C15—C14	110.24 (16)	H21A—C21—H21C	109.5
O15—C15—H15A	109.6	H21B—C21—H21C	109.5
O15—C15—H15B	109.6		

C4—C5—C6—N1	4.3 (2)	N3—C4—C5—C6	−4.4 (3)
C4—C5—C6—O6	−175.47 (17)	N3—C4—C5—N7	−179.94 (17)
C4—C5—N7—C8	0.2 (2)	N3—C4—N9—C8	−179.89 (17)
C5—C4—N3—C2	0.6 (3)	N3—C4—N9—C11	−1.5 (3)
C5—C4—N9—C8	0.69 (18)	N7—C5—C6—N1	178.83 (18)
C5—C4—N9—C11	179.13 (15)	N7—C5—C6—O6	−1.0 (3)
C5—C6—N1—C2	−1.6 (2)	N7—C8—N9—C4	−0.6 (2)
C6—C5—N7—C8	−174.68 (18)	N7—C8—N9—C11	−178.96 (16)
C11—C12—C13—C14	36.85 (17)	N9—C4—C5—C6	175.04 (15)
C11—C12—C13—O13	155.88 (17)	N9—C4—C5—N7	−0.55 (19)
C12—C11—N9—C4	77.0 (2)	N9—C4—N3—C2	−178.67 (16)
C12—C11—N9—C8	−104.9 (2)	N9—C8—N7—C5	0.3 (2)
C12—C11—O14—C14	0.18 (18)	N9—C11—C12—C13	93.88 (17)
C12—C13—C14—C15	−159.00 (16)	N9—C11—O14—C14	−120.12 (15)
C12—C13—C14—O14	−37.66 (16)	O6—C6—N1—C2	178.24 (16)
C13—C14—C15—O15	55.1 (2)	O13—C13—C14—C15	83.1 (2)
C13—C14—O14—C11	23.95 (18)	O13—C13—C14—O14	−155.58 (16)
C15—C14—O14—C11	150.21 (15)	O14—C11—C12—C13	−24.05 (18)
N1—C2—N3—C4	2.6 (2)	O14—C11—N9—C4	−165.65 (15)
N2—C2—N1—C6	178.46 (17)	O14—C11—N9—C8	12.4 (2)
N2—C2—N3—C4	−178.00 (16)	O14—C14—C15—O15	−63.8 (2)

N3—C2—N1—C6	−2.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C23—H23*B*···O6^{i}	0.96 (3)	2.35 (3)	3.239 (3)	153 (2)
N1—H1···N7^{ii}	0.87 (2)	2.09 (2)	2.962 (2)	173 (2)
N2—H2*A*···O22	0.83 (2)	2.09 (2)	2.902 (2)	168 (2)
N2—H2*B*···O6^{ii}	0.83 (2)	2.24 (2)	3.012 (2)	155 (2)
O13—H13*A*···O21	0.87 (3)	1.88 (4)	2.738 (3)	169 (3)
O15—H15···O22^{iii}	0.89 (3)	1.91 (3)	2.752 (2)	159 (3)

Symmetry codes: (i) −*x*+1/2, −*y*+1, *z*−1/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2; (iii) *x*, *y*−1, *z*.