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Crystal structures of two triazola-dioxola-benzena-cyclononaphanes

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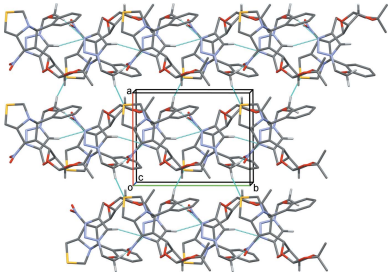
CCDC references: 1023614; 1023839
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compounds, $C_{25}H_{29}BrN_5O_7$, (I) [systematic name: (*Z*)-1^5-bromo-3^2,3^2-dimethyl-2^1-nitro-2^2,2^3,2^5,2^6,2^7,2^{7a},3^{3a},3^5,3^6,3^{6a}-decahydro-2^1*H*,6^1*H*-4,9-dioxa-2(3,2)-pyrrolizina-6(4,1)-triazola-3(5,6)-furo[2,3-*d*][1,3]dioxola-1(1,2)-benzenacyclononaphane], and $C_{24}H_{29}N_5O_7S$, (II) [systematic name: (*Z*)-3^2,3^2-dimethyl-2^7-nitro-2^5,2^6,2^7,2^{7a},3^{3a},3^5,3^6,3^{6a}-octahydro-2^1*H*,2^3*H*,6^1*H*-4,9-dioxa-2(5,6)-pyrrolo[1,2-*c*]thiazola-6(4,1)-triazola-3(5,6)-furo[2,3-*d*][1,3]dioxola-1(1,2)-benzenacyclononaphane], the triazole rings adopt almost planar conformations. In (I), the fused pyrrolidine rings adopt envelope conformations with the C atoms opposite the fused N—C bond as the flap in each ring, and their mean planes are inclined to one another by 52.8 (3)°. In (II), the pyrrolidine and thiazole rings are both twisted on the fused N—C bond, and their mean planes are inclined to one another by 70.8 (2)°. In both (I) and (II), the furan ring adopts an envelope conformation with the adjacent C atom of the macrocycle as the flap. In the crystal of (I), molecules are linked *via* C—H···N and C—H···O hydrogen bonds, forming sheets parallel to (10$\bar{1}$), while in (II), molecules are linked *via* C—H···N and C—H···O hydrogen bonds, forming helical chains propagating along [010], which are linked *via* C—H···S hydrogen bonds, forming slabs parallel to (001).

1. Chemical context

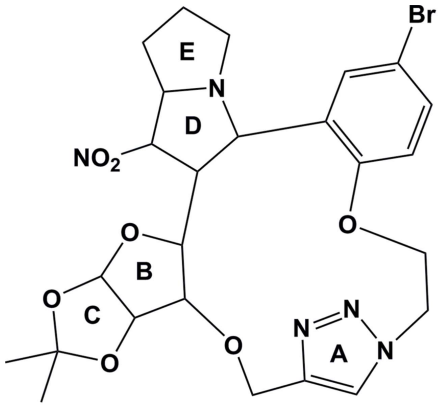
Triazoles and their derivatives are of great importance in medicinal chemistry and can be used for the synthesis of many heterocyclic compounds with different biological activities such as antiviral, antibacterial, antifungal (Mange *et al.*, 2013), anticancer (Singhal *et al.*, 2011), antituberculosis, anti-convulsant, antidepressant (Sahin *et al.*, 2012) and anti-inflammatory activities. They have been reported to be inhibitors of glycogen synthase kinase-3, antagonists of GABA receptors, agonists of muscarine receptors and have been shown to possess anti-HIV-1, cytotoxic, antihistaminic and antiproliferative activities (Pokhodylo *et al.*, 2013). Triazoles are stable to acid and basic hydrolysis and reductive and oxidative conditions because of their high aromatic stabilization. In addition, this heterocycle has a high dipole moment and might participate in hydrogen-bond formation as well as in dipole–dipole and π-stacking interactions (Pertino *et al.*, 2013).



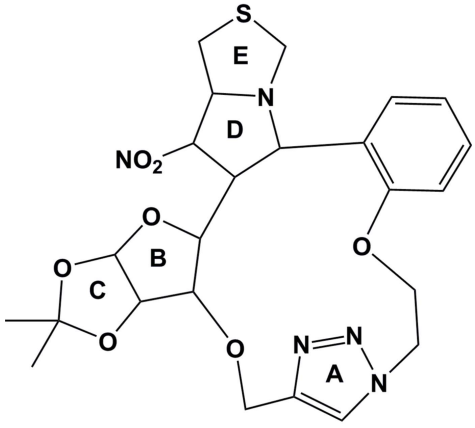
2. Structural commentary

The molecular structures of compounds (I) and (II) are illustrated in Figs. 1 and 2, respectively. The triazole rings (*A* =

N3–N5/C22/C23) adopt almost planar conformations in both compounds. In compound (I), the pyrrolidine rings (*D* = N1/C11–C13/C7 and *E* = N1/C8–C11) and the furan ring (*B* = O3/C15/C19/C20/C14) have envelope conformations with the maximum deviations from the respective mean planes of 0.465 (5) Å for atom C13, 0.490 (7) Å for C9 and 0.500 (4) Å for C14. The dioxalane ring (*C* = O4/C15/C19/O5/C16) has a twisted conformation on bond O5—C15. The mean planes of rings *B* and *C* are inclined to one another by 70.0 (3)°, and the mean planes of rings *D* and *E* are inclined to one another by 52.8 (3)°.



(I)



(II)

In compound (II), the pyrrolidine (*D*) and thiazole rings (*E* = N1/C8/S9/C10/C11) have twist conformations on bond N1—C11. The furan and dioxolane rings (*B* and *C*) adopt envelope conformations with maximum deviations from the mean planes of 0.631 (3) Å for atom C14 and 0.319 (4) Å for C16. The mean planes of rings *B* and *C* are inclined to one another by 68.5 (2)° and the mean planes of rings *D* and *E* are inclined to one another by 70.8 (2)°. This latter dihedral angle is much larger than that in compound (I), *cf.* 52.8 (3)°.

In compound (I), the triazole ring (*A*) makes dihedral angles of 74.0 (3), 65.8 (3) and 65.8 (3)° with the mean planes of rings *B* and *D* and the benzene ring (C1–C6), respectively. The corresponding dihedral angles in compound (II) are 51.9 (2), 37.1 (2) and 60.9 (2)°, respectively. The most notable differences between the compounds involve dihedral angles

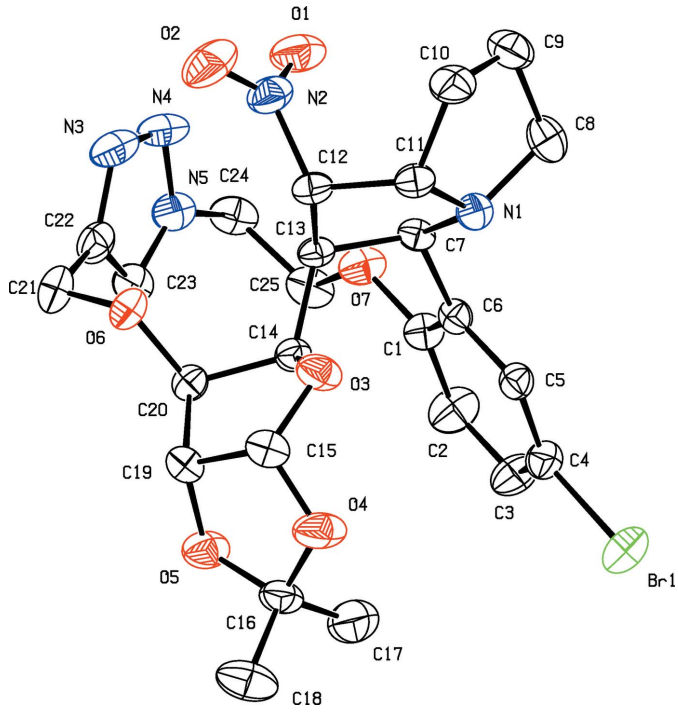


Figure 1
The molecular structure of compound (I), showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. H atoms are omitted for clarity.

A/*B* and *A*/*D*; 74.0 (3) and 65.8 (3), respectively, for (I), and 51.9 (2) and 37.1 (2)°, respectively, for (II).

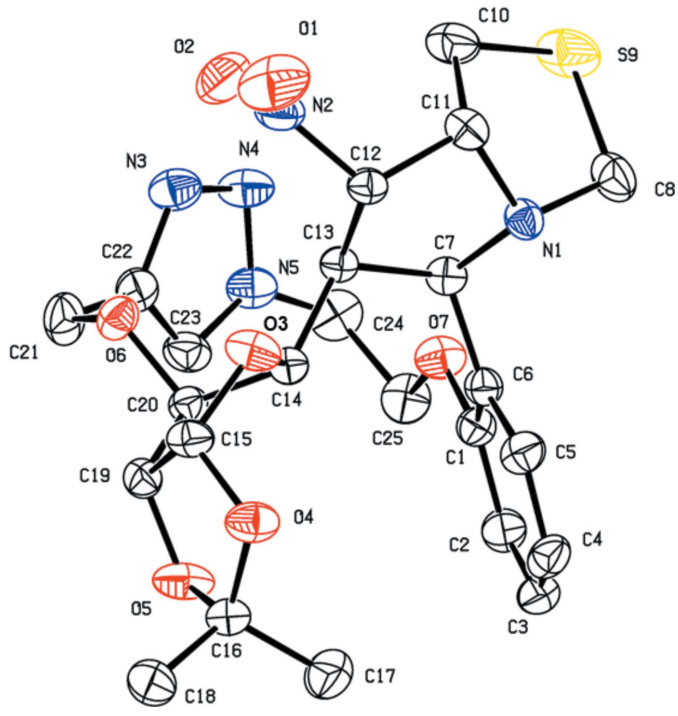


Figure 2
The molecular structure of compound (II), showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. H atoms are omitted for clarity.

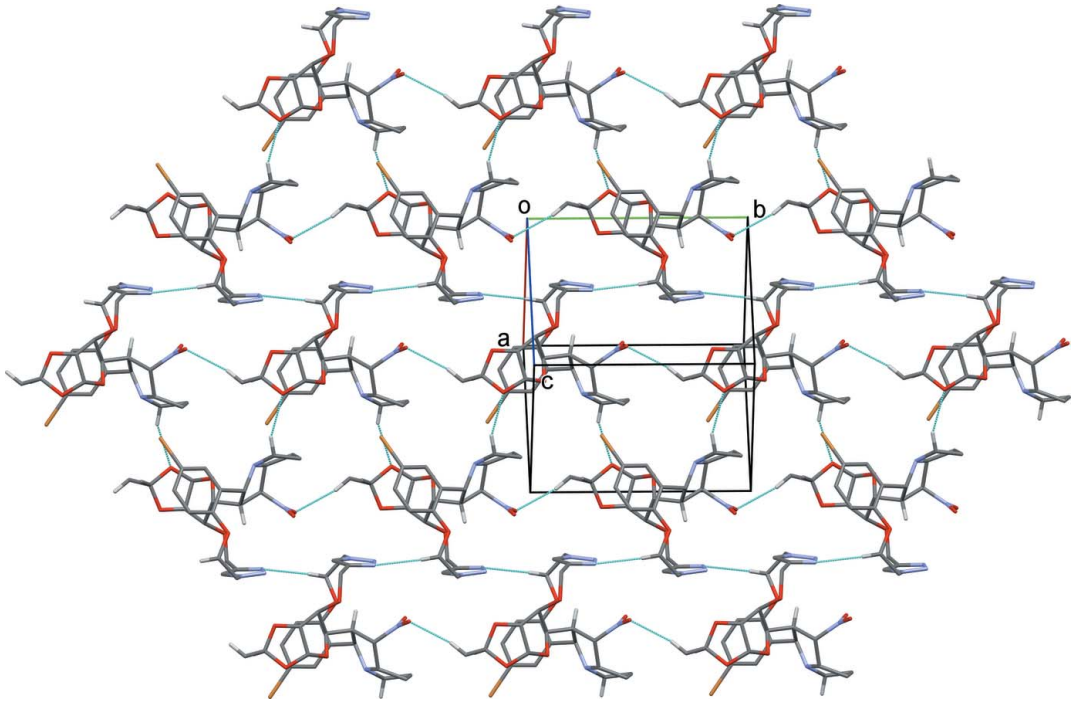


Figure 3
The crystal packing of compound (I), viewed approximately normal to plane (10$\bar{1}$). H atoms not involved in hydrogen bonding (dashed lines; Table 1) have been excluded for clarity.

3. Supramolecular features

In the crystal of (I), molecules are linked *via* C—H···N and C—H···O hydrogen bonds, forming sheets parallel to (10$\bar{1}$); Table 1 and Fig. 3. In the crystal of (II), molecules are linked *via* C—H···N and C—H···O hydrogen bonds, forming helical chains propagating along [010], which are linked *via* C—H···S hydrogen bonds, forming slabs parallel to (001); Table 2 and Fig. 4.

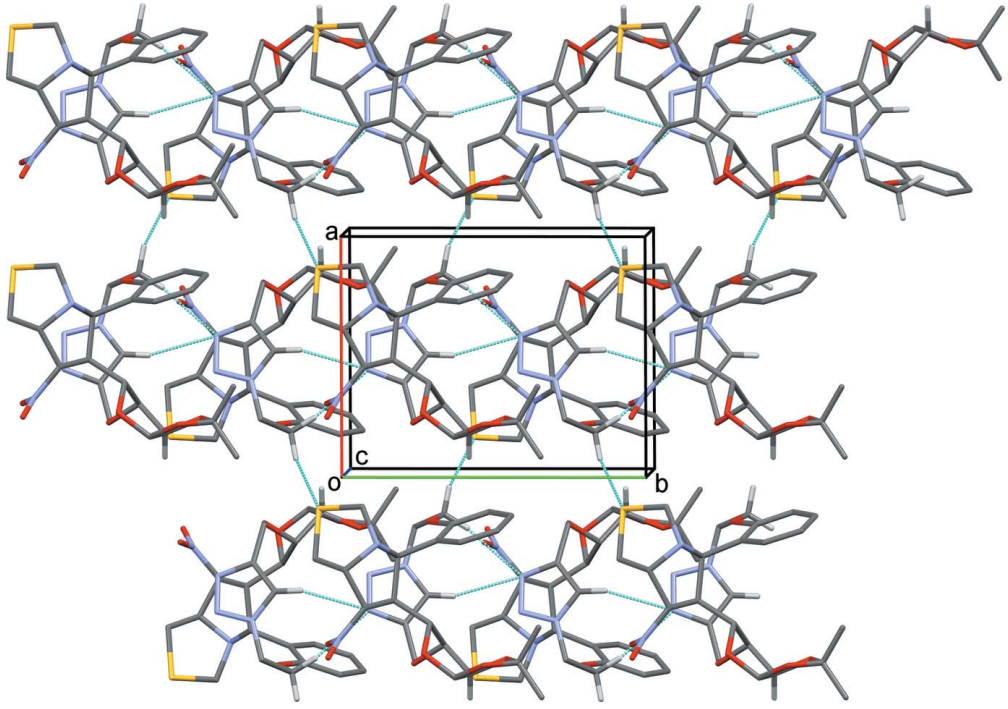


Figure 4
A view along the *c* axis of the crystal packing of compound (II), showing the hydrogen-bonded helical chains along [010], linked by C—H···S hydrogen bonds forming slabs parallel to the *ab* plane. H atoms not involved in hydrogen bonding (dashed lines; Table 2) have been excluded for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O4^{i}	0.97	2.51	3.295 (7)	138
C18—H18C···O2^{ii}	0.96	2.57	3.509 (9)	164
C25—H25A···N3^{iii}	0.97	2.62	3.589 (7)	173

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C23—H23···N3^{i}	0.93	2.58	3.433 (6)	152
C25—H25A···N3^{i}	0.97	2.60	3.553 (6)	168
C25—H25B···S9^{ii}	0.97	2.80	3.591 (4)	140

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

4. Synthesis and crystallization

Compound (I): A solution of 5-bromo-2-(2-{4-[({(3a*S*,6*R*,6a*S*)-2,2-dimethyl-5-[(*Z*)-2-nitrovinyl]tetrahydrofuro[2,3-*d*][1,3]dioxol-6-yl}oxy)methyl]-1*H*-1,2,3-triazol-1-yl}ethoxy)benzaldehyde (1 mmol) and proline (1.5 mmol) was refluxed in dry acetonitrile (50 ml) under a nitrogen atmosphere for 9 h. After completion of the reaction, as indicated by TLC, the acetonitrile was evaporated under reduced pressure. The crude product was purified by column chromatography using hexane/EtOAc (3:7) as eluent (yield 75%). After purification the compound was recrystallized in $CHCl_3$ by slow evaporation yielding colourless block-like crystals.

Compound (II): A solution of 5-bromo-2-(2-{4-[({(3a*S*,6*R*,6a*S*)-2,2-dimethyl-5-[(*Z*)-2-nitrovinyl]tetrahydrofuro[2,3-*d*][1,3]dioxol-6-yl}oxy)methyl]-1*H*-1,2,3-triazol-1-yl}ethoxy)benzaldehyde (1 mmol) and thiazolidine-4-carboxylic acid (1.5 m mol) was refluxed in dry acetonitrile (50 ml) under a nitrogen atmosphere for 9 h. After completion of reaction, as indicated by TLC,the acetonitrile was evaporated under reduced pressure. The crude product was purified by column chromatography using hexane/EtOAc (4:6) as eluent (yield 75%). After purification the compound was recrystallized in $CHCl_3$ by slow evaporation yielding colourless block-like crystals.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms were placed in calculated positions and refined as riding: C—H = 0.93–0.98 Å with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. Compound (I) was refined using the instructions TWIN/BASF (see Table 3).

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{25}H_{29}BrN_5O_7$	$C_{24}H_{29}N_5O_7S$
M_r	591.44	531.58
Crystal system, space group	Monoclinic, $P2_1$	Monoclinic, $P2_1$
Temperature (K)	293	293
a, b, c (Å)	9.913 (5), 11.414 (5), 12.144 (5)	8.756 (5), 10.811 (5), 13.569 (5)
β (°)	99.903 (5)	101.122 (5)
V (Å^3)	1353.6 (11)	1260.3 (10)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	1.57	0.18
Crystal size (mm)	0.20 × 0.15 × 0.10	0.20 × 0.15 × 0.10
Data collection		
Diffractometer	Bruker SMART APEXII area detector	Bruker SMART APEXII area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.744, 0.859	0.964, 0.982
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12444, 6278, 3587	11813, 4712, 2862
R_{int}	0.040	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.669	0.667
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.105, 0.95	0.046, 0.103, 1.00
No. of reflections	6278	4712
No. of parameters	346	336
No. of restraints	1	1
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.58, −0.46	0.17, −0.24
Absolute structure	Refined as an inversion twin.	Flack x determined using 794 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.007 (11)	−0.10 (9)

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008).

Acknowledgements

VV and DV thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collections. VV thanks the DBT, Government of India, for a fellowship.

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supporting information

Acta Cryst. (2015). E**71**, 827-831 [doi:10.1107/S205698901501141X]

Crystal structures of two triazola-dioxola-benzenacyclononaphanes

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (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).

(I) (*Z*)-1^5-Bromo-3^2,3^2-dimethyl-2^1-nitro-2^2,2^3,2^5,2^6,2^7,2^{7a},3^{3a},3^5,3^6,3^{6a}-decahydro-2^1*H*,6^1*H*-4,9-dioxa-2(3,2)-pyrrolizina-6(4,1)-triazola-3(5,6)-furo[2,3-*d*][1,3]dioxola-1(1,2)-benzenacyclononaphane

Crystal data

$C_{25}H_{29}BrN_5O_7$
M_r = 591.44
Monoclinic, $P2_1$
a = 9.913 (5) Å
b = 11.414 (5) Å
c = 12.144 (5) Å
β = 99.903 (5)°
V = 1353.6 (11) Å^3
Z = 2
$F(000)$ = 610
D_x = 1.451 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6278 reflections
θ = 1.7–28.4°
μ = 1.57 mm^{-1}
T = 293 K
Block, colourless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.744, T_{max} = 0.859
12444 measured reflections
6278 independent reflections
3587 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 28.4°, θ_{min} = 1.7°
h = −12→13
k = −14→15
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.105
S = 0.95
6278 reflections
346 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0281P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: −0.007 (11)

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}
Br1	0.67368 (6)	−0.19346 (6)	0.79878 (5)	0.0761 (2)
O1	0.1833 (5)	0.4138 (4)	0.6920 (4)	0.0819 (13)
O2	0.0613 (5)	0.4254 (4)	0.8212 (5)	0.0979 (16)
O3	0.2313 (4)	0.0525 (3)	0.9095 (3)	0.0615 (10)
O4	0.2390 (5)	−0.1342 (4)	0.9764 (4)	0.0874 (14)
O5	0.0701 (3)	−0.1835 (4)	0.8366 (3)	0.0654 (9)
O6	−0.0509 (3)	0.1090 (3)	0.7772 (3)	0.0521 (9)
O7	0.2747 (4)	0.1097 (3)	0.4964 (3)	0.0565 (9)
N1	0.4472 (4)	0.2353 (4)	0.8095 (3)	0.0452 (10)
N2	0.1474 (5)	0.3774 (4)	0.7764 (5)	0.0640 (14)
N3	−0.0839 (5)	0.2712 (4)	0.5621 (4)	0.0660 (13)
N4	−0.0173 (5)	0.2785 (4)	0.4772 (4)	0.0655 (12)
N5	−0.0008 (4)	0.1692 (4)	0.4422 (4)	0.0550 (11)
C1	0.3678 (5)	0.0384 (4)	0.5593 (4)	0.0477 (12)
C2	0.4117 (6)	−0.0673 (6)	0.5232 (5)	0.0692 (17)
H2	0.3791	−0.0924	0.4506	0.083*
C3	0.5033 (6)	−0.1362 (5)	0.5932 (5)	0.0659 (16)
H3	0.5325	−0.2075	0.5685	0.079*
C4	0.5503 (5)	−0.0981 (5)	0.6990 (5)	0.0521 (13)
C5	0.5094 (4)	0.0081 (4)	0.7364 (4)	0.0440 (12)
H5	0.5448	0.0330	0.8085	0.053*
C6	0.4170 (4)	0.0775 (4)	0.6685 (4)	0.0400 (11)
C7	0.3603 (4)	0.1881 (4)	0.7103 (4)	0.0387 (11)
H7	0.3533	0.2471	0.6509	0.046*
C8	0.5485 (5)	0.3221 (6)	0.7870 (4)	0.0616 (15)
H8A	0.5598	0.3183	0.7093	0.074*
H8B	0.6365	0.3074	0.8338	0.074*
C9	0.4947 (6)	0.4396 (6)	0.8129 (6)	0.0737 (18)
H9A	0.4350	0.4718	0.7483	0.088*
H9B	0.5689	0.4941	0.8371	0.088*
C10	0.4158 (6)	0.4135 (5)	0.9067 (5)	0.0657 (16)
H10A	0.3404	0.4679	0.9054	0.079*
H10B	0.4752	0.4184	0.9789	0.079*
C11	0.3630 (4)	0.2884 (4)	0.8834 (4)	0.0458 (12)
H11	0.3793	0.2453	0.9543	0.055*
C12	0.2136 (5)	0.2693 (4)	0.8294 (4)	0.0460 (12)
H12	0.1619	0.2414	0.8861	0.055*
C13	0.2173 (5)	0.1730 (4)	0.7424 (4)	0.0389 (11)
H13	0.1475	0.1897	0.6767	0.047*

C14	0.1887 (4)	0.0524 (4)	0.7888 (4)	0.0423 (11)
H14	0.2418	−0.0068	0.7562	0.051*
C15	0.1610 (6)	−0.0349 (5)	0.9550 (4)	0.0501 (14)
H15	0.1313	−0.0070	1.0234	0.060*
C16	0.1832 (6)	−0.2298 (4)	0.9097 (5)	0.0566 (14)
C17	0.2849 (7)	−0.2733 (7)	0.8436 (7)	0.101 (3)
H17A	0.2484	−0.3402	0.8007	0.152*
H17B	0.3672	−0.2952	0.8932	0.152*
H17C	0.3052	−0.2127	0.7942	0.152*
C18	0.1389 (8)	−0.3214 (6)	0.9836 (6)	0.087 (2)
H18A	0.0723	−0.2889	1.0237	0.130*
H18B	0.2168	−0.3482	1.0357	0.130*
H18C	0.0992	−0.3861	0.9389	0.130*
C19	0.0388 (5)	−0.0700 (4)	0.8692 (4)	0.0482 (13)
H19	−0.0472	−0.0670	0.8986	0.058*
C20	0.0399 (4)	0.0145 (4)	0.7713 (4)	0.0429 (11)
H20	0.0154	−0.0269	0.6999	0.051*
C21	−0.1598 (5)	0.1165 (6)	0.6832 (5)	0.0614 (15)
H21A	−0.2284	0.1712	0.6997	0.074*
H21B	−0.2028	0.0403	0.6694	0.074*
C22	−0.1072 (5)	0.1562 (5)	0.5805 (5)	0.0551 (14)
C23	−0.0554 (5)	0.0906 (5)	0.5042 (4)	0.0523 (13)
C24	0.0826 (6)	0.1482 (5)	0.3571 (4)	0.0568 (14)
H24A	0.1209	0.2219	0.3373	0.068*
H24B	0.0252	0.1173	0.2906	0.068*
C25	0.1960 (6)	0.0642 (5)	0.3954 (4)	0.0567 (14)
H25A	0.1591	−0.0121	0.4091	0.068*
H25B	0.2533	0.0557	0.3387	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0711 (3)	0.0748 (4)	0.0813 (4)	0.0282 (4)	0.0100 (3)	0.0041 (4)
O1	0.107 (3)	0.052 (3)	0.077 (3)	0.006 (2)	−0.011 (3)	0.013 (2)
O2	0.105 (3)	0.074 (3)	0.115 (4)	0.041 (3)	0.018 (3)	−0.016 (3)
O3	0.071 (2)	0.050 (2)	0.054 (2)	−0.015 (2)	−0.0156 (18)	0.0142 (19)
O4	0.098 (3)	0.046 (2)	0.096 (4)	−0.009 (2)	−0.047 (3)	0.010 (2)
O5	0.078 (2)	0.046 (2)	0.063 (2)	−0.002 (2)	−0.0130 (18)	−0.006 (2)
O6	0.0474 (18)	0.058 (2)	0.051 (2)	0.0146 (18)	0.0077 (16)	−0.0018 (17)
O7	0.077 (2)	0.046 (2)	0.041 (2)	0.0036 (19)	−0.0054 (18)	−0.0087 (17)
N1	0.047 (2)	0.046 (2)	0.040 (2)	−0.001 (2)	0.0014 (19)	−0.0033 (19)
N2	0.071 (3)	0.044 (3)	0.071 (4)	0.010 (3)	−0.005 (3)	−0.009 (3)
N3	0.082 (3)	0.046 (3)	0.063 (3)	0.018 (2)	−0.008 (2)	−0.002 (2)
N4	0.094 (3)	0.036 (3)	0.061 (3)	0.007 (2)	−0.002 (3)	0.004 (2)
N5	0.065 (3)	0.049 (3)	0.045 (3)	0.002 (2)	−0.007 (2)	−0.005 (2)
C1	0.056 (3)	0.046 (3)	0.040 (3)	0.001 (3)	0.008 (2)	−0.003 (2)
C2	0.088 (4)	0.068 (4)	0.050 (4)	0.015 (4)	0.008 (3)	−0.018 (3)
C3	0.077 (4)	0.055 (3)	0.067 (4)	0.021 (3)	0.017 (3)	−0.013 (3)

C4	0.048 (3)	0.051 (3)	0.059 (4)	0.005 (3)	0.015 (3)	0.001 (3)
C5	0.041 (2)	0.045 (3)	0.048 (3)	0.000 (2)	0.013 (2)	−0.006 (2)
C6	0.041 (2)	0.043 (3)	0.039 (3)	−0.002 (2)	0.014 (2)	−0.003 (2)
C7	0.049 (3)	0.035 (2)	0.031 (2)	−0.005 (2)	0.005 (2)	−0.004 (2)
C8	0.057 (3)	0.073 (4)	0.052 (3)	−0.018 (3)	0.001 (2)	−0.011 (3)
C9	0.075 (4)	0.054 (4)	0.089 (5)	−0.017 (3)	0.003 (4)	0.006 (3)
C10	0.074 (4)	0.043 (3)	0.074 (4)	−0.002 (3)	−0.003 (3)	−0.022 (3)
C11	0.057 (3)	0.041 (3)	0.038 (2)	0.003 (3)	0.007 (2)	0.002 (2)
C12	0.058 (3)	0.035 (3)	0.046 (3)	0.006 (2)	0.012 (2)	−0.001 (2)
C13	0.048 (3)	0.031 (2)	0.037 (3)	0.003 (2)	0.003 (2)	−0.002 (2)
C14	0.046 (2)	0.039 (3)	0.041 (3)	0.001 (2)	0.004 (2)	0.002 (2)
C15	0.068 (3)	0.048 (3)	0.035 (3)	−0.007 (3)	0.011 (3)	−0.004 (2)
C16	0.068 (3)	0.041 (3)	0.057 (3)	−0.008 (3)	−0.001 (3)	0.012 (3)
C17	0.093 (5)	0.072 (5)	0.149 (8)	0.017 (4)	0.051 (5)	0.031 (5)
C18	0.135 (6)	0.062 (4)	0.064 (4)	−0.021 (4)	0.018 (4)	0.013 (4)
C19	0.050 (3)	0.046 (3)	0.049 (3)	−0.008 (3)	0.009 (2)	−0.001 (3)
C20	0.045 (3)	0.045 (3)	0.040 (3)	0.004 (2)	0.010 (2)	−0.002 (2)
C21	0.043 (3)	0.077 (4)	0.063 (4)	0.015 (3)	0.006 (3)	−0.003 (3)
C22	0.048 (3)	0.061 (4)	0.051 (3)	0.010 (3)	−0.007 (2)	−0.001 (3)
C23	0.058 (3)	0.056 (3)	0.039 (3)	−0.006 (3)	−0.003 (2)	0.004 (3)
C24	0.082 (4)	0.053 (3)	0.032 (3)	−0.008 (3)	−0.001 (3)	0.005 (2)
C25	0.080 (3)	0.056 (3)	0.033 (3)	−0.012 (3)	0.007 (3)	−0.008 (3)

Geometric parameters (Å, º)

Br1—C4	1.908 (5)	C9—C10	1.519 (8)
O1—N2	1.214 (6)	C9—H9A	0.9700
O2—N2	1.218 (6)	C9—H9B	0.9700
O3—C15	1.385 (6)	C10—C11	1.530 (8)
O3—C14	1.454 (5)	C10—H10A	0.9700
O4—C15	1.372 (7)	C10—H10B	0.9700
O4—C16	1.414 (7)	C11—C12	1.529 (7)
O5—C19	1.405 (6)	C11—H11	0.9800
O5—C16	1.408 (6)	C12—C13	1.530 (7)
O6—C20	1.415 (6)	C12—H12	0.9800
O6—C21	1.433 (6)	C13—C14	1.533 (7)
O7—C1	1.362 (6)	C13—H13	0.9800
O7—C25	1.432 (6)	C14—C20	1.516 (6)
N1—C7	1.458 (6)	C14—H14	0.9800
N1—C11	1.459 (6)	C15—C19	1.510 (7)
N1—C8	1.469 (6)	C15—H15	0.9800
N2—C12	1.490 (7)	C16—C17	1.478 (8)
N3—N4	1.319 (6)	C16—C18	1.492 (8)
N3—C22	1.358 (7)	C17—H17A	0.9600
N4—N5	1.337 (6)	C17—H17B	0.9600
N5—C23	1.344 (6)	C17—H17C	0.9600
N5—C24	1.450 (6)	C18—H18A	0.9600
C1—C2	1.380 (8)	C18—H18B	0.9600

C1—C6	1.405 (7)	C18—H18C	0.9600
C2—C3	1.378 (8)	C19—C20	1.533 (7)
C2—H2	0.9300	C19—H19	0.9800
C3—C4	1.361 (8)	C20—H20	0.9800
C3—H3	0.9300	C21—C22	1.501 (8)
C4—C5	1.380 (7)	C21—H21A	0.9700
C5—C6	1.373 (7)	C21—H21B	0.9700
C5—H5	0.9300	C22—C23	1.359 (7)
C6—C7	1.506 (6)	C24—C25	1.490 (8)
C7—C13	1.542 (6)	C24—H24A	0.9700
C7—H7	0.9800	C24—H24B	0.9700
C8—C9	1.497 (9)	C25—H25A	0.9700
C8—H8A	0.9700	C25—H25B	0.9700
C8—H8B	0.9700		

C15—O3—C14	108.8 (4)	C12—C13—C14	111.3 (4)
C15—O4—C16	112.1 (4)	C12—C13—C7	103.1 (4)
C19—O5—C16	111.0 (4)	C14—C13—C7	115.5 (4)
C20—O6—C21	113.8 (4)	C12—C13—H13	108.9
C1—O7—C25	118.7 (4)	C14—C13—H13	108.9
C7—N1—C11	110.0 (3)	C7—C13—H13	108.9
C7—N1—C8	114.9 (4)	O3—C14—C20	104.4 (3)
C11—N1—C8	108.4 (4)	O3—C14—C13	109.3 (4)
O1—N2—O2	123.5 (6)	C20—C14—C13	116.2 (4)
O1—N2—C12	118.5 (5)	O3—C14—H14	108.9
O2—N2—C12	118.0 (5)	C20—C14—H14	108.9
N4—N3—C22	108.2 (4)	C13—C14—H14	108.9
N3—N4—N5	107.1 (4)	O4—C15—O3	111.4 (4)
N4—N5—C23	111.2 (4)	O4—C15—C19	105.7 (4)
N4—N5—C24	119.7 (5)	O3—C15—C19	108.3 (4)
C23—N5—C24	128.6 (4)	O4—C15—H15	110.4
O7—C1—C2	124.4 (5)	O3—C15—H15	110.4
O7—C1—C6	115.6 (4)	C19—C15—H15	110.4
C2—C1—C6	120.1 (5)	O5—C16—O4	105.2 (4)
C3—C2—C1	120.8 (5)	O5—C16—C17	109.1 (5)
C3—C2—H2	119.6	O4—C16—C17	109.5 (5)
C1—C2—H2	119.6	O5—C16—C18	110.9 (5)
C4—C3—C2	118.9 (5)	O4—C16—C18	108.8 (5)
C4—C3—H3	120.6	C17—C16—C18	113.0 (5)
C2—C3—H3	120.6	C16—C17—H17A	109.5
C3—C4—C5	121.3 (5)	C16—C17—H17B	109.5
C3—C4—Br1	119.7 (4)	H17A—C17—H17B	109.5
C5—C4—Br1	119.0 (4)	C16—C17—H17C	109.5
C6—C5—C4	120.8 (5)	H17A—C17—H17C	109.5
C6—C5—H5	119.6	H17B—C17—H17C	109.5
C4—C5—H5	119.6	C16—C18—H18A	109.5
C5—C6—C1	118.1 (4)	C16—C18—H18B	109.5
C5—C6—C7	122.0 (4)	H18A—C18—H18B	109.5

C1—C6—C7	119.7 (4)	C16—C18—H18C	109.5
N1—C7—C6	112.7 (4)	H18A—C18—H18C	109.5
N1—C7—C13	105.7 (3)	H18B—C18—H18C	109.5
C6—C7—C13	113.8 (4)	O5—C19—C15	104.5 (4)
N1—C7—H7	108.1	O5—C19—C20	109.3 (4)
C6—C7—H7	108.1	C15—C19—C20	104.9 (4)
C13—C7—H7	108.1	O5—C19—H19	112.5
N1—C8—C9	106.6 (4)	C15—C19—H19	112.5
N1—C8—H8A	110.4	C20—C19—H19	112.5
C9—C8—H8A	110.4	O6—C20—C14	112.8 (4)
N1—C8—H8B	110.4	O6—C20—C19	110.5 (3)
C9—C8—H8B	110.4	C14—C20—C19	102.0 (4)
H8A—C8—H8B	108.6	O6—C20—H20	110.4
C8—C9—C10	103.3 (5)	C14—C20—H20	110.4
C8—C9—H9A	111.1	C19—C20—H20	110.4
C10—C9—H9A	111.1	O6—C21—C22	111.0 (4)
C8—C9—H9B	111.1	O6—C21—H21A	109.4
C10—C9—H9B	111.1	C22—C21—H21A	109.4
H9A—C9—H9B	109.1	O6—C21—H21B	109.4
C9—C10—C11	104.4 (4)	C22—C21—H21B	109.4
C9—C10—H10A	110.9	H21A—C21—H21B	108.0
C11—C10—H10A	110.9	N3—C22—C23	109.0 (5)
C9—C10—H10B	110.9	N3—C22—C21	121.5 (5)
C11—C10—H10B	110.9	C23—C22—C21	128.7 (5)
H10A—C10—H10B	108.9	N5—C23—C22	104.5 (5)
N1—C11—C12	106.9 (4)	N5—C24—C25	112.1 (4)
N1—C11—C10	106.7 (4)	N5—C24—H24A	109.2
C12—C11—C10	119.2 (4)	C25—C24—H24A	109.2
N1—C11—H11	107.9	N5—C24—H24B	109.2
C12—C11—H11	107.9	C25—C24—H24B	109.2
C10—C11—H11	107.9	H24A—C24—H24B	107.9
N2—C12—C11	112.9 (4)	O7—C25—C24	107.7 (4)
N2—C12—C13	111.0 (4)	O7—C25—H25A	110.2
C11—C12—C13	105.2 (4)	C24—C25—H25A	110.2
N2—C12—H12	109.2	O7—C25—H25B	110.2
C11—C12—H12	109.2	C24—C25—H25B	110.2
C13—C12—H12	109.2	H25A—C25—H25B	108.5

C22—N3—N4—N5	0.7 (6)	C6—C7—C13—C12	153.8 (4)
N3—N4—N5—C23	−0.4 (6)	N1—C7—C13—C14	−92.1 (5)
N3—N4—N5—C24	−173.2 (4)	C6—C7—C13—C14	32.2 (5)
C25—O7—C1—C2	13.2 (7)	C15—O3—C14—C20	32.4 (5)
C25—O7—C1—C6	−165.4 (4)	C15—O3—C14—C13	157.4 (4)
O7—C1—C2—C3	−178.0 (5)	C12—C13—C14—O3	−27.6 (5)
C6—C1—C2—C3	0.4 (8)	C7—C13—C14—O3	89.5 (4)
C1—C2—C3—C4	−0.2 (9)	C12—C13—C14—C20	90.2 (5)
C2—C3—C4—C5	−0.8 (8)	C7—C13—C14—C20	−152.8 (4)
C2—C3—C4—Br1	178.5 (4)	C16—O4—C15—O3	−114.9 (5)

C3—C4—C5—C6	1.6 (7)	C16—O4—C15—C19	2.5 (6)
Br1—C4—C5—C6	−177.7 (3)	C14—O3—C15—O4	98.4 (5)
C4—C5—C6—C1	−1.3 (6)	C14—O3—C15—C19	−17.4 (5)
C4—C5—C6—C7	174.1 (4)	C19—O5—C16—O4	−11.1 (5)
O7—C1—C6—C5	178.9 (4)	C19—O5—C16—C17	−128.5 (5)
C2—C1—C6—C5	0.3 (7)	C19—O5—C16—C18	106.4 (5)
O7—C1—C6—C7	3.4 (6)	C15—O4—C16—O5	4.9 (6)
C2—C1—C6—C7	−175.2 (4)	C15—O4—C16—C17	122.1 (5)
C11—N1—C7—C6	−145.0 (4)	C15—O4—C16—C18	−114.0 (5)
C8—N1—C7—C6	92.3 (5)	C16—O5—C19—C15	12.4 (5)
C11—N1—C7—C13	−20.1 (5)	C16—O5—C19—C20	124.2 (4)
C8—N1—C7—C13	−142.8 (4)	O4—C15—C19—O5	−8.9 (5)
C5—C6—C7—N1	21.8 (6)	O3—C15—C19—O5	110.5 (4)
C1—C6—C7—N1	−162.9 (4)	O4—C15—C19—C20	−123.9 (4)
C5—C6—C7—C13	−98.6 (5)	O3—C15—C19—C20	−4.4 (5)
C1—C6—C7—C13	76.7 (5)	C21—O6—C20—C14	128.0 (4)
C7—N1—C8—C9	103.5 (5)	C21—O6—C20—C19	−118.6 (4)
C11—N1—C8—C9	−20.0 (5)	O3—C14—C20—O6	85.4 (4)
N1—C8—C9—C10	31.6 (6)	C13—C14—C20—O6	−35.0 (5)
C8—C9—C10—C11	−30.9 (6)	O3—C14—C20—C19	−33.1 (5)
C7—N1—C11—C12	2.2 (5)	C13—C14—C20—C19	−153.5 (4)
C8—N1—C11—C12	128.6 (4)	O5—C19—C20—O6	151.2 (4)
C7—N1—C11—C10	−126.4 (4)	C15—C19—C20—O6	−97.2 (4)
C8—N1—C11—C10	0.0 (5)	O5—C19—C20—C14	−88.6 (4)
C9—C10—C11—N1	19.4 (6)	C15—C19—C20—C14	22.9 (5)
C9—C10—C11—C12	−101.6 (5)	C20—O6—C21—C22	−71.5 (6)
O1—N2—C12—C11	70.1 (6)	N4—N3—C22—C23	−0.8 (6)
O2—N2—C12—C11	−108.9 (5)	N4—N3—C22—C21	169.5 (4)
O1—N2—C12—C13	−47.7 (6)	O6—C21—C22—N3	−81.0 (6)
O2—N2—C12—C13	133.3 (5)	O6—C21—C22—C23	87.3 (7)
N1—C11—C12—N2	−104.5 (5)	N4—N5—C23—C22	−0.1 (6)
C10—C11—C12—N2	16.4 (6)	C24—N5—C23—C22	172.0 (5)
N1—C11—C12—C13	16.7 (5)	N3—C22—C23—N5	0.5 (6)
C10—C11—C12—C13	137.6 (4)	C21—C22—C23—N5	−168.9 (5)
N2—C12—C13—C14	−141.1 (4)	N4—N5—C24—C25	123.3 (5)
C11—C12—C13—C14	96.5 (4)	C23—N5—C24—C25	−48.1 (7)
N2—C12—C13—C7	94.6 (5)	C1—O7—C25—C24	167.4 (4)
C11—C12—C13—C7	−27.9 (5)	N5—C24—C25—O7	−56.2 (6)
N1—C7—C13—C12	29.5 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*B*···O4^{i}	0.97	2.51	3.295 (7)	138
C18—H18*C*···O2^{ii}	0.96	2.57	3.509 (9)	164
C25—H25*A*···N3^{iii}	0.97	2.62	3.589 (7)	173

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

(II) (*Z*)-3^2,3^2-Dimethyl-2^7-nitro-2^5,2^6,2^7,2^{7a},3^{3a},3^5,3^6,3^{6a}-octahydro-2^1*H*,2^3*H*,6^1*H*-4,9-dioxa-2(5,6)-pyrrolo[1,2-*c*]thiazola-6(4,1)-triazola-3(5,6)-furo[2,3-*d*][1,3]dioxola-1(1,2)-benzenacyclononaphane

Crystal data

$C_{24}H_{29}N_5O_7S$
M_r = 531.58
Monoclinic, $P2_1$
a = 8.756 (5) Å
b = 10.811 (5) Å
c = 13.569 (5) Å
β = 101.122 (5)°
V = 1260.3 (10) Å^3
Z = 2
$F(000)$ = 560
D_x = 1.401 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4712 reflections
θ = 1.5–22.3°
μ = 0.18 mm^{-1}
T = 293 K
Block, colourless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.964, T_{max} = 0.982
11813 measured reflections
4712 independent reflections
2862 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 28.3°, θ_{min} = 1.5°
h = −11→11
k = −13→14
l = −17→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.103
S = 1.00
4712 reflections
336 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack x determined using 794 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.10 (9)

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}
S9	0.85963 (16)	−0.08380 (12)	0.26658 (11)	0.0856 (5)
O1	0.2482 (4)	−0.0495 (3)	0.0593 (3)	0.0996 (13)
O2	0.2983 (4)	−0.0548 (3)	0.2195 (3)	0.0873 (11)
O3	0.2622 (3)	0.2632 (2)	0.11689 (17)	0.0547 (7)
O4	0.2164 (3)	0.4638 (3)	0.06070 (18)	0.0578 (7)
O5	0.2374 (4)	0.5327 (2)	0.2191 (2)	0.0631 (8)
O6	0.2153 (3)	0.2254 (2)	0.31708 (18)	0.0496 (7)

O7	0.8007 (3)	0.2715 (2)	0.43718 (18)	0.0538 (7)
N1	0.7152 (3)	0.1098 (3)	0.1692 (2)	0.0434 (8)
N2	0.3210 (4)	−0.0163 (3)	0.1400 (3)	0.0598 (10)
N3	0.4307 (4)	0.0753 (3)	0.4787 (3)	0.0597 (9)
N4	0.5760 (4)	0.0738 (3)	0.5266 (3)	0.0600 (10)
N5	0.6117 (4)	0.1899 (3)	0.5601 (2)	0.0521 (9)
C1	0.7925 (4)	0.3522 (4)	0.3582 (3)	0.0441 (9)
C2	0.8434 (5)	0.4733 (4)	0.3693 (3)	0.0553 (11)
H2	0.8865	0.5038	0.4326	0.066*
C3	0.8302 (5)	0.5485 (4)	0.2866 (4)	0.0615 (12)
H3	0.8615	0.6307	0.2946	0.074*
C4	0.7715 (4)	0.5040 (4)	0.1926 (4)	0.0558 (12)
H4	0.7642	0.5551	0.1368	0.067*
C5	0.7234 (4)	0.3827 (4)	0.1817 (3)	0.0512 (10)
H5	0.6863	0.3518	0.1176	0.061*
C6	0.7288 (4)	0.3054 (4)	0.2638 (3)	0.0400 (9)
C7	0.6631 (4)	0.1762 (3)	0.2522 (3)	0.0404 (9)
H7	0.7020	0.1310	0.3146	0.049*
C8	0.8712 (5)	0.0606 (4)	0.1945 (3)	0.0629 (12)
H8B	0.9400	0.1196	0.2348	0.076*
H8A	0.9110	0.0431	0.1340	0.076*
C10	0.6519 (5)	−0.0954 (4)	0.2256 (3)	0.0644 (12)
H10A	0.6232	−0.1768	0.1980	0.077*
H10B	0.5990	−0.0806	0.2811	0.077*
C11	0.6090 (4)	0.0046 (4)	0.1445 (3)	0.0458 (10)
H11	0.6163	−0.0289	0.0784	0.055*
C12	0.4536 (4)	0.0711 (3)	0.1392 (3)	0.0405 (9)
H12	0.4314	0.1185	0.0765	0.049*
C13	0.4804 (4)	0.1625 (3)	0.2271 (2)	0.0359 (8)
H13	0.4481	0.1222	0.2844	0.043*
C14	0.3855 (4)	0.2812 (3)	0.2042 (2)	0.0377 (8)
H14	0.4542	0.3478	0.1900	0.045*
C15	0.1637 (5)	0.3661 (3)	0.1128 (3)	0.0474 (10)
H15	0.0560	0.3442	0.0832	0.057*
C16	0.2264 (5)	0.5744 (4)	0.1192 (3)	0.0458 (10)
C17	0.3722 (5)	0.6412 (5)	0.1111 (4)	0.0824 (16)
H17A	0.4588	0.5852	0.1255	0.124*
H17B	0.3881	0.7083	0.1582	0.124*
H17C	0.3638	0.6731	0.0442	0.124*
C18	0.0832 (5)	0.6512 (4)	0.0852 (3)	0.0667 (13)
H18C	0.0792	0.6775	0.0171	0.100*
H18B	0.0864	0.7224	0.1278	0.100*
H18A	−0.0075	0.6029	0.0888	0.100*
C19	0.1766 (4)	0.4125 (4)	0.2205 (3)	0.0453 (9)
H19	0.0763	0.4113	0.2423	0.054*
C20	0.2947 (4)	0.3260 (3)	0.2824 (3)	0.0398 (9)
H20	0.3617	0.3694	0.3379	0.048*
C21	0.2186 (5)	0.2264 (5)	0.4235 (3)	0.0630 (12)

H21A	0.1419	0.1685	0.4389	0.076*
H21B	0.1911	0.3083	0.4436	0.076*
C22	0.3746 (5)	0.1924 (4)	0.4809 (3)	0.0504 (10)
C23	0.4913 (5)	0.2649 (4)	0.5322 (3)	0.0548 (11)
H23	0.4870	0.3492	0.5449	0.066*
C24	0.7723 (5)	0.2219 (4)	0.6012 (3)	0.0624 (12)
H24A	0.8373	0.1489	0.6036	0.075*
H24B	0.7806	0.2527	0.6692	0.075*
C25	0.8274 (5)	0.3194 (4)	0.5369 (3)	0.0585 (11)
H25A	0.7699	0.3957	0.5393	0.070*
H25B	0.9373	0.3361	0.5603	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S9	0.0720 (8)	0.0642 (8)	0.1050 (10)	0.0174 (7)	−0.0215 (7)	0.0085 (8)
O1	0.097 (3)	0.087 (3)	0.096 (3)	−0.032 (2)	−0.028 (2)	−0.016 (2)
O2	0.104 (3)	0.072 (3)	0.095 (3)	−0.032 (2)	0.042 (2)	−0.011 (2)
O3	0.0668 (19)	0.0457 (16)	0.0421 (15)	0.0084 (15)	−0.0129 (13)	−0.0093 (13)
O4	0.085 (2)	0.0445 (16)	0.0423 (15)	0.0019 (16)	0.0087 (14)	0.0044 (14)
O5	0.107 (2)	0.0362 (15)	0.0420 (17)	0.0049 (16)	0.0050 (16)	0.0013 (13)
O6	0.0474 (15)	0.0523 (17)	0.0492 (16)	−0.0038 (13)	0.0098 (12)	0.0093 (13)
O7	0.0715 (19)	0.0501 (17)	0.0370 (14)	−0.0061 (15)	0.0041 (12)	−0.0081 (13)
N1	0.0393 (18)	0.0464 (19)	0.0439 (18)	0.0011 (15)	0.0064 (14)	−0.0083 (15)
N2	0.063 (2)	0.040 (2)	0.075 (3)	−0.001 (2)	0.008 (2)	−0.010 (2)
N3	0.068 (3)	0.048 (2)	0.059 (2)	0.000 (2)	0.0015 (19)	0.0108 (18)
N4	0.075 (3)	0.039 (2)	0.059 (2)	−0.0001 (19)	−0.004 (2)	0.0038 (18)
N5	0.066 (2)	0.046 (2)	0.0411 (19)	0.0018 (19)	0.0010 (16)	0.0059 (16)
C1	0.040 (2)	0.042 (2)	0.049 (2)	−0.0014 (18)	0.0069 (18)	−0.002 (2)
C2	0.054 (3)	0.052 (3)	0.061 (3)	−0.006 (2)	0.012 (2)	−0.010 (2)
C3	0.049 (3)	0.041 (2)	0.096 (4)	−0.005 (2)	0.020 (3)	0.000 (3)
C4	0.039 (2)	0.053 (3)	0.073 (3)	−0.003 (2)	0.004 (2)	0.020 (2)
C5	0.044 (2)	0.050 (3)	0.056 (3)	−0.003 (2)	−0.0004 (19)	0.007 (2)
C6	0.035 (2)	0.040 (2)	0.044 (2)	0.0013 (17)	0.0037 (16)	0.0001 (18)
C7	0.042 (2)	0.040 (2)	0.037 (2)	0.0005 (18)	0.0015 (16)	−0.0002 (17)
C8	0.044 (2)	0.067 (3)	0.076 (3)	0.012 (2)	0.005 (2)	−0.008 (3)
C10	0.069 (3)	0.046 (2)	0.074 (3)	0.008 (2)	0.002 (2)	0.005 (2)
C11	0.045 (2)	0.049 (2)	0.039 (2)	0.0041 (19)	−0.0012 (17)	−0.0080 (18)
C12	0.045 (2)	0.036 (2)	0.040 (2)	−0.0010 (18)	0.0073 (16)	−0.0034 (17)
C13	0.040 (2)	0.035 (2)	0.0319 (19)	0.0008 (17)	0.0051 (15)	0.0013 (16)
C14	0.039 (2)	0.038 (2)	0.0340 (19)	−0.0019 (17)	0.0022 (15)	−0.0017 (16)
C15	0.044 (2)	0.038 (2)	0.053 (3)	0.0008 (19)	−0.0082 (18)	0.0029 (19)
C16	0.052 (2)	0.037 (2)	0.046 (2)	−0.001 (2)	0.0051 (18)	0.0005 (19)
C17	0.056 (3)	0.068 (3)	0.124 (4)	−0.008 (3)	0.019 (3)	0.008 (3)
C18	0.061 (3)	0.059 (3)	0.080 (3)	0.015 (2)	0.012 (2)	0.016 (3)
C19	0.044 (2)	0.044 (2)	0.050 (2)	0.005 (2)	0.0130 (18)	0.005 (2)
C20	0.042 (2)	0.035 (2)	0.042 (2)	−0.0008 (18)	0.0079 (16)	0.0038 (18)
C21	0.060 (3)	0.077 (3)	0.057 (3)	0.008 (3)	0.025 (2)	0.018 (3)

C22	0.055 (3)	0.053 (3)	0.045 (2)	0.006 (2)	0.014 (2)	0.014 (2)
C23	0.072 (3)	0.044 (2)	0.051 (2)	0.008 (2)	0.017 (2)	0.006 (2)
C24	0.073 (3)	0.072 (3)	0.038 (2)	−0.006 (3)	0.000 (2)	0.001 (2)
C25	0.061 (3)	0.069 (3)	0.042 (2)	−0.009 (2)	0.0005 (19)	−0.014 (2)

Geometric parameters (Å, º)

S9—C10	1.801 (4)	C8—H8B	0.9700
S9—C8	1.855 (5)	C8—H8A	0.9700
O1—N2	1.210 (4)	C10—C11	1.536 (6)
O2—N2	1.207 (4)	C10—H10A	0.9700
O3—C15	1.402 (4)	C10—H10B	0.9700
O3—C14	1.454 (4)	C11—C12	1.528 (5)
O4—C15	1.398 (5)	C11—H11	0.9800
O4—C16	1.428 (5)	C12—C13	1.532 (5)
O5—C19	1.406 (5)	C12—H12	0.9800
O5—C16	1.415 (4)	C13—C14	1.528 (5)
O6—C20	1.418 (4)	C13—H13	0.9800
O6—C21	1.439 (4)	C14—C20	1.523 (5)
O7—C1	1.373 (4)	C14—H14	0.9800
O7—C25	1.425 (4)	C15—C19	1.528 (5)
N1—C8	1.445 (5)	C15—H15	0.9800
N1—C11	1.466 (5)	C16—C17	1.490 (6)
N1—C7	1.480 (4)	C16—C18	1.501 (5)
N2—C12	1.498 (5)	C17—H17A	0.9600
N3—N4	1.312 (4)	C17—H17B	0.9600
N3—C22	1.360 (5)	C17—H17C	0.9600
N4—N5	1.351 (4)	C18—H18C	0.9600
N5—C23	1.326 (5)	C18—H18B	0.9600
N5—C24	1.451 (5)	C18—H18A	0.9600
C1—C2	1.382 (6)	C19—C20	1.521 (5)
C1—C6	1.390 (5)	C19—H19	0.9800
C2—C3	1.372 (6)	C20—H20	0.9800
C2—H2	0.9300	C21—C22	1.483 (6)
C3—C4	1.366 (6)	C21—H21A	0.9700
C3—H3	0.9300	C21—H21B	0.9700
C4—C5	1.377 (6)	C22—C23	1.366 (6)
C4—H4	0.9300	C23—H23	0.9300
C5—C6	1.385 (5)	C24—C25	1.506 (6)
C5—H5	0.9300	C24—H24A	0.9700
C6—C7	1.508 (5)	C24—H24B	0.9700
C7—C13	1.576 (5)	C25—H25A	0.9700
C7—H7	0.9800	C25—H25B	0.9700
C10—S9—C8	92.9 (2)	C12—C13—H13	107.8
C15—O3—C14	106.3 (3)	C7—C13—H13	107.8
C15—O4—C16	110.0 (3)	O3—C14—C20	101.8 (3)
C19—O5—C16	110.5 (3)	O3—C14—C13	110.0 (3)

C20—O6—C21	114.3 (3)	C20—C14—C13	117.5 (3)
C1—O7—C25	119.0 (3)	O3—C14—H14	109.0
C8—N1—C11	107.3 (3)	C20—C14—H14	109.0
C8—N1—C7	114.5 (3)	C13—C14—H14	109.0
C11—N1—C7	106.0 (3)	O4—C15—O3	111.0 (3)
O2—N2—O1	123.8 (4)	O4—C15—C19	105.4 (3)
O2—N2—C12	119.1 (4)	O3—C15—C19	106.9 (3)
O1—N2—C12	117.0 (4)	O4—C15—H15	111.1
N4—N3—C22	108.7 (4)	O3—C15—H15	111.1
N3—N4—N5	107.1 (3)	C19—C15—H15	111.1
C23—N5—N4	110.6 (4)	O5—C16—O4	104.6 (3)
C23—N5—C24	128.5 (4)	O5—C16—C17	108.9 (4)
N4—N5—C24	119.7 (4)	O4—C16—C17	109.0 (4)
O7—C1—C2	123.3 (4)	O5—C16—C18	111.7 (3)
O7—C1—C6	116.1 (3)	O4—C16—C18	109.5 (3)
C2—C1—C6	120.6 (4)	C17—C16—C18	112.7 (4)
C3—C2—C1	119.8 (4)	C16—C17—H17A	109.5
C3—C2—H2	120.1	C16—C17—H17B	109.5
C1—C2—H2	120.1	H17A—C17—H17B	109.5
C4—C3—C2	120.8 (4)	C16—C17—H17C	109.5
C4—C3—H3	119.6	H17A—C17—H17C	109.5
C2—C3—H3	119.6	H17B—C17—H17C	109.5
C3—C4—C5	119.2 (4)	C16—C18—H18C	109.5
C3—C4—H4	120.4	C16—C18—H18B	109.5
C5—C4—H4	120.4	H18C—C18—H18B	109.5
C4—C5—C6	121.7 (4)	C16—C18—H18A	109.5
C4—C5—H5	119.1	H18C—C18—H18A	109.5
C6—C5—H5	119.1	H18B—C18—H18A	109.5
C5—C6—C1	117.8 (4)	O5—C19—C20	110.9 (3)
C5—C6—C7	121.5 (3)	O5—C19—C15	104.4 (3)
C1—C6—C7	120.7 (3)	C20—C19—C15	104.1 (3)
N1—C7—C6	111.2 (3)	O5—C19—H19	112.3
N1—C7—C13	103.8 (3)	C20—C19—H19	112.3
C6—C7—C13	117.4 (3)	C15—C19—H19	112.3
N1—C7—H7	108.0	O6—C20—C19	109.3 (3)
C6—C7—H7	108.0	O6—C20—C14	110.1 (3)
C13—C7—H7	108.0	C19—C20—C14	101.6 (3)
N1—C8—S9	106.9 (3)	O6—C20—H20	111.8
N1—C8—H8B	110.4	C19—C20—H20	111.8
S9—C8—H8B	110.4	C14—C20—H20	111.8
N1—C8—H8A	110.4	O6—C21—C22	111.3 (3)
S9—C8—H8A	110.4	O6—C21—H21A	109.4
H8B—C8—H8A	108.6	C22—C21—H21A	109.4
C11—C10—S9	105.4 (3)	O6—C21—H21B	109.4
C11—C10—H10A	110.7	C22—C21—H21B	109.4
S9—C10—H10A	110.7	H21A—C21—H21B	108.0
C11—C10—H10B	110.7	N3—C22—C23	107.9 (4)
S9—C10—H10B	110.7	N3—C22—C21	121.3 (4)

H10A—C10—H10B	108.8	C23—C22—C21	130.3 (4)
N1—C11—C12	99.5 (3)	N5—C23—C22	105.7 (4)
N1—C11—C10	109.0 (3)	N5—C23—H23	127.2
C12—C11—C10	117.2 (4)	C22—C23—H23	127.2
N1—C11—H11	110.2	N5—C24—C25	109.8 (3)
C12—C11—H11	110.2	N5—C24—H24A	109.7
C10—C11—H11	110.2	C25—C24—H24A	109.7
N2—C12—C11	112.8 (3)	N5—C24—H24B	109.7
N2—C12—C13	113.8 (3)	C25—C24—H24B	109.7
C11—C12—C13	105.4 (3)	H24A—C24—H24B	108.2
N2—C12—H12	108.2	O7—C25—C24	106.5 (3)
C11—C12—H12	108.2	O7—C25—H25A	110.4
C13—C12—H12	108.2	C24—C25—H25A	110.4
C14—C13—C12	112.9 (3)	O7—C25—H25B	110.4
C14—C13—C7	117.0 (3)	C24—C25—H25B	110.4
C12—C13—C7	103.0 (3)	H25A—C25—H25B	108.6
C14—C13—H13	107.8		

C22—N3—N4—N5	0.6 (4)	N1—C7—C13—C12	8.4 (3)
N3—N4—N5—C23	−1.2 (4)	C6—C7—C13—C12	131.5 (3)
N3—N4—N5—C24	−169.7 (3)	C15—O3—C14—C20	43.5 (3)
C25—O7—C1—C2	15.5 (5)	C15—O3—C14—C13	168.8 (3)
C25—O7—C1—C6	−164.5 (3)	C12—C13—C14—O3	16.1 (4)
O7—C1—C2—C3	−179.6 (4)	C7—C13—C14—O3	135.5 (3)
C6—C1—C2—C3	0.4 (6)	C12—C13—C14—C20	131.9 (3)
C1—C2—C3—C4	−2.0 (6)	C7—C13—C14—C20	−108.7 (4)
C2—C3—C4—C5	0.9 (6)	C16—O4—C15—O3	−128.7 (3)
C3—C4—C5—C6	1.9 (6)	C16—O4—C15—C19	−13.3 (4)
C4—C5—C6—C1	−3.4 (6)	C14—O3—C15—O4	86.5 (3)
C4—C5—C6—C7	174.6 (4)	C14—O3—C15—C19	−28.0 (4)
O7—C1—C6—C5	−177.8 (3)	C19—O5—C16—O4	−22.5 (4)
C2—C1—C6—C5	2.2 (5)	C19—O5—C16—C17	−138.9 (4)
O7—C1—C6—C7	4.2 (5)	C19—O5—C16—C18	96.0 (4)
C2—C1—C6—C7	−175.8 (4)	C15—O4—C16—O5	22.0 (4)
C8—N1—C7—C6	79.3 (4)	C15—O4—C16—C17	138.3 (4)
C11—N1—C7—C6	−162.6 (3)	C15—O4—C16—C18	−97.9 (4)
C8—N1—C7—C13	−153.6 (3)	C16—O5—C19—C20	126.0 (3)
C11—N1—C7—C13	−35.5 (3)	C16—O5—C19—C15	14.4 (4)
C5—C6—C7—N1	47.2 (4)	O4—C15—C19—O5	−0.6 (4)
C1—C6—C7—N1	−134.8 (3)	O3—C15—C19—O5	117.6 (3)
C5—C6—C7—C13	−72.0 (5)	O4—C15—C19—C20	−116.9 (3)
C1—C6—C7—C13	105.9 (4)	O3—C15—C19—C20	1.2 (4)
C11—N1—C8—S9	−38.5 (3)	C21—O6—C20—C19	−110.0 (3)
C7—N1—C8—S9	78.8 (3)	C21—O6—C20—C14	139.3 (3)
C10—S9—C8—N1	16.8 (3)	O5—C19—C20—O6	156.1 (3)
C8—S9—C10—C11	8.6 (3)	C15—C19—C20—O6	−92.2 (3)
C8—N1—C11—C12	170.2 (3)	O5—C19—C20—C14	−87.7 (4)
C7—N1—C11—C12	47.5 (3)	C15—C19—C20—C14	24.1 (4)

C8—N1—C11—C10	47.0 (4)	O3—C14—C20—O6	75.0 (3)
C7—N1—C11—C10	−75.7 (4)	C13—C14—C20—O6	−45.2 (4)
S9—C10—C11—N1	−32.5 (4)	O3—C14—C20—C19	−40.6 (3)
S9—C10—C11—C12	−144.4 (3)	C13—C14—C20—C19	−160.8 (3)
O2—N2—C12—C11	83.3 (5)	C20—O6—C21—C22	−73.2 (4)
O1—N2—C12—C11	−92.5 (4)	N4—N3—C22—C23	0.1 (4)
O2—N2—C12—C13	−36.8 (5)	N4—N3—C22—C21	173.3 (3)
O1—N2—C12—C13	147.5 (4)	O6—C21—C22—N3	−69.4 (5)
N1—C11—C12—N2	−165.7 (3)	O6—C21—C22—C23	102.1 (5)
C10—C11—C12—N2	−48.5 (5)	N4—N5—C23—C22	1.2 (4)
N1—C11—C12—C13	−41.0 (3)	C24—N5—C23—C22	168.4 (4)
C10—C11—C12—C13	76.2 (4)	N3—C22—C23—N5	−0.8 (4)
N2—C12—C13—C14	−88.8 (4)	C21—C22—C23—N5	−173.1 (4)
C11—C12—C13—C14	147.1 (3)	C23—N5—C24—C25	−49.6 (5)
N2—C12—C13—C7	144.0 (3)	N4—N5—C24—C25	116.7 (4)
C11—C12—C13—C7	19.9 (4)	C1—O7—C25—C24	159.4 (3)
N1—C7—C13—C14	−116.0 (3)	N5—C24—C25—O7	−55.0 (4)
C6—C7—C13—C14	7.1 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C23—H23···N3^{i}	0.93	2.58	3.433 (6)	152
C25—H25*A*···N3^{i}	0.97	2.60	3.553 (6)	168
C25—H25*B*···S9^{ii}	0.97	2.80	3.591 (4)	140

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