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Bis(1,2,3,5-tetramethyl-4-nitropyrazolium) dihydronium tris(4-methyl-3,5-dinitrobenzenesulfonate) forms *C*(8) chains through O—H···O bonding *via* the dihydronium cation

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.145
Data-to-parameter ratio = 15.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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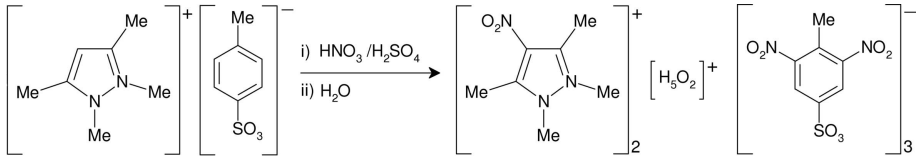
In the title salt, $2C_7H_{12}N_3O_2^+\cdot H_5O_2^+\cdot 3C_7H_5N_2O_7S^-$, strong hydrogen bonding from the donor ($H_5O_2^+$) group links to 4-methyl-3,5-dinitrobenzenesulfonate anions. The dihydronium ($H_5O_2^+$) cation could be considered intermediate between clearly distinct $(H_3O)^+$ and H_2O entities and an '$(H_5O_2)^+$' entity, although it tends to be closer to the former; the O···O distance is 2.434 (3) Å. Strong hydrogen bonding leads to the formation of chains along the [010] direction.

Comment

Reaction of 1,2,3,5-tetramethylpyrazolium 4-toluenesulfonate with fuming nitric acid and concentrated sulfuric acid leads to nitration of both the cation and the anion. The salt isolated after quenching in ice and recrystallization from ethanol was found to be the title salt, (I).



(I)

Two previous reports give support to this nitration reaction. A kinetic study has indicated that the cation of 1,2,3,5-tetramethylpyrazolium bisulfate undergoes nitration to give the 1,2,3,5-tetramethyl-4-nitropyrazolium cation in a mixture of fuming nitric acid and concentrated sulfuric acid (Burton *et al.*, 1971). In another study, 4-toluenesulfonyl chloride was reported to undergo nitration to give 4-methyl-3,5-dinitrobenzenesulfonic acid in a similar medium (Schmidt *et al.*, 1999).

The existence of the dihydronium ($H_5O_2^+$) unit was confirmed by the refinement. Initially, two O atoms (O1 and O2) were treated as water molecules and the associated H atoms were allowed to refine freely. On consideration of charge balance and the hydrogen-bonding scheme, and on examination of difference maps (visually using *PLATON*; Spek, 2003), it was believed likely that an additional H atom was located between the two water molecules [see, for example, Wells (1984) and Bernal & Fowler (1933)] but slightly closer to O2. Thus, an H atom was placed in this position and also refined freely, resulting in a position that was closer to O2, at a distance of 1.01 (5) Å. The group could be considered intermediate between clearly distinct $(H_3O)^+$ and (H_2O) entities and an '$(H_5O_2)^+$' entity, although it tends to be closer to the former. The O1···O2 distance is 2.434 (3) Å, with

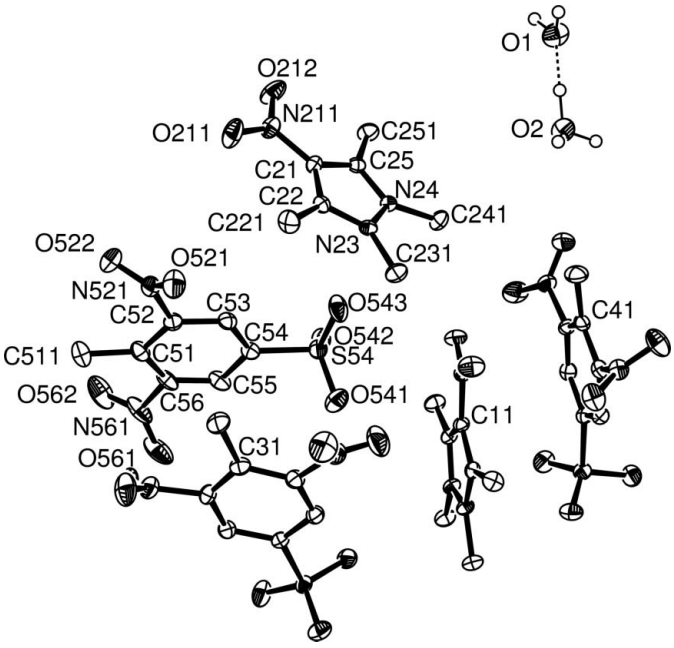


Figure 1
The asymmetric unit of the title compound, showing part of the atom-labelling scheme and indicating how it continues for similar ions within the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity, except those in the $(H_5O_2)^+$ unit, which are shown as circles of arbitrary radii.

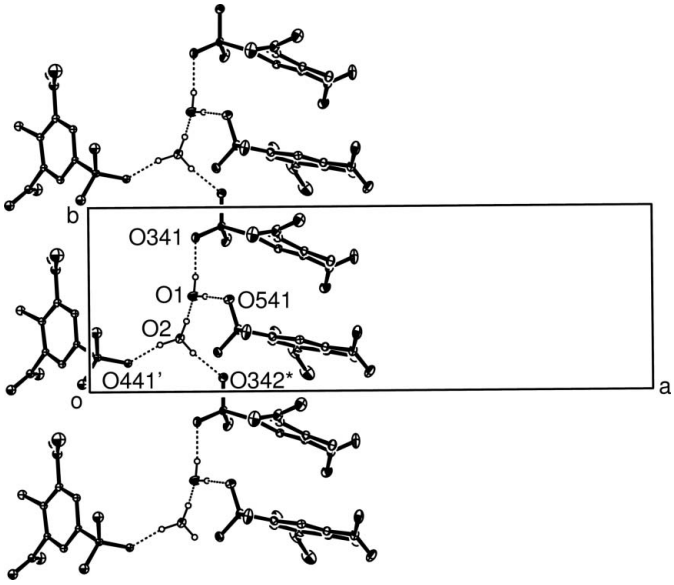


Figure 2
Part of the crystal structure of the title compound, showing the formation of hydrogen-bonded $C(8)$ chains along [010]. Atoms marked with an asterisk (*) or a prime (′) are at the symmetry positions $(-x, 1-y, -z)$ and $(x, y-1, z)$, respectively. The two 1,2,3,5-tetramethyl-4-nitropyrazolium cations have been omitted for clarity, as have all H atoms, except those of the $(H_5O_2)^+$ unit, which are shown as circles of arbitrary radii. Displacement ellipsoids are drawn at the 30% probability level.

an O—H···O angle of 174 (5)°; this distance is significantly shorter than for other O—H···O bonds within the structure (Table 1), again supporting the above interpretation.

Strong hydrogen bonds (Table 1) from the $H_2O\cdots H_3O^+$ unit link to sulfonate O atoms. Atom O1 hydrogen bonds within the asymmetric unit, whereas O2 not only hydrogen bonds to O1, but also to symmetry-related ions. This gives rise to $C(8)$ chains (Bernstein *et al.*, 1995) along the [010] direction (Fig. 2), involving the three 4-methyl-3,5-dinitrobenzenesulfonate groups and the $H_2O\cdots H_3O^+$ group.

Experimental

1,2,3,5-Tetramethylpyrazolium 4-toluenesulfonate was prepared from 1,3,5-trimethylpyrazine and methyl 4-toluenesulfonate following a similar procedure as used for 1,2,3,5-trimethyl-4-nitropyrazolium 4-toluenesulfonate (Burton, *et al.*, 1971). Nitration of 1,2,3,5-tetramethylpyrazolium 4-toluenesulfonate was carried out under similar conditions used for 4-toluenesulfonyl chloride (Schmidt *et al.*, 1999). The title compound was obtained by recrystallization from EtOH of the product isolated on careful addition of the reaction mixture to ice (m.p. 330–332 K).

Crystal data

$2C_7H_{12}N_3O_2^+\cdot H_5O_2^+\cdot 3C_7H_5N_2O_7S^-$
$M_r = 1161.00$
Monoclinic, $P2_1/c$
$a = 26.0927$ (5) Å
$b = 8.17230$ (10) Å
$c = 24.0602$ (6) Å
$\beta = 106.7241$ (8)°
$V = 4913.52$ (17) Å^3
$Z = 4$
$D_x = 1.569$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10634 reflections
$\theta = 2.9$–27.5°
$\mu = 0.26$ mm^{-1}
$T = 120$ (2) K
Slab, colourless
0.45 × 0.20 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.834$, $T_{max} = 0.990$
53726 measured reflections
11088 independent reflections
8214 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 27.5°$
$h = -33 \rightarrow 33$
$k = -10 \rightarrow 10$
$l = -29 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.145$
$S = 1.06$
11088 reflections
732 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2 + 0.908P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0067 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2C···O1	1.02 (5)	1.42 (5)	2.433 (3)	176 (4)
O1—H1A···O541	0.83 (4)	1.85 (4)	2.681 (3)	176 (4)
O1—H1B···O341	0.90 (3)	1.79 (4)	2.690 (2)	173 (3)
O2—H2A···O441^{i}	0.95 (3)	1.64 (4)	2.570 (2)	164 (3)
O2—H2B···O342^{ii}	0.87 (3)	1.73 (3)	2.590 (2)	170 (3)
C43—H43···O462^{iii}	0.95	2.46	3.401 (3)	172
C55—H55···O342^{ii}	0.95	2.55	3.212 (3)	127

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

H atoms were located in difference maps and, except for those in the H_5O_2 group, were then treated as riding atoms, with C—H distances of 0.95 (aromatic) or 0.96 Å (methyl) and U_{iso}(H) values of $1.2U_{eq}$(aromatic C) or $1.5U_{eq}$(methyl C). The existence and location of the additional H atom in the H_5O_2 group was demonstrated from a difference map (see *Comment*); all H atoms of this group were allowed to refine freely.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

We are indebted to the EPSRC for the use of both the Chemical Database Service at Daresbury, England (Fletcher *et al.*, 1996), primarily for access to the Cambridge Structural Database, and the X-ray service at the University of Southampton, England, for data collection. We thank CNPq, Brazil, for financial support.

References

Bernal, J. D. & Fowler, R. H. (1933). *J. Chem. Phys.* **1**, 515–548.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Burton, A. G., Forsythe, P. P., Johnson, C. D. & Katritzky, A. R. (1971). *J. Chem. Soc. B*, pp. 2365–2371.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565–565.
Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
McArdle, P. (2003). *OSCAIL for Windows*. Version 10. Crystallography Centre, Chemistry Department, National University of Ireland, Galway, Ireland.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Schmidt, T. C., Steinbach, K., Buetehorn, U., Heck, K., Volkwein, U. & Stork, G. (1999). *Chemosphere*, **38**, 3119–3130.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wells, A. F. (1984). *Structural Inorganic Chemistry*, 5th ed. Oxford University Press.

supporting information

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Bis[1,2,3,5-tetramethyl-4-nitropyrazolium] dihydronium tris(4-methyl-3,5-dinitrobenzenesulfonate)

Crystal data

$2C_7H_{12}N_3O_2^+\cdot H_5O_2^+\cdot 3C_7H_5N_2O_7S^-$
$M_r = 1161.00$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 26.0927\ (5)$ Å
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$c = 24.0602\ (6)$ Å
$\beta = 106.7241\ (8)°$
$V = 4913.52\ (17)$ Å^3
$Z = 4$
$F(000) = 2408$
$D_x = 1.569$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10634 reflections
$\theta = 2.9–27.5°$
$\mu = 0.26$ mm^{-1}
$T = 120$ K
Slab, colourless
0.45 × 0.20 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker–Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.834$, $T_{max} = 0.990$
53726 measured reflections
11088 independent reflections
8214 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -33\rightarrow33$
$k = -10\rightarrow10$
$l = -29\rightarrow31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.145$
$S = 1.06$
11088 reflections
732 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2 + 0.908P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0067 (5)

Special details

Experimental. IR: 3408 (*br*), 3080–2932, 1562, 1543, 1482, 1411, 1372, 1359, 1240, 1195, 1052, 1017, 991, 865, 814, 767, 722, 665, 594.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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 $F^2 > \sigma(F^2)$ 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.
All H atoms were located from difference maps and then treated as riding atoms, with C—H distances of 0.96 Å (methyl) or 0.93 Å (aromatic) and U_{iso}(H) values of -1.5U_{eq} (methyl) or -1.2U_{eq} (aromatic).
The exceptions were the H atoms for the two free O atoms. Initially these were treated as water molecules and the H atoms were allowed to refine freely. On consideration of charge balance and the hydrogen bonding scheme, and on examination of difference maps, visually using *PLATON* (Spek, 2003), it was believed likely that an additional H atom was located between the two water molecules, (see *e.g.* Wells, 1984 and Bernal & Fowler, 1933) but slightly closer to O2. Thus an H atom was placed at this position and also refined freely, resulting in a position that was closer to O2 but at a distance of 1.01 (5) Å. The moiety could be considered intermediate between clearly distinct $(H_3O)^+$ and (H_2O) entities and a $'(H_5O_2)^+'$ entity, although tends to be closer to the former.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C11	0.14584 (8)	0.6885 (2)	0.13331 (9)	0.0170 (4)
C12	0.14849 (8)	0.8567 (2)	0.12480 (9)	0.0172 (4)
N13	0.12285 (7)	0.88118 (19)	0.06894 (8)	0.0181 (4)
N14	0.10311 (6)	0.73665 (19)	0.04274 (7)	0.0176 (4)
C15	0.11657 (8)	0.6150 (2)	0.08144 (9)	0.0181 (4)
N111	0.16869 (7)	0.6022 (2)	0.18640 (8)	0.0201 (4)
O111	0.19612 (6)	0.67898 (19)	0.22839 (7)	0.0318 (4)
O112	0.15918 (6)	0.45560 (18)	0.18771 (7)	0.0269 (4)
C121	0.16984 (9)	0.9942 (3)	0.16537 (10)	0.0244 (5)
H12A	0.2014	1.0406	0.1568	0.037*
H12B	0.1799	0.9536	0.2054	0.037*
H12C	0.1423	1.0788	0.1606	0.037*
C131	0.11262 (9)	1.0357 (2)	0.03717 (10)	0.0230 (5)
H13A	0.0777	1.0786	0.0377	0.035*
H13B	0.1127	1.0175	−0.0031	0.035*
H13C	0.1406	1.1148	0.0556	0.035*
C141	0.07062 (9)	0.7327 (3)	−0.01811 (9)	0.0241 (5)
H14A	0.0933	0.7561	−0.0433	0.036*
H14B	0.0423	0.8152	−0.0244	0.036*
H14C	0.0545	0.6240	−0.0273	0.036*
C151	0.10304 (9)	0.4415 (2)	0.06744 (10)	0.0250 (5)
H15A	0.0824	0.4318	0.0265	0.038*
H15B	0.0817	0.4003	0.0919	0.038*
H15C	0.1360	0.3774	0.0746	0.038*
C21	0.36486 (8)	0.0935 (2)	0.32727 (9)	0.0181 (4)
C22	0.37418 (8)	0.2611 (2)	0.32547 (9)	0.0176 (4)

N23	0.32645 (6)	0.33193 (19)	0.31675 (7)	0.0167 (4)
N24	0.28810 (6)	0.21495 (19)	0.31325 (7)	0.0155 (3)
C25	0.31055 (8)	0.0670 (2)	0.31977 (9)	0.0178 (4)
N211	0.40486 (7)	−0.0321 (2)	0.33769 (8)	0.0248 (4)
O211	0.45073 (7)	0.0073 (2)	0.33971 (9)	0.0413 (5)
O212	0.39154 (7)	−0.17196 (19)	0.34463 (9)	0.0388 (5)
C221	0.42372 (9)	0.3577 (3)	0.33218 (11)	0.0278 (5)
H22A	0.4189	0.4311	0.2989	0.042*
H22B	0.4537	0.2834	0.3342	0.042*
H22C	0.4313	0.4225	0.3679	0.042*
C231	0.31238 (9)	0.5053 (2)	0.31111 (11)	0.0253 (5)
H23A	0.2867	0.5251	0.2731	0.050 (8)*
H23B	0.3447	0.5706	0.3147	0.039 (7)*
H23C	0.2964	0.5365	0.3417	0.030 (7)*
C241	0.23266 (8)	0.2629 (3)	0.30653 (10)	0.0241 (5)
H24A	0.2114	0.1654	0.3086	0.036*
H24B	0.2180	0.3165	0.2689	0.036*
H24C	0.2315	0.3388	0.3376	0.036*
C251	0.27972 (9)	−0.0856 (2)	0.31932 (10)	0.0248 (5)
H25A	0.2834	−0.1570	0.2880	0.062 (10)*
H25B	0.2419	−0.0587	0.3131	0.058 (9)*
H25C	0.2935	−0.1419	0.3566	0.050 (8)*
C31	0.38424 (8)	0.7753 (2)	0.10624 (9)	0.0218 (5)
C32	0.33774 (9)	0.8480 (3)	0.11359 (9)	0.0220 (4)
C33	0.29317 (9)	0.8897 (2)	0.06873 (9)	0.0220 (5)
H33	0.2627	0.9368	0.0765	0.026*
C34	0.29387 (8)	0.8613 (2)	0.01216 (9)	0.0187 (4)
C35	0.33820 (8)	0.7894 (2)	0.00160 (9)	0.0204 (4)
H35	0.3385	0.7665	−0.0370	0.024*
C36	0.38201 (8)	0.7512 (3)	0.04799 (9)	0.0210 (4)
C311	0.43059 (9)	0.7198 (3)	0.15595 (10)	0.0297 (5)
H31A	0.4496	0.6309	0.1429	0.045*
H31B	0.4172	0.6807	0.1877	0.045*
H31C	0.4551	0.8116	0.1696	0.045*
N321	0.33497 (8)	0.8851 (2)	0.17263 (8)	0.0306 (5)
O321	0.37542 (8)	0.9403 (2)	0.20756 (8)	0.0458 (5)
O322	0.29261 (8)	0.8644 (3)	0.18257 (8)	0.0502 (5)
S34	0.23781 (2)	0.91534 (6)	−0.04691 (2)	0.01945 (14)
O341	0.19139 (6)	0.85398 (19)	−0.03153 (7)	0.0278 (4)
O342	0.23841 (6)	1.09346 (18)	−0.04892 (7)	0.0297 (4)
O343	0.24647 (7)	0.8403 (2)	−0.09750 (7)	0.0354 (4)
N361	0.42865 (7)	0.6844 (2)	0.03287 (9)	0.0280 (4)
O361	0.47244 (7)	0.7431 (3)	0.05701 (8)	0.0458 (5)
O362	0.42053 (7)	0.5792 (2)	−0.00455 (9)	0.0432 (5)
C41	0.08216 (8)	0.5906 (3)	0.26649 (9)	0.0206 (4)
C42	0.07623 (8)	0.7576 (3)	0.25463 (9)	0.0192 (4)
C43	0.04739 (8)	0.8248 (2)	0.20219 (9)	0.0185 (4)
H43	0.0458	0.9399	0.1964	0.022*

C44	0.02081 (8)	0.7186 (2)	0.15830 (9)	0.0180 (4)
C45	0.02397 (8)	0.5517 (2)	0.16761 (9)	0.0204 (4)
H45	0.0055	0.4787	0.1378	0.025*
C46	0.05424 (8)	0.4915 (2)	0.22064 (10)	0.0203 (4)
C411	0.11985 (9)	0.5297 (3)	0.32259 (10)	0.0306 (5)
H41A	0.1005	0.5192	0.3518	0.046*
H41B	0.1494	0.6075	0.3363	0.046*
H41C	0.1342	0.4228	0.3162	0.046*
N421	0.10357 (7)	0.8734 (2)	0.30082 (8)	0.0238 (4)
O421	0.08983 (7)	0.8740 (2)	0.34507 (8)	0.0398 (4)
O422	0.13809 (6)	0.9613 (2)	0.29166 (8)	0.0337 (4)
S44	−0.01569 (2)	0.79219 (6)	0.08862 (2)	0.02083 (14)
O441	−0.06957 (6)	0.8249 (2)	0.09217 (7)	0.0317 (4)
O442	−0.01406 (7)	0.65911 (19)	0.04948 (7)	0.0318 (4)
O443	0.01169 (6)	0.93806 (18)	0.07960 (7)	0.0293 (4)
N461	0.05724 (7)	0.3114 (2)	0.22521 (9)	0.0270 (4)
O461	0.06066 (8)	0.2483 (2)	0.27143 (8)	0.0404 (4)
O462	0.05452 (8)	0.23438 (19)	0.18053 (8)	0.0407 (5)
C51	0.42046 (9)	0.2329 (3)	0.07659 (10)	0.0246 (5)
C52	0.41963 (8)	0.2537 (3)	0.13375 (9)	0.0212 (4)
C53	0.37436 (8)	0.2921 (2)	0.15039 (9)	0.0206 (4)
H53	0.3766	0.3047	0.1903	0.025*
C54	0.32621 (8)	0.3118 (2)	0.10830 (9)	0.0208 (4)
C55	0.32425 (9)	0.2940 (3)	0.05017 (9)	0.0259 (5)
H55	0.2915	0.3089	0.0205	0.031*
C56	0.37033 (9)	0.2547 (3)	0.03611 (9)	0.0264 (5)
C511	0.47115 (10)	0.2021 (3)	0.05949 (11)	0.0337 (6)
H51A	0.5017	0.2504	0.0886	0.051*
H51B	0.4678	0.2520	0.0216	0.051*
H51C	0.4767	0.0839	0.0572	0.051*
N521	0.46878 (7)	0.2319 (2)	0.18215 (9)	0.0281 (4)
O521	0.47639 (6)	0.3283 (2)	0.22266 (7)	0.0389 (4)
O522	0.49822 (7)	0.1174 (2)	0.17959 (9)	0.0418 (5)
S54	0.26573 (2)	0.35056 (7)	0.12659 (2)	0.02321 (14)
O541	0.24970 (7)	0.51473 (19)	0.10493 (8)	0.0365 (4)
O542	0.22889 (6)	0.22807 (19)	0.09453 (7)	0.0293 (4)
O543	0.27967 (7)	0.3350 (2)	0.18870 (7)	0.0410 (5)
N561	0.36349 (9)	0.2307 (3)	−0.02629 (9)	0.0391 (6)
O561	0.33258 (8)	0.3247 (3)	−0.05961 (8)	0.0515 (6)
O562	0.38745 (9)	0.1174 (3)	−0.04106 (9)	0.0545 (6)
O1	0.18732 (8)	0.5355 (2)	−0.00492 (9)	0.0378 (4)
H1A	0.2077 (15)	0.533 (4)	0.0286 (17)	0.067 (12)*
H1B	0.1906 (12)	0.640 (4)	−0.0153 (14)	0.055 (9)*
O2	0.16226 (7)	0.3084 (2)	−0.07275 (8)	0.0326 (4)
H2A	0.1267 (14)	0.274 (4)	−0.0756 (14)	0.058 (9)*
H2B	0.1850 (12)	0.229 (4)	−0.0640 (13)	0.045 (8)*
H2C	0.1741 (16)	0.401 (5)	−0.0433 (19)	0.089 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0159 (10)	0.0177 (10)	0.0160 (10)	0.0021 (7)	0.0025 (8)	0.0016 (8)
C12	0.0151 (10)	0.0181 (10)	0.0180 (10)	0.0007 (7)	0.0041 (8)	−0.0013 (8)
N13	0.0186 (9)	0.0135 (8)	0.0202 (9)	0.0002 (6)	0.0026 (7)	−0.0009 (7)
N14	0.0180 (8)	0.0154 (8)	0.0166 (9)	0.0011 (6)	0.0004 (7)	−0.0011 (7)
C15	0.0162 (10)	0.0169 (10)	0.0194 (10)	0.0016 (7)	0.0025 (8)	0.0000 (8)
N111	0.0153 (9)	0.0238 (10)	0.0198 (9)	0.0020 (7)	0.0027 (7)	0.0032 (7)
O111	0.0335 (9)	0.0332 (9)	0.0210 (8)	−0.0032 (7)	−0.0046 (7)	−0.0003 (7)
O112	0.0297 (8)	0.0209 (8)	0.0279 (9)	0.0013 (6)	0.0048 (7)	0.0074 (6)
C121	0.0279 (12)	0.0237 (11)	0.0202 (11)	−0.0050 (9)	0.0046 (9)	−0.0040 (9)
C131	0.0251 (11)	0.0161 (10)	0.0250 (11)	0.0012 (8)	0.0027 (9)	0.0042 (8)
C141	0.0245 (11)	0.0233 (11)	0.0190 (11)	0.0013 (8)	−0.0027 (9)	−0.0007 (9)
C151	0.0305 (12)	0.0175 (10)	0.0238 (12)	0.0003 (9)	0.0026 (9)	−0.0025 (9)
C21	0.0169 (10)	0.0186 (10)	0.0181 (10)	0.0018 (7)	0.0037 (8)	−0.0013 (8)
C22	0.0186 (10)	0.0200 (10)	0.0139 (10)	−0.0018 (8)	0.0043 (8)	−0.0020 (8)
N23	0.0188 (9)	0.0151 (8)	0.0153 (8)	−0.0024 (6)	0.0032 (7)	0.0009 (6)
N24	0.0153 (8)	0.0145 (8)	0.0155 (8)	−0.0022 (6)	0.0027 (6)	−0.0012 (6)
C25	0.0202 (10)	0.0183 (10)	0.0135 (10)	−0.0004 (8)	0.0028 (8)	0.0006 (8)
N211	0.0222 (10)	0.0233 (10)	0.0267 (10)	0.0030 (7)	0.0033 (8)	−0.0048 (8)
O211	0.0200 (9)	0.0370 (10)	0.0666 (14)	0.0035 (7)	0.0120 (8)	−0.0036 (9)
O212	0.0325 (9)	0.0179 (8)	0.0616 (13)	0.0045 (7)	0.0065 (8)	0.0037 (8)
C221	0.0225 (11)	0.0269 (12)	0.0339 (13)	−0.0080 (9)	0.0083 (10)	−0.0023 (10)
C231	0.0303 (12)	0.0137 (10)	0.0312 (13)	−0.0002 (8)	0.0075 (10)	−0.0007 (9)
C241	0.0171 (10)	0.0234 (11)	0.0298 (12)	0.0028 (8)	0.0034 (9)	0.0004 (9)
C251	0.0244 (12)	0.0171 (10)	0.0311 (13)	−0.0035 (8)	0.0050 (9)	0.0022 (9)
C31	0.0202 (11)	0.0213 (11)	0.0205 (11)	−0.0009 (8)	0.0004 (8)	0.0012 (8)
C32	0.0257 (11)	0.0231 (11)	0.0166 (11)	0.0019 (8)	0.0051 (8)	−0.0009 (8)
C33	0.0231 (11)	0.0231 (11)	0.0196 (11)	0.0031 (8)	0.0061 (9)	0.0000 (8)
C34	0.0179 (10)	0.0173 (10)	0.0195 (11)	0.0011 (8)	0.0029 (8)	0.0008 (8)
C35	0.0220 (11)	0.0208 (10)	0.0181 (11)	−0.0004 (8)	0.0055 (8)	−0.0021 (8)
C36	0.0158 (10)	0.0223 (10)	0.0240 (11)	0.0014 (8)	0.0043 (8)	−0.0018 (9)
C311	0.0226 (12)	0.0360 (13)	0.0255 (12)	0.0044 (9)	−0.0011 (9)	0.0036 (10)
N321	0.0319 (11)	0.0387 (11)	0.0200 (10)	0.0074 (9)	0.0054 (9)	0.0010 (8)
O321	0.0452 (11)	0.0594 (12)	0.0259 (10)	0.0059 (9)	−0.0011 (8)	−0.0111 (9)
O322	0.0471 (12)	0.0781 (14)	0.0307 (11)	0.0063 (10)	0.0198 (9)	−0.0012 (10)
S34	0.0177 (3)	0.0218 (3)	0.0172 (3)	0.00331 (19)	0.0023 (2)	−0.0008 (2)
O341	0.0193 (8)	0.0300 (8)	0.0324 (9)	0.0011 (6)	0.0046 (7)	0.0048 (7)
O342	0.0256 (8)	0.0246 (8)	0.0337 (9)	0.0026 (6)	0.0001 (7)	0.0036 (7)
O343	0.0307 (9)	0.0531 (11)	0.0192 (8)	0.0142 (8)	0.0020 (7)	−0.0080 (8)
N361	0.0213 (10)	0.0313 (10)	0.0304 (11)	0.0046 (8)	0.0058 (8)	−0.0003 (9)
O361	0.0196 (9)	0.0719 (13)	0.0440 (11)	−0.0008 (8)	0.0060 (8)	−0.0086 (10)
O362	0.0377 (10)	0.0424 (11)	0.0527 (12)	0.0071 (8)	0.0179 (9)	−0.0135 (9)
C41	0.0168 (10)	0.0263 (11)	0.0204 (11)	0.0024 (8)	0.0080 (8)	0.0058 (9)
C42	0.0149 (10)	0.0238 (11)	0.0186 (10)	−0.0033 (8)	0.0045 (8)	−0.0019 (8)
C43	0.0167 (10)	0.0169 (10)	0.0213 (11)	−0.0013 (7)	0.0044 (8)	−0.0007 (8)
C44	0.0139 (10)	0.0202 (10)	0.0184 (10)	0.0003 (7)	0.0021 (8)	0.0009 (8)

C45	0.0175 (10)	0.0207 (10)	0.0232 (11)	−0.0030 (8)	0.0059 (8)	−0.0023 (9)
C46	0.0190 (10)	0.0153 (10)	0.0277 (12)	0.0011 (8)	0.0083 (9)	0.0040 (8)
C411	0.0262 (12)	0.0351 (13)	0.0270 (13)	0.0034 (10)	0.0019 (10)	0.0113 (10)
N421	0.0203 (9)	0.0278 (10)	0.0211 (10)	−0.0014 (7)	0.0022 (7)	−0.0020 (8)
O421	0.0465 (11)	0.0525 (11)	0.0231 (9)	−0.0110 (8)	0.0145 (8)	−0.0095 (8)
O422	0.0290 (9)	0.0377 (9)	0.0349 (10)	−0.0134 (7)	0.0102 (7)	−0.0108 (8)
S44	0.0176 (3)	0.0246 (3)	0.0172 (3)	0.00078 (19)	0.0000 (2)	0.0009 (2)
O441	0.0185 (8)	0.0482 (10)	0.0260 (9)	0.0066 (7)	0.0024 (6)	0.0056 (7)
O442	0.0358 (9)	0.0337 (9)	0.0218 (8)	0.0033 (7)	0.0017 (7)	−0.0073 (7)
O443	0.0299 (9)	0.0286 (8)	0.0260 (9)	−0.0017 (7)	0.0029 (7)	0.0076 (7)
N461	0.0292 (10)	0.0193 (9)	0.0352 (12)	0.0034 (7)	0.0134 (9)	0.0057 (9)
O461	0.0551 (12)	0.0277 (9)	0.0453 (11)	0.0074 (8)	0.0253 (9)	0.0128 (8)
O462	0.0608 (12)	0.0204 (8)	0.0401 (11)	−0.0018 (8)	0.0133 (9)	−0.0040 (8)
C51	0.0283 (12)	0.0231 (11)	0.0251 (12)	−0.0057 (9)	0.0119 (9)	−0.0028 (9)
C52	0.0210 (10)	0.0220 (10)	0.0194 (11)	0.0004 (8)	0.0041 (8)	0.0006 (8)
C53	0.0257 (11)	0.0215 (10)	0.0145 (10)	0.0002 (8)	0.0055 (8)	−0.0011 (8)
C54	0.0208 (11)	0.0221 (10)	0.0188 (11)	−0.0026 (8)	0.0044 (8)	0.0006 (8)
C55	0.0250 (11)	0.0347 (12)	0.0155 (11)	−0.0097 (9)	0.0019 (9)	−0.0004 (9)
C56	0.0331 (13)	0.0332 (12)	0.0147 (10)	−0.0127 (10)	0.0095 (9)	−0.0040 (9)
C511	0.0320 (13)	0.0405 (14)	0.0339 (14)	−0.0052 (10)	0.0180 (11)	−0.0063 (11)
N521	0.0223 (10)	0.0365 (11)	0.0263 (11)	0.0052 (8)	0.0082 (8)	0.0050 (9)
O521	0.0275 (9)	0.0619 (12)	0.0231 (9)	0.0058 (8)	0.0003 (7)	−0.0076 (8)
O522	0.0337 (10)	0.0433 (10)	0.0493 (12)	0.0172 (8)	0.0133 (9)	0.0091 (9)
S54	0.0204 (3)	0.0285 (3)	0.0194 (3)	0.0016 (2)	0.0036 (2)	−0.0006 (2)
O541	0.0336 (9)	0.0247 (9)	0.0492 (11)	0.0037 (7)	0.0086 (8)	−0.0025 (8)
O542	0.0223 (8)	0.0295 (9)	0.0367 (10)	−0.0041 (6)	0.0093 (7)	−0.0040 (7)
O543	0.0286 (9)	0.0758 (13)	0.0193 (9)	0.0104 (9)	0.0078 (7)	−0.0009 (9)
N561	0.0392 (13)	0.0603 (15)	0.0200 (11)	−0.0264 (11)	0.0118 (10)	−0.0077 (11)
O561	0.0457 (12)	0.0890 (16)	0.0174 (9)	−0.0221 (11)	0.0050 (8)	0.0056 (10)
O562	0.0687 (14)	0.0712 (14)	0.0332 (11)	−0.0224 (11)	0.0299 (10)	−0.0256 (10)
O1	0.0469 (12)	0.0250 (10)	0.0378 (11)	0.0014 (8)	0.0063 (9)	0.0065 (8)
O2	0.0240 (9)	0.0292 (9)	0.0431 (11)	0.0052 (7)	0.0071 (8)	0.0008 (8)

Geometric parameters (Å, º)

C11—C12	1.394 (3)	C311—H31A	0.9800
C11—C15	1.398 (3)	C311—H31B	0.9800
C11—N111	1.430 (3)	C311—H31C	0.9800
C12—N13	1.333 (3)	N321—O322	1.208 (3)
C12—C121	1.487 (3)	N321—O321	1.230 (3)
N13—N14	1.368 (2)	S34—O343	1.4375 (17)
N13—C131	1.461 (3)	S34—O341	1.4536 (16)
N14—C15	1.338 (3)	S34—O342	1.4567 (16)
N14—C141	1.466 (3)	N361—O362	1.219 (3)
C15—C151	1.476 (3)	N361—O361	1.221 (3)
N111—O112	1.226 (2)	C41—C46	1.393 (3)
N111—O111	1.229 (2)	C41—C42	1.393 (3)
C121—H12A	0.9800	C41—C411	1.507 (3)

C121—H12B	0.9800	C42—C43	1.383 (3)
C121—H12C	0.9800	C42—N421	1.478 (3)
C131—H13A	0.9800	C43—C44	1.389 (3)
C131—H13B	0.9800	C43—H43	0.9500
C131—H13C	0.9800	C44—C45	1.381 (3)
C141—H14A	0.9800	C44—S44	1.778 (2)
C141—H14B	0.9800	C45—C46	1.383 (3)
C141—H14C	0.9800	C45—H45	0.9500
C151—H15A	0.9800	C46—N461	1.476 (3)
C151—H15B	0.9800	C411—H41A	0.9800
C151—H15C	0.9800	C411—H41B	0.9800
C21—C25	1.393 (3)	C411—H41C	0.9800
C21—C22	1.394 (3)	N421—O421	1.217 (2)
C21—N211	1.434 (3)	N421—O422	1.221 (2)
C22—N23	1.334 (3)	S44—O443	1.4379 (16)
C22—C221	1.483 (3)	S44—O442	1.4472 (17)
N23—N24	1.369 (2)	S44—O441	1.4577 (16)
N23—C231	1.460 (3)	N461—O461	1.206 (3)
N24—C25	1.333 (3)	N461—O462	1.230 (3)
N24—C241	1.462 (3)	C51—C52	1.392 (3)
C25—C251	1.483 (3)	C51—C56	1.399 (3)
N211—O212	1.220 (2)	C51—C511	1.515 (3)
N211—O211	1.227 (2)	C52—C53	1.388 (3)
C221—H22A	0.9800	C52—N521	1.474 (3)
C221—H22B	0.9800	C53—C54	1.378 (3)
C221—H22C	0.9800	C53—H53	0.9500
C231—H23A	0.9800	C54—C55	1.392 (3)
C231—H23B	0.9800	C54—S54	1.784 (2)
C231—H23C	0.9800	C55—C56	1.377 (3)
C241—H24A	0.9800	C55—H55	0.9500
C241—H24B	0.9800	C56—N561	1.473 (3)
C241—H24C	0.9800	C511—H51A	0.9800
C251—H25A	0.9800	C511—H51B	0.9800
C251—H25B	0.9800	C511—H51C	0.9800
C251—H25C	0.9800	N521—O522	1.223 (2)
C31—C36	1.400 (3)	N521—O521	1.224 (3)
C31—C32	1.407 (3)	S54—O543	1.4385 (18)
C31—C311	1.506 (3)	S54—O542	1.4472 (16)
C32—C33	1.382 (3)	S54—O541	1.4564 (17)
C32—N321	1.475 (3)	N561—O562	1.225 (3)
C33—C34	1.386 (3)	N561—O561	1.229 (3)
C33—H33	0.9500	O1—H1A	0.83 (4)
C34—C35	1.385 (3)	O1—H1B	0.90 (3)
C34—S34	1.777 (2)	O1—H2C	1.42 (5)
C35—C36	1.384 (3)	O2—H2A	0.95 (3)
C35—H35	0.9500	O2—H2B	0.87 (3)
C36—N361	1.472 (3)	O2—H2C	1.02 (5)

C12—C11—C15	109.28 (18)	C35—C36—C31	124.23 (19)
C12—C11—N111	126.27 (18)	C35—C36—N361	115.73 (19)
C15—C11—N111	124.45 (18)	C31—C36—N361	120.03 (18)
N13—C12—C11	105.33 (17)	C31—C311—H31A	109.5
N13—C12—C121	122.20 (18)	C31—C311—H31B	109.5
C11—C12—C121	132.31 (19)	H31A—C311—H31B	109.5
C12—N13—N14	110.38 (16)	C31—C311—H31C	109.5
C12—N13—C131	128.37 (17)	H31A—C311—H31C	109.5
N14—N13—C131	121.18 (16)	H31B—C311—H31C	109.5
C15—N14—N13	109.41 (16)	O322—N321—O321	124.7 (2)
C15—N14—C141	129.72 (17)	O322—N321—C32	117.60 (19)
N13—N14—C141	120.81 (16)	O321—N321—C32	117.6 (2)
N14—C15—C11	105.57 (17)	O343—S34—O341	114.45 (10)
N14—C15—C151	123.86 (18)	O343—S34—O342	113.10 (11)
C11—C15—C151	130.54 (19)	O341—S34—O342	111.77 (9)
O112—N111—O111	123.51 (18)	O343—S34—C34	106.10 (10)
O112—N111—C11	117.99 (17)	O341—S34—C34	105.36 (10)
O111—N111—C11	118.49 (17)	O342—S34—C34	105.11 (9)
C12—C121—H12A	109.5	O362—N361—O361	124.6 (2)
C12—C121—H12B	109.5	O362—N361—C36	117.64 (18)
H12A—C121—H12B	109.5	O361—N361—C36	117.66 (19)
C12—C121—H12C	109.5	C46—C41—C42	113.89 (18)
H12A—C121—H12C	109.5	C46—C41—C411	125.07 (19)
H12B—C121—H12C	109.5	C42—C41—C411	120.81 (19)
N13—C131—H13A	109.5	C43—C42—C41	125.11 (19)
N13—C131—H13B	109.5	C43—C42—N421	116.72 (18)
H13A—C131—H13B	109.5	C41—C42—N421	118.16 (18)
N13—C131—H13C	109.5	C42—C43—C44	117.80 (18)
H13A—C131—H13C	109.5	C42—C43—H43	121.1
H13B—C131—H13C	109.5	C44—C43—H43	121.1
N14—C141—H14A	109.5	C45—C44—C43	120.07 (19)
N14—C141—H14B	109.5	C45—C44—S44	118.56 (15)
H14A—C141—H14B	109.5	C43—C44—S44	121.33 (15)
N14—C141—H14C	109.5	C44—C45—C46	119.49 (19)
H14A—C141—H14C	109.5	C44—C45—H45	120.3
H14B—C141—H14C	109.5	C46—C45—H45	120.3
C15—C151—H15A	109.5	C45—C46—C41	123.60 (19)
C15—C151—H15B	109.5	C45—C46—N461	115.27 (19)
H15A—C151—H15B	109.5	C41—C46—N461	121.08 (19)
C15—C151—H15C	109.5	C41—C411—H41A	109.5
H15A—C151—H15C	109.5	C41—C411—H41B	109.5
H15B—C151—H15C	109.5	H41A—C411—H41B	109.5
C25—C21—C22	109.20 (17)	C41—C411—H41C	109.5
C25—C21—N211	124.98 (18)	H41A—C411—H41C	109.5
C22—C21—N211	125.77 (18)	H41B—C411—H41C	109.5
N23—C22—C21	105.53 (17)	O421—N421—O422	125.03 (19)
N23—C22—C221	122.09 (18)	O421—N421—C42	117.41 (18)
C21—C22—C221	132.37 (19)	O422—N421—C42	117.57 (18)

C22—N23—N24	109.86 (15)	O443—S44—O442	114.57 (10)
C22—N23—C231	129.34 (17)	O443—S44—O441	112.73 (10)
N24—N23—C231	120.80 (16)	O442—S44—O441	112.66 (10)
C25—N24—N23	109.72 (16)	O443—S44—C44	105.57 (9)
C25—N24—C241	130.04 (17)	O442—S44—C44	104.55 (9)
N23—N24—C241	120.15 (16)	O441—S44—C44	105.71 (9)
N24—C25—C21	105.69 (17)	O461—N461—O462	123.85 (19)
N24—C25—C251	122.90 (18)	O461—N461—C46	118.9 (2)
C21—C25—C251	131.41 (18)	O462—N461—C46	117.18 (19)
O212—N211—O211	123.63 (18)	C52—C51—C56	113.3 (2)
O212—N211—C21	118.25 (18)	C52—C51—C511	123.5 (2)
O211—N211—C21	118.12 (18)	C56—C51—C511	123.1 (2)
C22—C221—H22A	109.5	C53—C52—C51	124.68 (19)
C22—C221—H22B	109.5	C53—C52—N521	114.74 (19)
H22A—C221—H22B	109.5	C51—C52—N521	120.57 (19)
C22—C221—H22C	109.5	C54—C53—C52	119.1 (2)
H22A—C221—H22C	109.5	C54—C53—H53	120.5
H22B—C221—H22C	109.5	C52—C53—H53	120.5
N23—C231—H23A	109.5	C53—C54—C55	119.2 (2)
N23—C231—H23B	109.5	C53—C54—S54	121.58 (17)
H23A—C231—H23B	109.5	C55—C54—S54	119.14 (16)
N23—C231—H23C	109.5	C56—C55—C54	119.3 (2)
H23A—C231—H23C	109.5	C56—C55—H55	120.4
H23B—C231—H23C	109.5	C54—C55—H55	120.4
N24—C241—H24A	109.5	C55—C56—C51	124.5 (2)
N24—C241—H24B	109.5	C55—C56—N561	115.0 (2)
H24A—C241—H24B	109.5	C51—C56—N561	120.5 (2)
N24—C241—H24C	109.5	C51—C511—H51A	109.5
H24A—C241—H24C	109.5	C51—C511—H51B	109.5
H24B—C241—H24C	109.5	H51A—C511—H51B	109.5
C25—C251—H25A	109.5	C51—C511—H51C	109.5
C25—C251—H25B	109.5	H51A—C511—H51C	109.5
H25A—C251—H25B	109.5	H51B—C511—H51C	109.5
C25—C251—H25C	109.5	O522—N521—O521	124.9 (2)
H25A—C251—H25C	109.5	O522—N521—C52	117.94 (19)
H25B—C251—H25C	109.5	O521—N521—C52	117.15 (18)
C36—C31—C32	113.35 (18)	O543—S54—O542	115.03 (11)
C36—C31—C311	123.1 (2)	O543—S54—O541	114.45 (11)
C32—C31—C311	123.5 (2)	O542—S54—O541	111.60 (10)
C33—C32—C31	124.6 (2)	O543—S54—C54	105.28 (10)
C33—C32—N321	115.96 (19)	O542—S54—C54	104.17 (10)
C31—C32—N321	119.42 (18)	O541—S54—C54	105.00 (10)
C32—C33—C34	118.7 (2)	O562—N561—O561	125.2 (2)
C32—C33—H33	120.7	O562—N561—C56	118.3 (2)
C34—C33—H33	120.7	O561—N561—C56	116.5 (2)
C35—C34—C33	119.94 (19)	H1A—O1—H1B	102 (3)
C35—C34—S34	119.84 (16)	H1A—O1—H2C	126 (3)
C33—C34—S34	120.22 (16)	H1B—O1—H2C	126 (3)

C36—C35—C34	119.2 (2)	H2A—O2—H2B	112 (3)
C36—C35—H35	120.4	H2A—O2—H2C	111 (3)
C34—C35—H35	120.4	H2B—O2—H2C	110 (3)
C15—C11—C12—N13	−1.8 (2)	C33—C34—S34—O343	−168.30 (17)
N111—C11—C12—N13	178.77 (19)	C35—C34—S34—O341	132.94 (17)
C15—C11—C12—C121	173.7 (2)	C33—C34—S34—O341	−46.56 (19)
N111—C11—C12—C121	−5.8 (4)	C35—C34—S34—O342	−108.87 (18)
C11—C12—N13—N14	1.7 (2)	C33—C34—S34—O342	71.64 (19)
C121—C12—N13—N14	−174.35 (18)	C35—C36—N361—O362	−44.8 (3)
C11—C12—N13—C131	178.4 (2)	C31—C36—N361—O362	136.4 (2)
C121—C12—N13—C131	2.4 (3)	C35—C36—N361—O361	132.2 (2)
C12—N13—N14—C15	−1.0 (2)	C31—C36—N361—O361	−46.5 (3)
C131—N13—N14—C15	−178.01 (18)	C46—C41—C42—C43	−2.3 (3)
C12—N13—N14—C141	176.53 (18)	C411—C41—C42—C43	172.5 (2)
C131—N13—N14—C141	−0.5 (3)	C46—C41—C42—N421	179.04 (18)
N13—N14—C15—C11	−0.2 (2)	C411—C41—C42—N421	−6.1 (3)
C141—N14—C15—C11	−177.4 (2)	C41—C42—C43—C44	2.1 (3)
N13—N14—C15—C151	−178.23 (19)	N421—C42—C43—C44	−179.27 (18)
C141—N14—C15—C151	4.5 (3)	C42—C43—C44—C45	−0.4 (3)
C12—C11—C15—N14	1.2 (2)	C42—C43—C44—S44	−178.08 (15)
N111—C11—C15—N14	−179.33 (18)	C43—C44—C45—C46	−0.7 (3)
C12—C11—C15—C151	179.1 (2)	S44—C44—C45—C46	176.98 (16)
N111—C11—C15—C151	−1.4 (4)	C44—C45—C46—C41	0.4 (3)
C12—C11—N111—O112	174.5 (2)	C44—C45—C46—N461	−177.05 (19)
C15—C11—N111—O112	−4.9 (3)	C42—C41—C46—C45	1.0 (3)
C12—C11—N111—O111	−4.6 (3)	C411—C41—C46—C45	−173.6 (2)
C15—C11—N111—O111	176.05 (19)	C42—C41—C46—N461	178.36 (19)
C25—C21—C22—N23	−0.4 (2)	C411—C41—C46—N461	3.7 (3)
N211—C21—C22—N23	−177.86 (19)	C43—C42—N421—O421	117.8 (2)
C25—C21—C22—C221	178.5 (2)	C41—C42—N421—O421	−63.5 (3)
N211—C21—C22—C221	1.0 (4)	C43—C42—N421—O422	−62.1 (3)
C21—C22—N23—N24	0.2 (2)	C41—C42—N421—O422	116.7 (2)
C221—C22—N23—N24	−178.82 (19)	C45—C44—S44—O443	−146.56 (17)
C21—C22—N23—C231	−179.9 (2)	C43—C44—S44—O443	31.1 (2)
C221—C22—N23—C231	1.2 (3)	C45—C44—S44—O442	−25.34 (19)
C22—N23—N24—C25	0.1 (2)	C43—C44—S44—O442	152.36 (17)
C231—N23—N24—C25	−179.87 (18)	C45—C44—S44—O441	93.77 (18)
C22—N23—N24—C241	177.07 (18)	C43—C44—S44—O441	−88.53 (18)
C231—N23—N24—C241	−2.9 (3)	C45—C46—N461—O461	−147.5 (2)
N23—N24—C25—C21	−0.3 (2)	C41—C46—N461—O461	35.0 (3)
C241—N24—C25—C21	−176.9 (2)	C45—C46—N461—O462	30.5 (3)
N23—N24—C25—C251	178.74 (19)	C41—C46—N461—O462	−147.0 (2)
C241—N24—C25—C251	2.2 (3)	C56—C51—C52—C53	0.0 (3)
C22—C21—C25—N24	0.4 (2)	C511—C51—C52—C53	175.8 (2)
N211—C21—C25—N24	177.95 (19)	C56—C51—C52—N521	178.73 (19)
C22—C21—C25—C251	−178.5 (2)	C511—C51—C52—N521	−5.5 (3)
N211—C21—C25—C251	−1.0 (4)	C51—C52—C53—C54	0.0 (3)

C25—C21—N211—O212	−5.4 (3)	N521—C52—C53—C54	−178.77 (18)
C22—C21—N211—O212	171.7 (2)	C52—C53—C54—C55	−0.5 (3)
C25—C21—N211—O211	175.3 (2)	C52—C53—C54—S54	176.65 (16)
C22—C21—N211—O211	−7.6 (3)	C53—C54—C55—C56	1.0 (3)
C36—C31—C32—C33	1.3 (3)	S54—C54—C55—C56	−176.26 (17)
C311—C31—C32—C33	−175.3 (2)	C54—C55—C56—C51	−1.0 (3)
C36—C31—C32—N321	−178.28 (19)	C54—C55—C56—N561	177.1 (2)
C311—C31—C32—N321	5.1 (3)	C52—C51—C56—C55	0.5 (3)
C31—C32—C33—C34	−1.1 (3)	C511—C51—C56—C55	−175.3 (2)
N321—C32—C33—C34	178.51 (18)	C52—C51—C56—N561	−177.5 (2)
C32—C33—C34—C35	1.3 (3)	C511—C51—C56—N561	6.7 (3)
C32—C33—C34—S34	−179.18 (16)	C53—C52—N521—O522	137.4 (2)
C33—C34—C35—C36	−1.9 (3)	C51—C52—N521—O522	−41.5 (3)
S34—C34—C35—C36	178.60 (16)	C53—C52—N521—O521	−41.0 (3)
C34—C35—C36—C31	2.3 (3)	C51—C52—N521—O521	140.1 (2)
C34—C35—C36—N361	−176.42 (18)	C53—C54—S54—O543	−6.6 (2)
C32—C31—C36—C35	−1.9 (3)	C55—C54—S54—O543	170.62 (18)
C311—C31—C36—C35	174.7 (2)	C53—C54—S54—O542	−127.98 (18)
C32—C31—C36—N361	176.76 (18)	C55—C54—S54—O542	49.20 (19)
C311—C31—C36—N361	−6.6 (3)	C53—C54—S54—O541	114.59 (18)
C33—C32—N321—O322	38.2 (3)	C55—C54—S54—O541	−68.23 (19)
C31—C32—N321—O322	−142.1 (2)	C55—C56—N561—O562	−138.9 (2)
C33—C32—N321—O321	−139.3 (2)	C51—C56—N561—O562	39.3 (3)
C31—C32—N321—O321	40.3 (3)	C55—C56—N561—O561	39.1 (3)
C35—C34—S34—O343	11.2 (2)	C51—C56—N561—O561	−142.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*C*···O1	1.02 (5)	1.42 (5)	2.433 (3)	176 (4)
O1—H1*A*···O541	0.83 (4)	1.85 (4)	2.681 (3)	176 (4)
O1—H1*B*···O341	0.90 (3)	1.79 (4)	2.690 (2)	173 (3)
O2—H2*A*···O441^{i}	0.95 (3)	1.64 (4)	2.570 (2)	164 (3)
O2—H2*B*···O342^{ii}	0.87 (3)	1.73 (3)	2.590 (2)	170 (3)
C43—H43···O462^{iii}	0.95	2.46	3.401 (3)	172
C55—H55···O342^{ii}	0.95	2.55	3.212 (3)	127

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