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10,13-Bis(*p*-tolylsulfonyl)-1,4,7-trithia-10,13-diazacyclopentadecane

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Key indicators

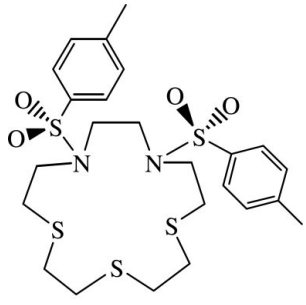
Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.013 Å
Disorder in main residue
R factor = 0.070
wR factor = 0.149
Data-to-parameter ratio = 8.4

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

The title compound, $C_{24}H_{34}N_2O_4S_5$, is the ditosylated precursor to the mixed aza–thia macrocycle 1,4,7-trithia-10,13-diazacyclopentadecane ([15]aneN_2S_3) and is prepared by reacting bis(2-mercaptoethyl) sulfide with *O*,*O*′,*N*,*N*′-tetratosyl-*N*,*N*′-bis(2-oxyethyl)ethylendiamine in dimethylformamide in the presence of Cs_2CO_3. Molecules lie across crystallographic twofold axes. The macrocyclic framework adopts a [33333] conformation and the two tolylsulfonyl groups are directed away from the ring cavity. There is extensive disorder of the methylene groups of the macrocyclic backbone.

Comment

Recently, aza–thioether macrocycles have been effectively used for the design and synthesis of selective heteroditopic receptors capable of binding both cationic and anionic moieties of a metal salt (Love *et al.*, 2001; Glenny *et al.*, 2003). The synthetic routes to aza–thioether macrocycles generally involve tosylated ring precursors, the only drawback to which is the detosylation procedure which may require long reaction times and generally gives low yields. For the synthesis of [15]aneN_2S_3 we prepared the title compound, (I), following a well established cyclization procedure under high dilution conditions. Unfortunately, all attempts to detosylate (I) failed to afford the deprotected macrocycle.



(I)

Molecules of (I) lie across crystallographic twofold axes (Fig. 1), with the axes passing through S7 and the mid-point of the C15—C15^{i} bond [symmetry code: (i) y, x, $-z$]. The macrocyclic framework exhibits a [33333] conformation (Fig. 2) and the two tolylsulfonyl groups are directed away from the ring cavity. The C atoms of the N—C—C—S and S—C—C—S linkages are each disordered over two sites, with group occupancies of 0.705 (12) and 0.295 (12) for the major and minor components, respectively.

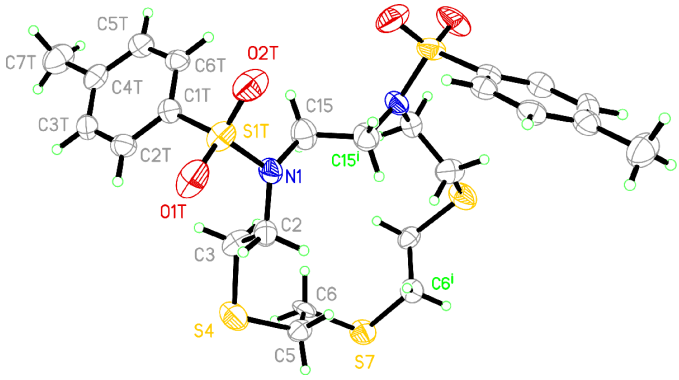


Figure 1
A view of the structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. The minor disorder component has been omitted for clarity. [Symmetry code: (i) $y, x, -z$.]

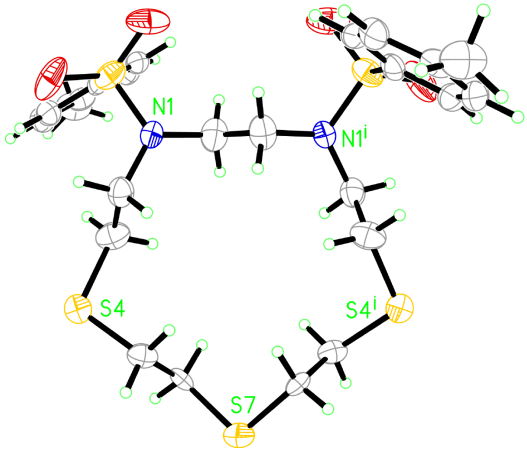


Figure 2
An alternative view showing the [33333] ring conformation. Ellipsoids are drawn at the 30% probability level and the minor disorder component has been omitted for clarity. [Symmetry code: (i) $y, x, -z$.]

Experimental

A 5 l three-necked flask fitted with a precision dropping funnel and a mechanical stirrer was purged with nitrogen. Freshly distilled dimethylformamide (DMF; 1.8 l) and dry Cs_2CO_3 (11.0 g, 0.0338 mol) were added and the solution was heated to 333 K. A solution of bis(2-mercaptoethyl) sulfide (2.61 g, 0.0169 mol) and *O,O′,N,N′*-tetratosyl-*N,N′*-bis(2-oxyethyl)ethylendiamine (12.93 g, 0.0169 mol) in DMF (250 ml) was then added dropwise over a period of 12 h. After the addition was complete, a second portion of Cs_2CO_3 (11.0 g, 0.0338 mol) was added and an identical solution as before was added over a further 12 h period. Once all the reagents had been added, the reaction mixture was stirred for 6 h at 333 K. The DMF was then removed *in vacuo* and the residue was dissolved in CH_2Cl_2, washed with water and concentrated *in vacuo*. The residue was crystallized from hot ethanol to give a white solid of the desired product (9.45 g, 48.6% yield). Crystals suitable for X-ray diffraction analysis were grown by diffusion of Et_2O vapour into a CH_2Cl_2 solution of the product. Elemental analysis, found (calculated for $C_{24}H_{34}N_2O_4S_5$): C 49.95 (50.15), H 5.88 (5.96), N 4.77 (4.87)%.

Crystal data

$C_{24}H_{34}N_2O_4S_5$
M_r = 574.83
Tetragonal, $P4_12_12$
a = 12.377 (3) Å
c = 18.296 (1) Å
V = 2802.8 (10) Å^3
Z = 4
D_x = 1.362 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35 reflections
θ = 10.1–14.9°
μ = 0.45 mm^{-1}
T = 220 (2) K
Column, colourless
0.50 × 0.39 × 0.20 mm

Data collection

Stoe STADI-4 four-circle diffractometer
ω scans
Absorption correction: none
3449 measured reflections
1491 independent reflections
911 reflections with $I > 2\sigma(I)$
R_{int} = 0.115
θ_{max} = 25.1°
$h = -9 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity variation: ±3.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.149
S = 1.11
1491 reflections
178 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.017P)^2 + 7.15P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.018
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 997 Friedel pairs
Flack parameter = 0.0 (3)

The C atoms of the N—C—C—S and S—C—C—S linkages are each disordered over two sites. This was modelled in terms of two orientations for each linkage and restraints were applied to the relevant C—C, C—N and C—S distances. The occupancies of the two components (C2/C3/C5/C6 and C2′/C3′/C5′/C6′) converged at 0.705 (12) and 0.295 (12), respectively. Our decision to refine a single parameter to describe the disorder of C2/C3 and C5/C6 was based on two observations. The first was that independent refinement of the occupancies of the two dimethylene links gave very similar values. The second is that the angles at the central S atom are both more consistent and typical (104.0 and 104.8°) when the major and minor components are not mixed, but less so (87.7 and 99.0°) with major/minor and minor/major combinations. For these reasons, we believe that the disorder of the two $-CH_2-CH_2-$ units is a concerted phenomenon.

Methyl H atoms were located in ΔF syntheses and refined as part of rigid rotating groups, with $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were positioned geometrically and refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$. C—H distances of 0.94, 0.97 and 0.98 Å were used for aryl, methyl and methylene H atoms, respectively. Rigid-bond restraints were applied to the anisotropic displacement parameters. With a value of 0.0 (3), the Flack (1983) parameter was not reliably determined.

Data collection: *STADI*4 (Stoe & Cie, 1997); cell refinement: *STADI*4; data reduction: *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *PLATON* (Spek, 2003).

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supporting information

Acta Cryst. (2004). E**60**, o901–o903 [https://doi.org/10.1107/S1600536804009201]

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Stoe Stadi-4 four-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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3449 measured reflections
1491 independent reflections
911 reflections with $I > 2\sigma(I)$
$R_{int} = 0.115$
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$h = -9 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 21$
3 standard reflections every 60 min
intensity decay: 7.4%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.149$
$S = 1.11$
1491 reflections
178 parameters
55 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.017P)^2 + 7.15P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.018$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.0 (3)

Special details

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. Model file used for *SHELXL97* refinement:
TITL N2S3TS #433 for VL/MS in P 41 21 2 *CELL* 0.71073 12.3770 12.3770 18.2960 90.000 90.000 90.000 ZERR 4.00 0.003 0.003 0.0010 0.000 0.000 0.000 L A T T -1 SYMM –*X*, –Y, 0.5+*Z* SYMM 0.5-Y, 0.5+*X*, 0.25+*Z* SYMM 0.5+Y, 0.5-*X*, 0.75+*Z* SYMM 0.5-*X*, 0.5+Y, 0.25-*Z* SYMM 0.5+*X*, 0.5-Y, 0.75-*Z* SYMM Y, *X*, –*Z* SYMM –Y, –*X*, 0.5-*Z* SFAC C H N O *S* UNIT 96 136 8 16 20 *OMIT* -4 MERG 4 TEMP -53 SIZE 0.50 0.39 0.20 *L·S*. 4 BOND $H CONF ACTA FMAP 2 PLAN 10 EQIV $1 +Y, +*X*, –*Z DFIX* 1.52 0.005 C2 C3 C2′ C3′ C5 C6 C5′ C6′ C15 C15_$1 *DFIX* 1.47 0.005 N1 C2 N1 C2′ *DFIX* 1.82 0.005 S4 C3 S4 C3′ S4 C5 S4 C5′ S7 C6 S7 C6′ DELU 0.01 W GH T 0.0168 7.15 REM +++ EXTI refined to within less than three σ of zero FVAR 0.49538 0.70476 N1 3 0.735062 0.927651 0.043156 11.00000 0.05970 0.04729 = 0.07075 - 0.01013 0.00964 0.00513 PART 1 C2 1 0.640727 0.879603 0.080370 21.00000 0.06581 0.05802 = 0.06347 0.00394 0.00342 0.01167 AFIX 23 H2A 2 0.617862 0.926621 0.120595 21.00000 - 1.20000 H2B 2 0.660707 0.809302 0.100978 21.00000 - 1.20000 AFIX 0 C3 1 0.548333 0.865367 0.026984 21.00000 0.09527 0.06778 = 0.05940 0.00981 - 0.00573 - 0.01719 AFIX 23 H3A 2 0.572327 0.821150 - 0.014378 21.00000 - 1.20000 H3B 2 0.526279 0.936085 0.008067 21.00000 - 1.20000 AFIX 0 PART 2 C2′ 1 0.619903 0.900815 0.034206 - 21.00000 0.05683 AFIX 23 H2C 2 0.609599 0.867727 - 0.013955 - 21.00000 - 1.20000 H2D 2 0.577896 0.967915 0.035240 - 21.00000 - 1.20000 AFIX 0 C3′ 1 0.575562 0.824740 0.092029 - 21.00000 0.07925 AFIX 23 H3C 2 0.582847 0.857239 0.140624 - 21.00000 - 1.20000 H3D 2 0.615683 0.756483 0.091483 - 21.00000 - 1.20000 AFIX 0 PART 0 S4 5 0.433598 0.800436 0.071422 11.00000 0.06760 0.07369 = 0.12502 - 0.02089 0.02130 - 0.00326 PART 1 C5 1 0.464859 0.657719 0.069440 21.00000 0.05062 0.06294 = 0.06178 0.00940 - 0.00779 - 0.00998 AFIX 23 H5A 2 0.540430 0.647473 0.083845 21.00000 - 1.20000 H5B 2 0.419516 0.620336 0.105338 21.00000 - 1.20000 AFIX 0 C6 1 0.447306 0.607106 - 0.004996 21.00000 0.03221 0.06648 = 0.07131 - 0.00366 0.02165 - 0.00805 AFIX 23 H6A 2 0.500259 0.636124 - 0.039644 21.00000 - 1.20000 H6B 2 0.374957 0.625544 - 0.022830 21.00000 - 1.20000 AFIX 0 PART 2 C5′ 1 0.434389 0.673012 0.021875 - 21.00000 0.06710 AFIX 23 H5C 2 0.360854 0.659150 0.004367 - 21.00000 - 1.20000 H5D 2 0.480506 0.681675 - 0.021269 - 21.00000 - 1.20000 AFIX 0 C6′ 1 0.472442 0.573803 0.063263 - 21.00000 0.10067 AFIX 23 H6C 2 0.427187 0.561538 0.106425 - 21.00000 - 1.20000 H6D 2 0.547503 0.582849 0.079178 - 21.00000 - 1.20000 AFIX 0 PART 0 S7 5 0.461100 0.461100 0.000000 10.50000 0.06350 0.06350 = 0.10442 - 0.00451 0.00451 - 0.01544 C15 1 0.792950 0.872234 - 0.016487 11.00000 0.08153 0.07895 = 0.07217 - 0.01077 - 0.00253 0.01636 AFIX 23 H15A 2 0.831582 0.925195 - 0.046528 11.00000 - 1.20000 H15B 2 0.741374 0.833867 - 0.047762 11.00000 - 1.20000 AFIX 0 S1T 5 0.754410 1.057717 0.046421 11.00000 0.09290 0.05383 = 0.05038 - 0.00765 - 0.00739 - 0.00741 O1T 4 0.699563 1.097345 0.109116 11.00000 0.18739 0.06157 = 0.04539 - 0.01209 0.00841 0.01674 O2T 4 0.868456 1.073434 0.040521 11.00000 0.08381 0.09360 = 0.10118 0.02606 - 0.03988 - 0.03498 C1T 1 0.693513 1.114643 - 0.031886 11.00000 0.06165 0.04223 = 0.04823 - 0.01376 0.00417 - 0.00018 C2T 1 0.586384 1.147639 - 0.027346 11.00000 0.06479 0.05854 = 0.06288 - 0.00413 0.01500 - 0.00455 AFIX 43 H2TA 2 0.548196 1.142142 0.016908 11.00000 - 1.20000 AFIX 0 C3T 1 0.536909 1.188864 - 0.089447 11.00000 0.05371 0.05237 = 0.09108 0.00657 0.00319 0.00717 AFIX 43 H3TA 2 0.464629 1.211866 - 0.086878 11.00000 - 1.20000 AFIX 0 C4T 1 0.592127 1.196791 - 0.155316 11.00000 0.07742 0.04549 = 0.07335 0.00420 - 0.00491 - 0.00842 C5T 1 0.698365 1.160743 - 0.157140 11.00000 0.07718 0.05803 = 0.04430 - 0.00819 0.00887 - 0.00212 AFIX 43 H5T 2 0.736728 1.163149 - 0.201451 11.00000 - 1.20000 AFIX 0 C6T 1 0.748295 1.121920 - 0.096063 11.00000 0.05646 0.04718 = 0.05703 - 0.00600 0.01037 - 0.00748 AFIX 43 H6T 2 0.820957 1.100096 - 0.098390 11.00000 - 1.20000 AFIX 0 C7T 1 0.538574 1.241837 - 0.223153 11.00000 0.09661 0.09977 = 0.09504 0.02563 - 0.02563 - 0.01422 AFIX 137 H7T1 2 0.563486 1.315128 - 0.231431 11.00000 - 1.50000 H7T2 2 0.460809 1.241960 - 0.216571 11.00000 - 1.50000 H7T3 2 0.557103 1.197292 - 0.264933 11.00000 - 1.50000
HKLF 4

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.7351 (6)	0.9277 (6)	0.0432 (4)	0.0592 (19)	
C2	0.6407 (7)	0.8796 (11)	0.0804 (6)	0.062 (4)	0.705 (12)
H2A	0.6179	0.9266	0.1206	0.075*	0.705 (12)
H2B	0.6607	0.8093	0.1010	0.075*	0.705 (12)
C3	0.5483 (7)	0.8654 (11)	0.0270 (6)	0.074 (4)	0.705 (12)
H3A	0.5723	0.8211	−0.0144	0.089*	0.705 (12)
H3B	0.5263	0.9361	0.0081	0.089*	0.705 (12)
C2′	0.6199 (11)	0.901 (3)	0.0342 (16)	0.057 (10)*	0.295 (12)

H2C	0.6096	0.8677	−0.0140	0.068*	0.295 (12)
H2D	0.5779	0.9679	0.0352	0.068*	0.295 (12)
C3′	0.5756 (9)	0.825 (3)	0.0920 (16)	0.079 (12)*	0.295 (12)
H3C	0.5828	0.8572	0.1406	0.095*	0.295 (12)
H3D	0.6157	0.7565	0.0915	0.095*	0.295 (12)
S4	0.4336 (3)	0.8004 (3)	0.07142 (19)	0.0888 (11)	
C5	0.4649 (11)	0.6577 (5)	0.0694 (5)	0.058 (4)	0.705 (12)
H5A	0.5404	0.6475	0.0838	0.070*	0.705 (12)
H5B	0.4195	0.6203	0.1053	0.070*	0.705 (12)
C6	0.4473 (9)	0.6071 (4)	−0.0050 (5)	0.057 (3)	0.705 (12)
H6A	0.5003	0.6361	−0.0396	0.068*	0.705 (12)
H6B	0.3750	0.6255	−0.0228	0.068*	0.705 (12)
C5′	0.434 (3)	0.6730 (13)	0.0219 (15)	0.067 (11)*	0.295 (12)
H5C	0.3609	0.6591	0.0044	0.081*	0.295 (12)
H5D	0.4805	0.6817	−0.0213	0.081*	0.295 (12)
C6′	0.472 (4)	0.5738 (15)	0.0633 (13)	0.101 (15)*	0.295 (12)
H6C	0.4272	0.5615	0.1064	0.121*	0.295 (12)
H6D	0.5475	0.5828	0.0792	0.121*	0.295 (12)
S7	0.4611 (2)	0.4611 (2)	0.0000	0.0771 (12)	
C15	0.7930 (7)	0.8722 (7)	−0.0165 (5)	0.078 (3)	
H15A	0.8316	0.9252	−0.0465	0.093*	
H15B	0.7414	0.8339	−0.0478	0.093*	
S1T	0.7544 (3)	1.0577 (2)	0.04642 (13)	0.0657 (8)	
O1T	0.6996 (8)	1.0973 (6)	0.1091 (3)	0.098 (3)	
O2T	0.8685 (6)	1.0734 (6)	0.0405 (4)	0.093 (2)	
C1T	0.6935 (8)	1.1146 (7)	−0.0319 (4)	0.051 (2)	
C2T	0.5864 (8)	1.1476 (7)	−0.0273 (5)	0.062 (3)	
H2TA	0.5482	1.1421	0.0169	0.074*	
C3T	0.5369 (8)	1.1889 (8)	−0.0894 (5)	0.066 (3)	
H3TA	0.4646	1.2119	−0.0869	0.079*	
C4T	0.5921 (9)	1.1968 (8)	−0.1553 (6)	0.065 (3)	
C5T	0.6984 (9)	1.1607 (7)	−0.1571 (5)	0.060 (2)	
H5T	0.7367	1.1631	−0.2015	0.072*	
C6T	0.7483 (8)	1.1219 (7)	−0.0961 (4)	0.054 (2)	
H6T	0.8210	1.1001	−0.0984	0.064*	
C7T	0.5386 (9)	1.2418 (9)	−0.2232 (5)	0.097 (4)	
H7T1	0.5635	1.3151	−0.2314	0.146*	
H7T2	0.4608	1.2420	−0.2166	0.146*	
H7T3	0.5571	1.1973	−0.2649	0.146*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.060 (5)	0.047 (4)	0.071 (5)	0.005 (4)	0.010 (4)	−0.010 (4)
C2	0.066 (7)	0.058 (8)	0.063 (9)	0.012 (6)	0.003 (6)	0.004 (8)
C3	0.095 (9)	0.068 (9)	0.059 (8)	−0.017 (8)	−0.006 (7)	0.010 (8)
S4	0.068 (2)	0.074 (2)	0.125 (3)	−0.0033 (17)	0.0213 (19)	−0.0209 (19)
C5	0.051 (9)	0.063 (6)	0.062 (8)	−0.010 (6)	−0.008 (8)	0.009 (6)

C6	0.032 (7)	0.066 (6)	0.071 (8)	−0.008 (6)	0.022 (7)	−0.004 (6)
S7	0.0635 (15)	0.0635 (15)	0.104 (3)	−0.015 (2)	0.0045 (18)	−0.0045 (18)
C15	0.082 (7)	0.079 (7)	0.072 (7)	0.016 (6)	−0.003 (6)	−0.011 (6)
S1T	0.093 (2)	0.0538 (15)	0.0504 (13)	−0.0074 (15)	−0.0074 (15)	−0.0076 (13)
O1T	0.187 (9)	0.062 (5)	0.045 (4)	0.017 (5)	0.008 (5)	−0.012 (3)
O2T	0.084 (5)	0.094 (6)	0.101 (5)	−0.035 (4)	−0.040 (4)	0.026 (5)
C1T	0.062 (5)	0.042 (5)	0.048 (4)	0.000 (4)	0.004 (4)	−0.014 (4)
C2T	0.065 (6)	0.059 (6)	0.063 (5)	−0.005 (5)	0.015 (5)	−0.004 (5)
C3T	0.054 (6)	0.052 (6)	0.091 (6)	0.007 (5)	0.003 (5)	0.007 (6)
C4T	0.077 (6)	0.045 (6)	0.073 (5)	−0.008 (5)	−0.005 (5)	0.004 (5)
C5T	0.077 (6)	0.058 (6)	0.044 (4)	−0.002 (5)	0.009 (5)	−0.008 (5)
C6T	0.056 (5)	0.047 (5)	0.057 (5)	−0.007 (5)	0.010 (4)	−0.006 (4)
C7T	0.097 (9)	0.100 (9)	0.095 (7)	−0.014 (7)	−0.026 (7)	0.026 (7)

Geometric parameters (Å, º)

N1—C2	1.477 (5)	C5′—H5D	0.9800
N1—C2′	1.473 (5)	C6′—S7	1.818 (5)
N1—C15	1.475 (11)	C6′—H6C	0.9800
N1—S1T	1.629 (7)	C6′—H6D	0.9800
C2—C3	1.514 (5)	S7—C6^{i}	1.817 (5)
C2—H2A	0.9800	S7—C6′^{i}	1.818 (5)
C2—H2B	0.9800	C15—C15^{i}	1.513 (12)
C3—S4	1.823 (5)	C15—H15A	0.9800
C3—H3A	0.9800	C15—H15B	0.9800
C3—H3B	0.9800	S1T—O1T	1.420 (7)
C2′—C3′	1.519 (5)	S1T—O2T	1.429 (7)
C2′—H2C	0.9800	S1T—C1T	1.766 (9)
C2′—H2D	0.9800	C1T—C6T	1.359 (11)
C3′—S4	1.822 (5)	C1T—C2T	1.390 (12)
C3′—H3C	0.9800	C2T—C3T	1.388 (12)
C3′—H3D	0.9800	C2T—H2TA	0.9400
S4—C5	1.809 (5)	C3T—C4T	1.389 (12)
S4—C5′	1.819 (5)	C3T—H3TA	0.9400
C5—C6	1.515 (5)	C4T—C5T	1.389 (14)
C5—H5A	0.9800	C4T—C7T	1.513 (12)
C5—H5B	0.9800	C5T—C6T	1.364 (12)
C6—S7	1.817 (5)	C5T—H5T	0.9400
C6—H6A	0.9800	C6T—H6T	0.9400
C6—H6B	0.9800	C7T—H7T1	0.9700
C5′—C6′	1.518 (5)	C7T—H7T2	0.9700
C5′—H5C	0.9800	C7T—H7T3	0.9700
C15—N1—C2′	106.4 (16)	S4—C5′—H5D	108.0
C15—N1—C2	122.6 (9)	H5C—C5′—H5D	107.3
C15—N1—S1T	114.5 (6)	C5′—C6′—S7	106.2 (8)
C2—N1—S1T	119.8 (7)	C5′—C6′—H6C	110.5
C2′—N1—S1T	111.7 (14)	S7—C6′—H6C	110.5

N1—C2—C3	110.3 (7)	C5′—C6′—H6D	110.5
N1—C2—H2A	109.6	S7—C6′—H6D	110.5
C3—C2—H2A	109.6	H6C—C6′—H6D	108.7
N1—C2—H2B	109.6	C6—S7—C6^{i}	100.9 (8)
C3—C2—H2B	109.6	C6′—S7—C6′^{i}	107 (3)
H2A—C2—H2B	108.1	N1—C15—C15^{i}	108.8 (9)
C2—C3—S4	110.6 (6)	N1—C15—H15A	109.9
C2—C3—H3A	109.5	C15^{i}—C15—H15A	109.9
S4—C3—H3A	109.5	N1—C15—H15B	109.9
C2—C3—H3B	109.5	C15^{i}—C15—H15B	109.9
S4—C3—H3B	109.5	H15A—C15—H15B	108.3
H3A—C3—H3B	108.1	O1T—S1T—O2T	119.1 (5)
N1—C2′—C3′	114.3 (13)	O1T—S1T—N1	107.5 (5)
N1—C2′—H2C	108.7	O2T—S1T—N1	106.1 (4)
C3′—C2′—H2C	108.7	O1T—S1T—C1T	108.3 (5)
N1—C2′—H2D	108.7	O2T—S1T—C1T	107.8 (5)
C3′—C2′—H2D	108.7	N1—S1T—C1T	107.6 (4)
H2C—C2′—H2D	107.6	C6T—C1T—C2T	120.5 (9)
C2′—C3′—S4	107.9 (9)	C6T—C1T—S1T	121.0 (8)
C2′—C3′—H3C	110.1	C2T—C1T—S1T	118.4 (7)
S4—C3′—H3C	110.1	C3T—C2T—C1T	118.7 (9)
C2′—C3′—H3D	110.1	C3T—C2T—H2TA	120.7
S4—C3′—H3D	110.1	C1T—C2T—H2TA	120.7
H3C—C3′—H3D	108.4	C2T—C3T—C4T	121.3 (9)
C5′—S4—C3′	103.9 (18)	C2T—C3T—H3TA	119.4
C5—S4—C3	104.8 (7)	C4T—C3T—H3TA	119.4
C6—C5—S4	113.0 (5)	C3T—C4T—C5T	117.6 (9)
C6—C5—H5A	109.0	C3T—C4T—C7T	121.5 (10)
S4—C5—H5A	109.0	C5T—C4T—C7T	120.9 (10)
C6—C5—H5B	109.0	C6T—C5T—C4T	121.5 (9)
S4—C5—H5B	109.0	C6T—C5T—H5T	119.3
H5A—C5—H5B	107.8	C4T—C5T—H5T	119.3
C5—C6—S7	110.6 (5)	C1T—C6T—C5T	120.3 (9)
C5—C6—H6A	109.5	C1T—C6T—H6T	119.8
S7—C6—H6A	109.5	C5T—C6T—H6T	119.8
C5—C6—H6B	109.5	C4T—C7T—H7T1	109.5
S7—C6—H6B	109.5	C4T—C7T—H7T2	109.5
H6A—C6—H6B	108.1	H7T1—C7T—H7T2	109.5
C6′—C5′—S4	117.0 (12)	C4T—C7T—H7T3	109.5
C6′—C5′—H5C	108.0	H7T1—C7T—H7T3	109.5
S4—C5′—H5C	108.0	H7T2—C7T—H7T3	109.5
C6′—C5′—H5D	108.0		

C15—N1—C2—C3	−63.6 (13)	C2′—N1—S1T—O2T	−168.8 (13)
S1T—N1—C2—C3	95.5 (11)	C2—N1—S1T—O2T	151.6 (7)
N1—C2—C3—S4	177.4 (8)	C15—N1—S1T—C1T	67.4 (7)
C15—N1—C2′—C3′	109 (3)	C2′—N1—S1T—C1T	−53.7 (14)
C2—N1—C2′—C3′	−14 (2)	C2—N1—S1T—C1T	−93.3 (8)

S1T—N1—C2′—C3′	−125 (3)	O1T—S1T—C1T—C6T	157.5 (8)
N1—C2′—C3′—S4	−180 (2)	O2T—S1T—C1T—C6T	27.4 (9)
C2′—C3′—S4—C5′	97 (3)	N1—S1T—C1T—C6T	−86.6 (8)
C2—C3—S4—C5	−82.1 (11)	O1T—S1T—C1T—C2T	−25.3 (9)
C3—S4—C5—C6	−76.6 (10)	O2T—S1T—C1T—C2T	−155.4 (8)
S4—C5—C6—S7	−171.7 (7)	N1—S1T—C1T—C2T	90.6 (8)
C3′—S4—C5′—C6′	64 (3)	C6T—C1T—C2T—C3T	−0.5 (14)
S4—C5′—C6′—S7	179 (2)	S1T—C1T—C2T—C3T	−177.7 (7)
C5—C6—S7—C6^{i}	−76.4 (8)	C1T—C2T—C3T—C4T	0.4 (15)
C5′—C6′—S7—C6′^{i}	76 (3)	C2T—C3T—C4T—C5T	0.8 (15)
C2′—N1—C15—C15^{i}	−118.6 (12)	C2T—C3T—C4T—C7T	−179.9 (9)
C2—N1—C15—C15^{i}	−82.4 (8)	C3T—C4T—C5T—C6T	−2.0 (14)
S1T—N1—C15—C15^{i}	117.5 (4)	C7T—C4T—C5T—C6T	178.7 (9)
C15—N1—S1T—O1T	−176.2 (6)	C2T—C1T—C6T—C5T	−0.7 (14)
C2′—N1—S1T—O1T	62.7 (14)	S1T—C1T—C6T—C5T	176.5 (7)
C2—N1—S1T—O1T	23.1 (8)	C4T—C5T—C6T—C1T	2.0 (14)
C15—N1—S1T—O2T	−47.8 (7)		

Symmetry code: (i) *y*, *x*, −*z*.