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4,17,25,26-Tetraaza-6,9,12,15-tetraoxa-2,19,21,24-tetrathiatricyclo[18.4.1^{1,4}.1^{17,20}]hexacosa-1(25),20(26)-diene-3,5,16,18-tetraone

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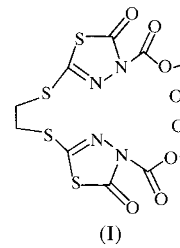
The title compound, $C_{14}H_{16}N_4O_8S_4$, has crystallographic C_2 symmetry with half a molecule in the asymmetric unit and a dihedral angle of 58.7 (1)° between the two planar 1,3,4-thiadiazole five-membered rings of the macrocyclic, giving the molecule a twisted conformation.

Comment

The determination of the structure of the title compound, (I), is part of our continuing study of the molecular structures of macrocycles containing 1,3,4-thiadiazole subunits (Cho, Park & Hwang, 1999; Cho, Park, Kim *et al.*, 1999). These compounds are of interest because of their potential activity as artificial receptors of transition metals and other small organic molecules.



(I)

Half a molecule of (I) belongs to the asymmetric unit and a molecule is completed by the crystallographic twofold axis (see Fig. 1). The S—C bond lengths range from 1.739 (3) to 1.800 (3) Å, with a mean value of 1.766 (2) Å, which is similar to that found in the *International Tables for Crystallography* (Vol. C). The C2—S2—C3 angle of 99.55 (17)° is similar to that found in (2*S*,4*S*,5*R*)-(−)-3,4-dimethyl-5-phenyl-2-(1,3-thiazol-2-yl)-1,3-oxazolidine (Fitzsimons & Gallagher, 1999). The O2—C4 [1.188 (4) Å], O4—C3 [1.193 (4) Å] and N1—C2 [1.285 (4) Å] bond lengths all show clearly double-bond character; the remainder of the bonds are single bonds. The five-membered ring, 5-mercapto-3*H*-1,3,4-thiadiazolin-2-one, is planar to within 0.008 (2) Å. The dihedral angle between the two five-membered rings of the molecule is 58.7 (1)°. The two half molecules are twisted around the twofold axis with torsion angles S1—C1—C1^{i}—S1^{i} of 82.9 (3)° [symmetry code: (i) $1 - x, y, \frac{3}{2} - z$] and O1—C7—C7^{i}—O1^{i} of −76.2 (5)°, so that O3···O3^{i} = 6.984 (5), N1···N1^{i} = 5.706 (6) and N1···O3^{i} = 6.661 (4) Å. The S1, N1, O3 and O1 atoms in an asymmetric unit lie in a plane within 0.042 (4) Å, with C1 and C7 deviating by −0.595 (4) and 0.194 (5) Å, respectively, from the best plane.

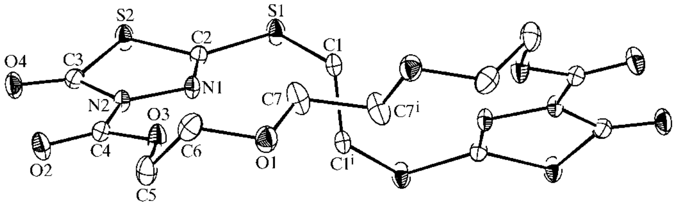


Figure 1
ORTEP (Farrugia, 1998) diagram, drawn with 40% probability displacement ellipsoids, showing the twisted conformation of (I). Only the asymmetric unit is labelled and H atoms are omitted for clarity. [Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.]

Experimental

The macrocycle is derived from α,α′-bis[(5-oxa-2,3-dihydro-1,3,4-thiadiazol-2-yl)thio]ethane (Cho, Park, Kim *et al.*, 1999). The details of the synthesis will be reported elsewhere.

Crystal data

$C_{14}H_{16}N_4O_8S_4$
M_r = 496.55
Monoclinic, $C2/c$
a = 15.151 (3) Å
b = 12.739 (3) Å
c = 10.5509 (13) Å
β = 97.888 (13)°
V = 2017.1 (7) Å^3
Z = 4

D_x = 1.635 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.902–14.06°
μ = 0.523 mm^{-1}
T = 291 K
Block, colourless
0.20 × 0.17 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: empirical using intensity measurements (Harms & Wocadlo, 1995)
$T_{\rm min}$ = 0.904, $T_{\rm max}$ = 0.942
3616 measured reflections
1772 independent reflections

1059 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.054
$\theta_{\rm max}$ = 24.95°
$h = -17 \rightarrow 17$
$k = -15 \rightarrow 15$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 169 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.092
S = 1.018
1772 reflections
136 parameters

H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.24 e Å^{-3}
$\Delta\rho_{\rm max}$ = −0.20 e Å^{-3}

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *CAD*-4 *EXPRESS*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1998). Molecular graphics: *ORTEP*3 for Windows (Farrugia, 1998). Software used to prepare material for publication: *WinGX* (Farrugia, 1998).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JA1010). Services for accessing these data are described at the back of the journal.

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