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1,2,4-Triazolo[2,3-*h*]-7,9-thiaza-11-crown-4

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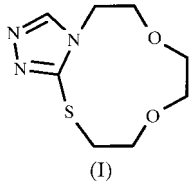
The title compound, 4,7-dioxa-10-thia-1,12,13-triazabicyclo[9.3.0]tetradeca-11,13-diene, $C_8H_{13}N_3O_2S$, contains an 11-membered ring, which appears in a chair conformation and has approximate mirror symmetry. It may be used for the complexation of metal atoms.

Comment

The elaboration of new molecular models for the recognition and the transport of metal atoms constitutes a field of research which has developed rapidly during recent years (Pedersen, 1967; Reinhoudt *et al.*, 1976; Cram & Ho, 1986; Bradshaw *et al.*, 1986; Kumar *et al.*, 1992). The title compound, (I), had been synthesized with the aim to study its complexing properties. Since the condensation of dichlorotriethylene glycol with 1,2,4-triazole-5-thione could lead to two different constitutional isomers, which could not be distinguished by NMR spectroscopy, we have carried out an X-ray structure analysis to establish unambiguously the constitution of the reaction product.



(I)

The geometry of the title compound shows no unusual features. The conformation of the crown ether can be described as a chair, with C5 and the bond N10—C11 being the two end points. The torsion-angle pattern in the ring shows an approximate mirror symmetry with the mirror plane running through C5 and the centre of the N10—C11 bond. Only the torsion angles C2—C3—O4—C5 and C5—C6—O7—C8 are close to an antiperiplanar conformation, while apart from C9—N10—C11—S1, which is synperiplanar, all other torsion angles are more or less anti- or synclinal, respectively.

Experimental

To a solution of 1,2,4-triazole-5-thione (1.01 g, 0.01 mol) and dichlorotriethylene glycol (1.87 g, 0.01 mol) in dimethylformamide (60 ml), potassium carbonate (4.15 g, 0.03 mol) were added. The mixture was stirred for 24 h at 303 K, then filtred and dried. The residue was extracted, dried and recrystallized from ethyl acetate.

Crystal data

$C_8H_{13}N_3O_2S$	D_x = 1.453 Mg m^{-3}
M_r = 215.27	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1147 reflections
a = 8.230 (1) Å	θ = 1–25°
b = 15.423 (2) Å	μ = 0.307 mm^{-1}
c = 8.476 (1) Å	T = 173 (2) K
β = 113.81 (3)°	Block, colourless
V = 984.3 (2) Å^3	0.50 × 0.30 × 0.20 mm
Z = 4	

Data collection

Siemens CCD three-circle diffractometer	R_{int} = 0.066
ω scans	θ_{max} = 27.48°
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)	$h = -10 \rightarrow 10$
T_{min} = 0.862, T_{max} = 0.941	$k = -20 \rightarrow 20$
13 240 measured reflections	$l = -11 \rightarrow 11$
2257 independent reflections	115 standard reflections
1673 reflections with $I > 2\sigma(I)$	frequency: 960 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 0.3771P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.081	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
S = 1.032	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
2257 reflections	
127 parameters	
H-atom parameters constrained	

H-atom positions were idealized and constrained to ride on their parent atoms; aromatic C—H = 0.95 Å or secondary C—H = 0.99 Å, and fixed individual displacement parameters [U(H) = 1.2U_{eq}(C)].

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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