

## 1,5-[5,5'-(1,3-Phenylenedimethylenedithio)bis(2,3-dihydro-2-oxo-1,3,4-thiadiazol-3-yl)]-3-oxapentane

Nam Sook Cho,<sup>a</sup> Soon Il Hong,<sup>a</sup>  
Geum-Hong Choo,<sup>b</sup> Jin-Gyu  
Kim<sup>c</sup> and Il-Hwan Suh<sup>c\*</sup><sup>a</sup>Department of Chemistry, Chungnam National University, Taejeon 305-764, Korea, <sup>b</sup>Korea University of Technology and Education, Cheonan PO Box 55, 330-800, Korea, and <sup>c</sup>Department of Physics, Chungnam National University, Taejeon 305-764, Korea

Correspondence e-mail: ihsuh@cnu.ac.kr

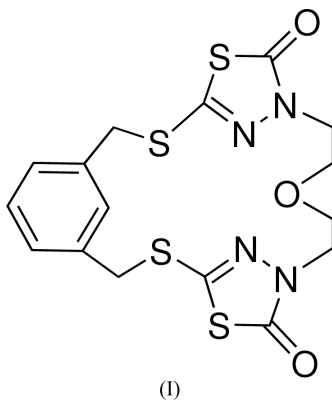
## Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(C-C)$  = 0.008 Å  
R factor = 0.061  
wR factor = 0.142  
Data-to-parameter ratio = 14.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Two molecules of the title compound, 3,5,15,17-tetra-thia-7,13,24,25-tetraazatetracyclo[17.3.1.1<sup>4,7</sup>.1<sup>13,16</sup>]pentacos-1(23),4(24),16(25),19,21-pentaene-6,14-dione, C<sub>16</sub>H<sub>16</sub>N<sub>4</sub>O<sub>3</sub>S<sub>4</sub>, having slightly different hollow and curved conformations, form the asymmetric unit. Each molecule provides an internal cavity of cage-like shape to form a host–guest complex.

## Comment

As a continuation of our studies relating to the synthesis of host compounds containing 5-mercapto-2,3-dihydro-1,3,4-thiadiazol-2-ones (Cho, Park, Kim *et al.*, 1999; Cho, Park, Hwang *et al.*, 1999; Cho *et al.*, 2000), we have prepared macrocycle (I) composed of two 5-mercapto-2,3-dihydro-1,3,4-thiadiazol-2-ones and *m*-xylene. The determination of the structure of (I) is part of our interest in dealing with the macrocycles and this compound might be a potential host molecule for a palladium metal ion. Thus, we are in the process of examining the role of this compound as an artificial receptor.

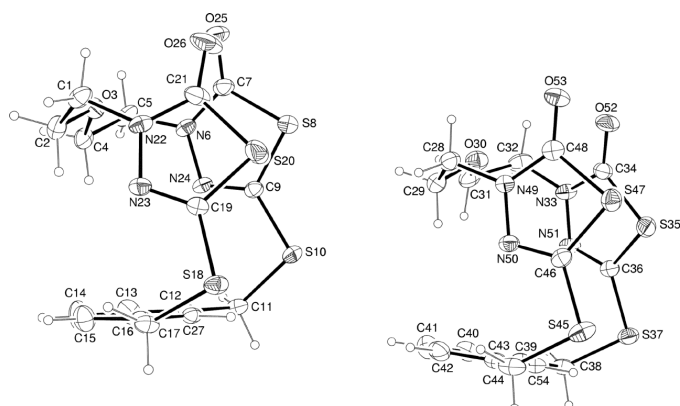


There are two molecules of (I) in the asymmetric unit. The S–C distances in the two molecules range from 1.720 (5) to 1.815 (5) Å, with a mean value of 1.766 (1) Å. The distances O25–C7 1.216 (5), O26–C21 1.207 (5), O52–C34 = 1.218 (5) and O53–C48 = 1.203 (5) Å clearly show double-bond character. These values are comparable with those found in 4,17,25,26-tetraaza-6,9,12,15-tetraoxa-2,19,21,24-tetrathiatricyclo[18.4.1<sup>1,4</sup>.1<sup>17,20</sup>]hexacos-1(25),20(26)-diene-3,5,16,18-tetraone (Cho *et al.*, 2000). The C–S–C angles in the five-membered rings range from 88.7 (2) to 89.9 (2)°, with a mean value of 89.1 (1)°, which is similar to the value of 90.7 (1)° found in 2,3-dihydro-3-methyl-2-nitrimino-1,3-thiazole (Kyziol *et al.*, 2000). All the five-membered rings are planar within 0.012 (3) Å, the two benzene rings are planar within 0.016 (3) Å and the two ethyl ether linkages are planar within

Received 13 March 2001

Accepted 22 March 2001

Online 31 March 2001



**Figure 1**  
View of the two molecules (I) in the asymmetric unit, showing 20% probability displacement ellipsoids. C atoms are shown with ellipsoids with enveloping ellipses only and the remaining atoms are shown with ellipsoids with octant shading. H atoms have been omitted for clarity.

0.107 (3) Å. In the first molecule (Fig. 1), the dihedral angle between the five-membered rings N6/C7/S8/C9/N24 and C19/S20/C21/N22/N23 is 30.4 (2)°, with distances N6···N22 = 4.640 (5) Å, N23···N24 = 5.080 (5) Å, S10···S18 = 5.197 (2) Å and S8···S20 = 3.957 (2) Å, so that the inner parts of the two rings approach each other. The dihedral angle between benzene ring C12/C13/C14/C15/C16/C27 and ethyl ether linkage C1/C2/O3/C4/C5 is 37.35 (19)°, and the inner parts of the two rings also approach each other, with a C4···C14 distance 4.125 (8) Å. In the second molecule (Fig. 1), the dihedral angle between the five-membered rings N33/C34/C35/C36/N51 and C46/S47/C48/N49/N50 is 30.5 (1)°, with distances N33···N49 = 4.561 (6) Å, N50···N51 = 5.015 (5) Å, S37···S45 = 5.111 (2) Å and S35···S47 = 3.812 (2) Å, and the dihedral angle between benzene ring C39/C40/C41/C42/C43/C54 and ethyl ether linkage C28/C29/O30/C31/C32 is 36.9 (2)°, the inner parts of the two rings approaching each other with a C29···C41 distance of 4.125 (8) Å. Therefore, both molecules have a cage-like form with similar conformations and each molecule appears to have sufficient space inside it to introduce a guest atom into the molecule. The closest intermolecular distance, 2.403 Å, is for O26···H32A(1 - x, 1/2 + y, 1/2 - z).

## Experimental

The preparation of (I) followed the general procedure previously reported by Cho, Park, Hwang *et al.* (1999). The details will be reported elsewhere.

### Crystal data

C<sub>16</sub>H<sub>16</sub>N<sub>4</sub>O<sub>3</sub>S<sub>4</sub>  
M<sub>r</sub> = 440.57  
Monoclinic, P2<sub>1</sub>/c  
a = 16.827 (2) Å  
b = 14.1071 (16) Å  
c = 16.760 (4) Å  
β = 93.950 (16)°  
V = 3969.0 (12) Å<sup>3</sup>  
Z = 8

D<sub>x</sub> = 1.475 Mg m<sup>-3</sup>  
Mo Kα radiation  
Cell parameters from 25 reflections  
θ = 11.3–12.7°  
μ = 0.50 mm<sup>-1</sup>  
T = 293 (2) K  
Plate, light brown  
0.50 × 0.43 × 0.13 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer  
Non-profiled ω/2θ scans  
Absorption correction: ψ scan (North *et al.*, 1968)  
T<sub>min</sub> = 0.779, T<sub>max</sub> = 0.936  
7087 measured reflections  
6845 independent reflections  
3946 reflections with I > 2σ(I)

R<sub>int</sub> = 0.028  
θ<sub>max</sub> = 25.0°  
h = 0 → 19  
k = 0 → 16  
l = -19 → 19  
3 standard reflections  
frequency: 300 min  
intensity decay: none

### Refinement

Refinement on F<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.061  
wR(F<sup>2</sup>) = 0.142  
S = 1.02  
6845 reflections  
481 parameters  
H-atom parameters constrained

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0505P)<sup>2</sup> + 1.3810P]  
where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> < 0.001  
Δρ<sub>max</sub> = 0.95 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.27 e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

C7—O25	1.216 (5)	C34—O52	1.218 (5)
C7—S8	1.785 (5)	C34—S35	1.773 (5)
S8—C9	1.738 (5)	S35—C36	1.720 (5)
C9—S10	1.746 (5)	C36—S37	1.736 (5)
S10—C11	1.815 (5)	S37—C38	1.803 (4)
C17—S18	1.802 (5)	C44—S45	1.811 (5)
S18—C19	1.747 (5)	S45—C46	1.743 (5)
C19—S20	1.723 (5)	C46—S47	1.731 (5)
S20—C21	1.790 (5)	S47—C48	1.788 (5)
C21—O26	1.207 (5)	C48—O53	1.203 (5)
C9—S8—C7	88.7 (2)	C36—S35—C34	89.9 (2)
C19—S20—C21	89.0 (2)	C46—S47—C48	88.8 (2)

All H atoms were placed in calculated positions [C—H<sub>methylene</sub> = 0.97 Å and C—H<sub>aromatic</sub> = 0.93 Å], with U<sub>iso</sub> constrained to be 1.2 times the equivalent isotropic displacement parameter of the attached C atom. The highest peak and the deepest hole in the final difference density map are at 0.81 Å from H40 and 0.96 Å from S35, respectively.

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994); cell refinement: CAD-4 EXPRESS; data reduction: XCAD4 (Harms & Wocadlo, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

## References

- Cho, N. S., Hong, S. I., Kim, J.-G. & Suh, I.-H. (2000). *Acta Cryst.* **C56**, 229–230.  
 Cho, N. S., Park, C. K., Hwang, H. J., Hong, S. I., Park, J. K. & Suh, I.-H. (1999). *J. Chem. Res. (S)*, pp. 730–731.  
 Cho, N. S., Park, C. K., Kim, H. S., Oh, J. G., Suh, I.-H. & Oh, M. R. (1999). *Heterocycles*, **51**, 2739–2746.  
 Enraf–Nonius (1994). CAD-4 EXPRESS. Enraf–Nonius, Delft, The Netherlands.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Farrugia, L. J. (1999). WinGX. University of Glasgow, Scotland.  
 Harms, K. & Wocadlo, S. (1995). XCAD4. University of Marburg, Germany.  
 Kyziol, J., Daszkiewicz, Z. & Zaleski, J. (2000). *Acta Cryst.* **C56**, 1401–1403.  
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.