Crystal structure of 3,6,9,12,20,23,26,29-octaoxa-36,49-dithia-51,52-diaza-hexacyclo[16.16.16.1^{38,42}.1^{43,47}.0^{14,50}.0^{31,35}]dopentaconta-1(35),14,16,18(50),31,33,38,40,42(52),43,45,47(51)-dodecaene, C₄₀H₄₈N₂O₈S₂, a 2,2'-bipyridine-bridged macrocyclic polyether

T. Nabeshima^{*I}, E. Horn^{II}, T. Aoki^{III} and Y. Yano^{III}

^I University of Tsukuba, Department of Chemistry, Tsukuba, Ibaraki 305-8571, Japan

^{II} Rikkyo University, Department of Chemistry, Nishi-Ikebukuro, Tokyo 171-8501, Japan, and University of Tsukuba, Tsukuba Advanced Research Alliance, Tsukuba, Ibaraki 305-8577, Japan

^{III} Gunma University, Department of Chemistry, Kiryu, Gunma 376-8515, Japan

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Discussion

Macrocyclic polyethers containing a thiol or a sulfide group have been studied due to their high Ag⁺ selectivity [2–4]. Recently we have reported the novel and remarkably Ag⁺ selective macrocycle which is bridged by a 2,2'-bipyridine moiety via sulfide linkages [1]. In the crystal structure, the polyether molecule (ORTEP plot; 30% probability ellipsoid equal to $-x, -y, 1-z$ with respect to the corresponding unique atoms at x, y, z) has a center of inversion located at the center of the C(1)—C(1') bond. The planar 2,2'-bipyridine spans a distance of 10.2 Å (S(1)···S(1')) across the middle of the polyether. The average S—C bond length is 1.805 Å, the bond angle C(6)—S(1)—C(7) is 101.0(1)°, the torsion angle C(5)—C(6)—S(6)—C(7) is $-64.5(3)^\circ$. All other bonds and angles are normal.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.4 × 0.6 × 0.8 mm
Wavelength:	Mo K α radiation (0.7107 Å)
μ :	1.94 cm ⁻¹
Diffractometer, scan mode:	Rigaku CAD4, $\omega/2\theta$
$2\theta_{\max}$:	53°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	4374, 4091
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2626
$N(\text{param})_{\text{refined}}$:	307
Programs:	teXsan [5], DIFABS [6]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.103(4)	0.183(3)	0.493(2)	0.0597
H(2)	4e	0.228(5)	0.217(3)	0.403(2)	0.0699
H(3)	4e	0.243(4)	0.064(3)	0.327(2)	0.0635
H(4)	4e	0.154(4)	-0.234(3)	0.361(2)	0.0562
H(5)	4e	0.225(4)	-0.155(3)	0.310(2)	0.0562
H(6)	4e	-0.345(4)	-0.418(3)	0.397(2)	0.0599
H(7)	4e	-0.519(4)	-0.253(3)	0.424(2)	0.0638
H(8)	4e	-0.477(4)	-0.069(3)	0.383(1)	0.0558
H(9)	4e	-0.294(4)	0.001(3)	0.255(2)	0.0572
H(10)	4e	-0.167(4)	0.044(3)	0.321(2)	0.0572
H(12)	4e	-0.435(5)	0.182(3)	0.243(2)	0.0766
H(14)	4e	-0.486(5)	0.360(4)	0.288(2)	0.0894

Abstract

C₄₀H₄₈N₂O₈S₂, monoclinic, $P12_1/c1$ (No. 14), $a = 8.283(1)$ Å, $b = 11.310(2)$ Å, $c = 20.669(7)$ Å, $\beta = 99.11(1)^\circ$, $V = 1911.9$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.049$, $wR(F) = 0.045$, $T = 298$ K.

Source of material

The synthesis of the 2,2'-bipyridine-bridged macrocyclic polyether has been reported in [1]. Purification was performed by the recrystallization from a mixture of acetone and n-hexane; mp 443 K.

* Correspondence author

(e-mail: nabesima@staff.chem.tsukuba.ac.jp)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(15)	4e	-0.429(5)	0.227(3)	0.444(2)	0.0763
H(16)	4e	-0.272(5)	0.201(3)	0.420(2)	0.0763
H(17)	4e	-0.151(4)	0.390(3)	0.467(2)	0.0642
H(18)	4e	-0.172(4)	0.285(3)	0.518(2)	0.0642
H(19)	4e	-0.178(5)	0.556(3)	0.513(2)	0.0689
H(20)	4e	-0.329(5)	0.565(3)	0.548(2)	0.0689

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(21)	4e	-0.219(5)	0.455(3)	0.637(2)	0.0703
H(22)	4e	-0.117(5)	0.572(3)	0.628(2)	0.0703
H(23)	4e	0.151(4)	0.494(3)	0.667(2)	0.0621
H(24)	4e	0.033(4)	0.409(3)	0.700(2)	0.0621
H(25)	4e	-0.277(5)	0.230(3)	0.303(2)	0.0805
H(26)	4e	-0.558(5)	0.249(3)	0.346(2)	0.0805

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4e	-0.0356(1)	-0.17763(8)	0.27228(4)	0.0484(5)	0.0551(5)	0.0391(4)	-0.0015(5)	0.0113(3)	-0.0039(4)
O(1)	4e	-0.3952(3)	0.0846(2)	0.3206(1)	0.064(2)	0.043(1)	0.059(1)	0.011(1)	0.019(1)	0.008(1)
O(2)	4e	-0.3739(4)	0.3419(2)	0.3774(1)	0.101(2)	0.061(2)	0.055(2)	0.015(2)	-0.013(2)	-0.001(1)
O(3)	4e	-0.3299(3)	0.4158(2)	0.5126(1)	0.045(1)	0.056(1)	0.066(2)	0.004(1)	0.009(1)	-0.015(1)
O(4)	4e	-0.0211(3)	0.4259(2)	0.6037(1)	0.054(1)	0.054(1)	0.046(1)	0.016(1)	-0.006(1)	-0.003(1)
N(1)	4e	0.0593(3)	-0.0768(2)	0.4313(1)	0.034(1)	0.036(1)	0.045(1)	0.001(1)	0.004(1)	-0.004(1)
C(1)	4e	0.0440(4)	0.0130(3)	0.4720(1)	0.032(2)	0.036(2)	0.045(2)	0.001(1)	0.003(1)	-0.003(1)
C(2)	4e	0.1077(5)	0.1248(3)	0.4636(2)	0.054(2)	0.036(2)	0.061(2)	-0.002(2)	0.015(2)	-0.011(2)
C(3)	4e	0.1854(5)	0.1434(3)	0.4105(2)	0.064(3)	0.040(2)	0.077(3)	-0.011(2)	0.028(2)	-0.002(2)
C(4)	4e	0.1967(4)	0.0531(3)	0.3669(2)	0.051(2)	0.051(2)	0.059(2)	-0.006(2)	0.020(2)	-0.003(2)
C(5)	4e	0.1339(4)	-0.0567(3)	0.3794(1)	0.030(2)	0.041(2)	0.047(2)	0.001(1)	0.004(1)	-0.004(1)
C(6)	4e	0.1437(4)	-0.1609(3)	0.3354(2)	0.038(2)	0.052(2)	0.051(2)	0.002(2)	0.011(2)	-0.009(2)
C(7)	4e	-0.1906(4)	-0.2068(3)	0.3212(1)	0.035(2)	0.042(2)	0.033(1)	-0.003(1)	-0.001(1)	0.000(1)
C(8)	4e	-0.2101(4)	-0.3198(3)	0.3460(1)	0.042(2)	0.043(2)	0.038(2)	-0.003(2)	-0.005(1)	0.000(1)
C(9)	4e	-0.3337(4)	-0.3390(3)	0.3830(2)	0.048(2)	0.048(2)	0.051(2)	-0.008(2)	-0.002(2)	0.014(2)
C(10)	4e	-0.4331(4)	-0.2485(3)	0.3961(2)	0.039(2)	0.065(3)	0.054(2)	-0.006(2)	0.005(2)	0.014(2)
C(11)	4e	-0.4144(4)	-0.1356(3)	0.3717(2)	0.035(2)	0.055(2)	0.050(2)	0.001(2)	0.007(2)	0.003(2)
C(12)	4e	-0.2949(4)	-0.1151(3)	0.3329(1)	0.035(2)	0.043(2)	0.037(2)	-0.005(1)	0.000(1)	0.002(1)
C(13)	4e	-0.2771(5)	0.0060(3)	0.3039(2)	0.046(2)	0.041(2)	0.056(2)	0.001(2)	0.009(2)	0.003(2)
C(14)	4e	-0.3883(6)	0.1958(3)	0.2886(2)	0.095(3)	0.042(2)	0.052(2)	0.010(2)	0.010(2)	0.010(2)
C(15)	4e	-0.4629(6)	0.2909(4)	0.3225(2)	0.105(4)	0.050(2)	0.057(2)	0.004(2)	0.000(2)	0.001(2)
C(16)	4e	-0.3236(6)	0.2678(3)	0.4324(2)	0.083(3)	0.045(2)	0.057(2)	0.016(2)	-0.002(2)	-0.005(2)
C(17)	4e	-0.2251(5)	0.3395(3)	0.4840(2)	0.056(2)	0.052(2)	0.052(2)	0.012(2)	0.004(2)	-0.004(2)
C(18)	4e	-0.2539(5)	0.5182(3)	0.5424(2)	0.059(3)	0.044(2)	0.064(2)	0.015(2)	-0.006(2)	-0.005(2)
C(19)	4e	-0.1584(5)	0.4978(4)	0.6092(2)	0.064(3)	0.049(2)	0.058(2)	0.019(2)	-0.001(2)	-0.007(2)
C(20)	4e	0.0961(5)	0.4206(3)	0.6624(2)	0.062(2)	0.037(2)	0.049(2)	0.005(2)	-0.007(2)	-0.002(2)

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