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## Structure Reports

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# 1,4,10,13-Tetraoxa-7,16-diazoniacyclo-octadecane bis[tetrachloridoaurate(III)] dihydrate

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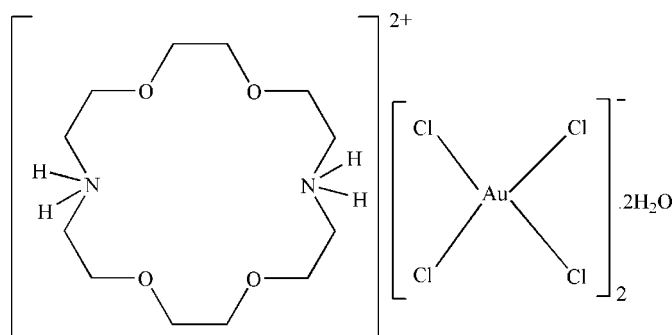
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.018;  $wR$  factor = 0.046; data-to-parameter ratio = 16.6.

The asymmetric unit of the title compound,  $(\text{C}_{12}\text{H}_{28}\text{N}_2\text{O}_4)\text{[AuCl}_4\text{]}_2 \cdot 2\text{H}_2\text{O}$ , contains one half-cation, one anion and one water molecule; the cation is centrosymmetric. The Au ion has a square-planar coordination. In the crystal structure, intramolecular  $\text{N}-\text{H} \cdots \text{O}$  and  $\text{O}-\text{H} \cdots \text{O}$ , and intermolecular  $\text{N}-\text{H} \cdots \text{O}$ ,  $\text{O}-\text{H} \cdots \text{Cl}$  and  $\text{N}-\text{H} \cdots \text{Cl}$  hydrogen bonds link the ions and water molecules, forming a supramolecular structure.

## Related literature

For related literature, see: Calleja *et al.* (2001); Chekhlov (2000, 2001, 2005); Chekhlov & Martynov (1998); Chekhlov *et al.* (1994); Fonari *et al.* (2004); Hasan *et al.* (1999); Johnson & Steed (1998); Moers *et al.* (2000); Simonov *et al.* (2003); Yap *et al.* (1995); Yousefi, Amani & Khavasi (2007); Yousefi, Teimouri *et al.* (2007); Zhang *et al.* (2006).



## Experimental

### Crystal data

$(\text{C}_{12}\text{H}_{28}\text{N}_2\text{O}_4)\text{[AuCl}_4\text{]}_2 \cdot 2\text{H}_2\text{O}$

$M_r = 977.94$

Triclinic,  $P\bar{1}$

$a = 8.0168$  (10) Å

$b = 8.3359$  (9) Å

$c = 11.2989$  (15) Å

$\alpha = 73.063$  (11)°

$\beta = 75.965$  (10)°

$\gamma = 74.929$  (9)°  
 $V = 686.02$  (15) Å<sup>3</sup>  
 $Z = 1$   
 Mo  $K\alpha$  radiation

$\mu = 11.49$  mm<sup>-1</sup>  
 $T = 120$  (2) K  
 $0.32 \times 0.22 \times 0.20$  mm

### Data collection

Stoe IPDSII diffractometer  
 Absorption correction: numerical  
 ( $X\text{-SHAPE}$  and  $X\text{-RED}$ ; Stoe & Cie, 2005)  
 $T_{\min} = 0.065$ ,  $T_{\max} = 0.108$

4188 measured reflections  
 2390 independent reflections  
 2381 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$   
 $wR(F^2) = 0.045$   
 $S = 1.18$   
 2390 reflections  
 144 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.59$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.67$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Cl1—Au1	2.2796 (11)	Cl3—Au1	2.2912 (11)
Cl2—Au1	2.2877 (10)	Cl4—Au1	2.2751 (11)
Cl4—Au1—Cl1	90.20 (4)	Cl4—Au1—Cl3	90.30 (4)
Cl4—Au1—Cl2	176.52 (4)	Cl1—Au1—Cl3	176.79 (3)
Cl1—Au1—Cl2	89.96 (4)	Cl2—Au1—Cl3	89.74 (4)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1C $\cdots$ O1 <sup>i</sup>	0.90	2.49	2.791 (5)	100
N1—H1C $\cdots$ O3	0.90	1.98	2.844 (3)	160
N1—H1D $\cdots$ Cl1 <sup>ii</sup>	0.90	2.81	3.540 (4)	139
N1—H1D $\cdots$ Cl2 <sup>ii</sup>	0.90	2.49	3.262 (3)	143
O3—H3C $\cdots$ O1	0.76 (6)	2.14 (6)	2.858 (4)	158 (6)
O3—H3C $\cdots$ O2	0.76 (6)	2.51 (6)	3.057 (3)	130 (5)
O3—H3D $\cdots$ Cl3 <sup>iii</sup>	0.81 (7)	2.59 (6)	3.378 (4)	167.00

Symmetry codes: (i)  $-x + 1, -y + 1, -z$ ; (ii)  $-x + 1, -y + 1, -z + 1$ ; (iii)  $x, y - 1, z$ .

Data collection:  $X\text{-AREA}$  (Stoe & Cie, 2005); cell refinement:  $X\text{-AREA}$ ; data reduction:  $X\text{-RED}$  (Stoe & Cie, 2005); program(s) used to solve structure:  $SHELXS97$  (Sheldrick, 2008); program(s) used to refine structure:  $SHELXL97$  (Sheldrick, 2008); molecular graphics:  $ORTEP-3$  for Windows (Farrugia, 1997); software used to prepare material for publication:  $WinGX$  (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2465).

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**supplementary materials**

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## 1,4,10,13-Tetraoxa-7,16-diazoniacyclooctadecane bis[tetrachloridoaurate(III)] dihydrate

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### Comment

Recently, we reported the synthesis and crystal structure of the  $[(\text{H}_2\text{DA18C6})\text{Cl}_2]$ , (II), (Yousefi, Amani & Khavasi, 2007) and  $[(\text{H}_2\text{DA18C6})][\text{PtCl}_6]\cdot 2\text{H}_2\text{O}$ , (III), (Yousefi, Teimouri *et al.*, 2007) [where  $\text{H}_2\text{DA18C6}$  is 1,10-Diazonia-18-crown-6]. Several proton transfer systems using 1,10-diaza-18-crown-6, with proton donor molecules, such as  $[(\text{H}_2\text{DA18C6})\text{I}_2\cdot 2\text{H}_2\text{O}]$ , (IV), (Chekhlov, 2005),  $[(\text{H}_2\text{DA18C6})(\text{C}_2\text{HO}_4)_2]$ , (V), and  $[(\text{H}_2\text{DA18C6})_2(\text{C}_2\text{O}_4)_2\cdot 2\text{H}_2\text{O}]$ , (VI), (Chekhlov, 2000),  $[(\text{H}_2\text{DA18C6})(\text{picrate})_2]$ , (VII), (Chekhlov, 2001),  $[(\text{H}_2\text{DA18C6})(\text{HPTD})_2]$ , (VIII), (Simonov *et al.*, 2003),  $[(\text{H}_2\text{DA18C6})(\text{PD})_2\cdot (\text{H}_2\text{O})_4]$ , (IX), and  $[(\text{H}_2\text{DA18C6})(\text{PS})_2\cdot (\text{H}_2\text{O})_2]$ , (X), (Fonari *et al.*, 2004),  $[(\text{H}_2\text{DA18C6})(\text{CCl}_3\text{COO})_2(\text{CCl}_3\text{COOH})_2]$ , (XI), (Chekhlov *et al.*, 1994),  $[(\text{H}_2\text{DA18C6})(\text{CCl}_3\text{COO})_2]$ , (XII), (Chekhlov & Martynov, 1998), and  $\{[\text{H}_2\text{DA18C6}][(\text{ArSO}_2)_2\text{N}]_2\}$ , (XIII), (Moers *et al.*, 2000) [where  $\text{C}_2\text{O}_4$  is oxalate, HPTD is (4Z,5E)-pyrimidine-2,4,5,6(1*H*,3*H*)-tetraone 4,5-dioxime anion, PD is 2-(2-methylphenyl)-2*H*-[1,2,3]-triazolo[4,5-*d*]pyrimidine-5,7(4*H*,6*H*)-dione 3-oxide anion, PS is 6-amino-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylsulfamate and  $(\text{ArSO}_2)_2\text{N}$  is bis(4-chlorobenzenesulfonyl)imide] have been synthesized and characterized by single-crystal X-ray diffraction methods.

There are also several proton transfer systems using  $\text{HAuCl}_4$  with proton acceptor molecules, such as  $[\text{EMI}][\text{AuCl}_4]$  (XIV) and  $[\text{BMI}]_2[\text{AuCl}_4]\cdot 2\text{H}_2\text{O}$ , (XV), (Hasan *et al.*, 1999),  $[\text{H}_2\text{bipy}][\text{AuCl}_4][\text{Cl}]$ , (XVI), (Zhang *et al.*, 2006),  $[\text{H}_7\text{O}_3][15\text{-crown-5}][\text{AuCl}_4]$ , (XVII) and  $[\text{H}_5\text{O}_2][\text{benzo-15-crown-5}]_2[\text{AuCl}_4]$ , (XVIII), (Johnson & Steed, 1998),  $[\text{H}_5\text{O}_2]_2[12\text{-crown-4}]_2[\text{AuCl}_4]_2$ , (XIX),  $[\text{H}_3\text{O}][18\text{-crown-6}][\text{AuCl}_4]$ , (XX) and  $[\text{H}_3\text{O}][4\text{-nitrobenzo-18-crown-6}][\text{AuCl}_4]$ , (XXI), (Calleja *et al.*, 2001) and  $[\text{DPPy.H}][\text{AuCl}_4]$ , (XXII), (Yap *et al.*, 1995) [where EMI is 1-ethyl-3-methylimidazolium, BMI is 1-butyl-3-methylimidazolium,  $\text{H}_2\text{bipy}$  is 2,2'-bipyridinium and DPPy.H is 2,6-Diphenylpyridinium] have been synthesized and characterized by single-crystal X-ray diffraction methods. We report herein the synthesis and crystal structure of the title compound, (I).

The asymmetric unit of (I), (Fig. 1) contains one half-cation, one anion and one water molecule; the cation is centrosymmetric. The Au ion has a square-planar coordination (Table 1). The bond lengths and angles, in cation, are in good agreement with the corresponding values in (II), (III) and (IV). Also, the Au-Cl bond lengths and angles (Table 1) are within normal range [XXII].

In the crystal structure, intramolecular N-H $\cdots$ O and O-H $\cdots$ O and intermolecular N-H $\cdots$ O, O-H $\cdots$ Cl and N-H $\cdots$ Cl hydrogen bonds (Table 2) link the molecules to form a supramolecular structure (Fig. 2), in which they may be effective in the stabilization of the structure.

### Experimental

For the preparation of the title compound, (I), a solution of 1,10-diaza-18-crown-6 (0.10 g, 0.37 mmol) in EtOH (20 ml) was added to a solution of  $\text{HAuCl}_4\cdot 3\text{H}_2\text{O}$ , (0.29 g, 0.74 mmol) in water (30 ml) and the resulting yellow solution was stirred

## supplementary materials

for 10 min at 313 K. Then, it was left to evaporate slowly at room temperature. After one week, yellow prismatic crystals of (I) were isolated (yield; 0.26 g; 72.0%).

### Refinement

H atoms (for H<sub>2</sub>O) were located in a difference syntheses and refined [O-H = 0.71 (6) and 0.76 (6) Å;  $U_{\text{iso}}(\text{H}) = 0.019$  (15) and 0.034 (17) Å<sup>2</sup>]. The remaining H atoms were positioned geometrically, with N-H = 0.90 Å (for NH<sub>2</sub>) and C-H = 0.97 Å for methylene H and constrained to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C},\text{N})$ .

### Figures

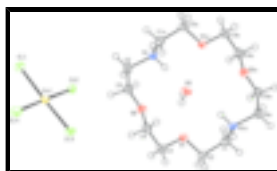


Fig. 1. The asymmetric unit of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry code: (a) 1 - x, 1 - y, -z].

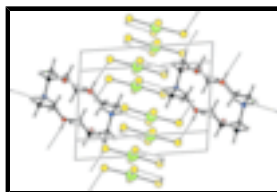


Fig. 2. A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

### 1,4,10,13-Tetraoxa-7,16-diazoniacyclooctadecane bis[tetrachloridoaurate(III)] dihydrate

#### Crystal data

(C<sub>12</sub>H<sub>28</sub>N<sub>2</sub>O<sub>4</sub>)[AuCl<sub>4</sub>]<sub>2</sub>·2H<sub>2</sub>O

$M_r = 977.94$

Triclinic, *PT*

Hall symbol: -P 1

$a = 8.0168$  (10) Å

$b = 8.3359$  (9) Å

$c = 11.2989$  (15) Å

$\alpha = 73.063$  (11)°

$\beta = 75.965$  (10)°

$\gamma = 74.929$  (9)°

$V = 686.02$  (15) Å<sup>3</sup>

$Z = 1$

$F_{000} = 460$

$D_x = 2.367$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 1139 reflections

$\theta = 1.9$ – $25.2$ °

$\mu = 11.49$  mm<sup>-1</sup>

$T = 120$  (2) K

Block, yellow

$0.32 \times 0.22 \times 0.20$  mm

#### Data collection

Stoe IPDSII  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

2390 independent reflections

2381 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$



## supplementary materials

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H3D	0.689 (8)	0.264 (8)	0.118 (5)	0.034 (17)*
N1	0.3824 (4)	0.5189 (4)	0.2598 (3)	0.0193 (7)
H1C	0.4424	0.4823	0.1909	0.023*
H1D	0.3672	0.4259	0.3230	0.023*
C1	0.7938 (5)	0.3778 (5)	-0.2343 (4)	0.0231 (9)
H1A	0.7787	0.2856	-0.1594	0.028*
H1B	0.8544	0.3272	-0.3043	0.028*
C2	0.9019 (5)	0.4874 (5)	-0.2162 (4)	0.0228 (9)
H2A	1.0152	0.4194	-0.1983	0.027*
H2B	0.9210	0.5783	-0.2913	0.027*
C3	0.8743 (5)	0.6921 (5)	-0.0984 (4)	0.0232 (9)
H3A	0.9062	0.7677	-0.1798	0.028*
H3B	0.9779	0.6447	-0.0597	0.028*
C4	0.7321 (6)	0.7883 (5)	-0.0161 (4)	0.0259 (9)
H4A	0.7733	0.8800	-0.0025	0.031*
H4B	0.6303	0.8385	-0.0566	0.031*
C5	0.5532 (5)	0.7528 (5)	0.1851 (4)	0.0242 (9)
H5A	0.4568	0.8215	0.1431	0.029*
H5B	0.5998	0.8274	0.2147	0.029*
C6	0.4895 (5)	0.6163 (5)	0.2940 (4)	0.0232 (9)
H6A	0.4190	0.6689	0.3610	0.028*
H6B	0.5900	0.5367	0.3257	0.028*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Au1	0.01758 (11)	0.01715 (11)	0.02133 (12)	-0.00433 (7)	-0.00015 (7)	-0.00455 (7)
Cl1	0.0296 (5)	0.0268 (5)	0.0227 (6)	-0.0041 (4)	-0.0003 (4)	-0.0077 (4)
Cl2	0.0334 (6)	0.0204 (5)	0.0217 (5)	-0.0106 (4)	-0.0006 (4)	-0.0055 (4)
Cl3	0.0271 (5)	0.0229 (5)	0.0236 (5)	-0.0074 (4)	0.0019 (4)	-0.0037 (4)
Cl4	0.0433 (7)	0.0205 (5)	0.0411 (7)	-0.0105 (5)	0.0085 (5)	-0.0118 (5)
O1	0.0223 (14)	0.0256 (15)	0.0229 (16)	-0.0110 (12)	0.0022 (12)	-0.0083 (12)
O2	0.0249 (15)	0.0196 (14)	0.0227 (16)	-0.0063 (12)	-0.0022 (12)	-0.0039 (12)
O3	0.0223 (17)	0.0229 (17)	0.0312 (19)	-0.0092 (16)	0.0008 (15)	-0.0049 (14)
N1	0.0197 (17)	0.0206 (17)	0.0162 (17)	-0.0061 (13)	0.0016 (14)	-0.0046 (14)
C1	0.020 (2)	0.023 (2)	0.027 (2)	-0.0009 (16)	-0.0022 (17)	-0.0108 (18)
C2	0.018 (2)	0.027 (2)	0.022 (2)	-0.0043 (17)	0.0019 (17)	-0.0077 (18)
C3	0.023 (2)	0.024 (2)	0.025 (2)	-0.0102 (17)	-0.0032 (17)	-0.0057 (18)
C4	0.032 (2)	0.019 (2)	0.027 (2)	-0.0099 (18)	-0.0062 (19)	-0.0021 (17)
C5	0.025 (2)	0.021 (2)	0.029 (2)	-0.0057 (17)	-0.0029 (18)	-0.0100 (18)
C6	0.022 (2)	0.026 (2)	0.026 (2)	-0.0046 (17)	-0.0044 (17)	-0.0123 (18)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Cl1—Au1	2.2796 (11)	C2—H2B	0.9700
Cl2—Au1	2.2877 (10)	C3—O1	1.432 (5)
Cl3—Au1	2.2912 (11)	C3—C4	1.500 (6)
Cl4—Au1	2.2751 (11)	C3—H3A	0.9700
O3—H3C	0.71 (6)	C3—H3B	0.9700

O3—H3D	0.76 (6)	C4—O2	1.419 (5)
N1—C1 <sup>i</sup>	1.496 (5)	C4—H4A	0.9700
N1—H1C	0.9000	C4—H4B	0.9700
N1—H1D	0.9000	C5—O2	1.412 (5)
C1—C2	1.495 (6)	C5—C6	1.501 (6)
C1—N1 <sup>i</sup>	1.496 (5)	C5—H5A	0.9700
C1—H1A	0.9700	C5—H5B	0.9700
C1—H1B	0.9700	C6—N1	1.501 (5)
C2—O1	1.429 (5)	C6—H6A	0.9700
C2—H2A	0.9700	C6—H6B	0.9700
Cl4—Au1—Cl1	90.20 (4)	H2A—C2—H2B	108.6
Cl4—Au1—Cl2	176.52 (4)	O1—C3—C4	106.8 (3)
Cl1—Au1—Cl2	89.96 (4)	O1—C3—H3A	110.4
Cl4—Au1—Cl3	90.30 (4)	C4—C3—H3A	110.4
Cl1—Au1—Cl3	176.79 (3)	O1—C3—H3B	110.4
Cl2—Au1—Cl3	89.74 (4)	C4—C3—H3B	110.4
C2—O1—C3	113.3 (3)	H3A—C3—H3B	108.6
C5—O2—C4	112.8 (3)	O2—C4—C3	108.8 (3)
H3C—O3—H3D	103 (6)	O2—C4—H4A	109.9
C1 <sup>i</sup> —N1—C6	113.5 (3)	C3—C4—H4A	109.9
C1 <sup>i</sup> —N1—H1C	108.9	O2—C4—H4B	109.9
C6—N1—H1C	108.9	C3—C4—H4B	109.9
C1 <sup>i</sup> —N1—H1D	108.9	H4A—C4—H4B	108.3
C6—N1—H1D	108.9	O2—C5—C6	108.5 (3)
H1C—N1—H1D	107.7	O2—C5—H5A	110.0
C2—C1—N1 <sup>i</sup>	110.8 (3)	C6—C5—H5A	110.0
C2—C1—H1A	109.5	O2—C5—H5B	110.0
N1 <sup>i</sup> —C1—H1A	109.5	C6—C5—H5B	110.0
C2—C1—H1B	109.5	H5A—C5—H5B	108.4
N1 <sup>i</sup> —C1—H1B	109.5	C5—C6—N1	112.9 (3)
H1A—C1—H1B	108.1	C5—C6—H6A	109.0
O1—C2—C1	106.6 (3)	N1—C6—H6A	109.0
O1—C2—H2A	110.4	C5—C6—H6B	109.0
C1—C2—H2A	110.4	N1—C6—H6B	109.0
O1—C2—H2B	110.4	H6A—C6—H6B	107.8
C1—C2—H2B	110.4		
N1 <sup>i</sup> —C1—C2—O1	59.1 (4)	C1—C2—O1—C3	-168.3 (3)
O1—C3—C4—O2	58.5 (4)	C4—C3—O1—C2	162.2 (3)
O2—C5—C6—N1	-71.6 (4)	C6—C5—O2—C4	169.1 (3)
C5—C6—N1—C1 <sup>i</sup>	-70.3 (4)	C3—C4—O2—C5	179.4 (3)

Symmetry codes: (i)  $-x+1, -y+1, -z$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1C $\cdots$ O1 <sup>i</sup>	0.90	2.49	2.791 (5)	100

## supplementary materials

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N1—H1C···O3	0.90	1.98	2.844 (3)	160
N1—H1D···C11 <sup>ii</sup>	0.90	2.81	3.540 (4)	139
N1—H1D···C12 <sup>ii</sup>	0.90	2.49	3.262 (3)	143
O3—H3C···O1	0.76 (6)	2.14 (6)	2.858 (4)	158 (6)
O3—H3C···O2	0.76 (6)	2.51 (6)	3.057 (3)	130 (5)
O3—H3D···C13 <sup>iii</sup>	0.81 (7)	2.59 (6)	3.378 (4)	167.00

Symmetry codes: (i)  $-x+1, -y+1, -z$ ; (ii)  $-x+1, -y+1, -z+1$ ; (iii)  $x, y-1, z$ .

Fig. 1

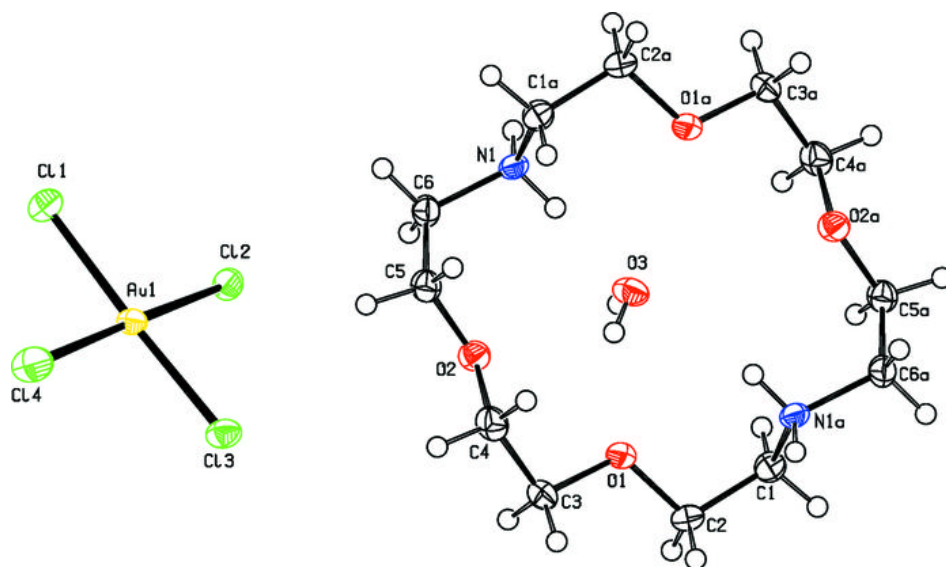


Fig. 2

