



Open Archive Toulouse Archive Ouverte (OATAO)

OATAO is an open access repository that collects the work of Toulouse researchers and makes it freely available over the web where possible.

This is an author-deposited version published in: <http://oatao.univ-toulouse.fr/>
Eprints ID : 2498

To link to this article :

URL : <http://dx.doi.org/10.1107/S1600536807003935>

To cite this version : Le Gal, Julien and Benoist, Eric and Gressier , Marie and Bélanger-Gariépy, Francine and Beauchamp, André L. (2007) [*Sodium \[2-\(mercaptomethylcarbonylamino\)-N-\(2-mercaptophenyl\)ethanamide\(4-\)-\[kappa\]4S,N,N',S'\]oxorhenate\(V\) monohydrate*](#). Acta Crystallographica Section E, vol. 63 . m647-m649. ISSN 1600-5368

Any correspondence concerning this service should be sent to the repository administrator: staff-oatao@inp-toulouse.fr

Sodium [2-(mercaptomethylcarbonylamino)-*N*-(2-mercaptophenyl)ethanamide(4-)- $\kappa^4 S, N, N', S'$]oxorhenate(V) monohydrate

Julien Le Gal,^a Éric Benoist,^a
Marie Gressier,^a Francine
Bélanger-Gariépy^b and André L.
Beauchamp^{b*}

^aLaboratoire de Chimie Inorganique, Université Paul Sabatier, 118 route de Narbonne, 31062 Toulouse, France, and ^bDépartement de Chimie, Université de Montréal, CP 6128, Succ. Centre-ville, Montréal, Québec, Canada H3C 3J7

Correspondence e-mail:
andre.beauchamp@umontreal.ca

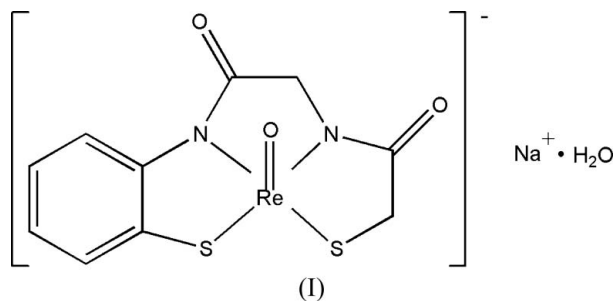
Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.012$ Å
 R factor = 0.044
 wR factor = 0.117
Data-to-parameter ratio = 13.4

In the complex anion of the title compound, $\text{Na}[\text{Re}(\text{C}_{10}\text{H}_8\text{N}_2\text{O}_2\text{S}_2)\text{O}]\cdot\text{H}_2\text{O}$, the Re atom adopts a square-pyramidal coordination, in which the $\text{Re}=\text{O}$ bond is apical and the S, N, N', S' -tetradentate ligand spans the four basal sites. The Na^+ counter-ion is octahedrally surrounded by one S and five O atoms.

Comment

As part of our ongoing research on semi-rigid ligands for the development of efficient Tc and Re radiopharmaceuticals (Le Gal *et al.*, 2004, 2005; Allali *et al.*, 2006), the title oxorhenium(V) compound, (I), was prepared and characterized by spectroscopic methods. The present crystallographic study confirms that it contains the complex anion shown in Fig. 1.



The environment of the metal is square pyramidal. The apical position is occupied by the oxo ligand, and the basal sites are filled by the S, N, N', S' -tetradentate ligand.

Selected distances and angles are listed in Table 1. The description in terms of a square pyramid is supported by the nearly equal $\text{N}4-\text{Re}-\text{S}10$ and $\text{N}7-\text{Re}-\text{S}1$ angles, leading to a very low trigonality index τ of 0.03 (Addison *et al.*, 1984). The Re atom lies 0.709 (2) Å above the basal plane, which is reflected by the $\text{O}=\text{Re}-\text{N}/\text{S}$ angles ranging from 108.2 (2) to

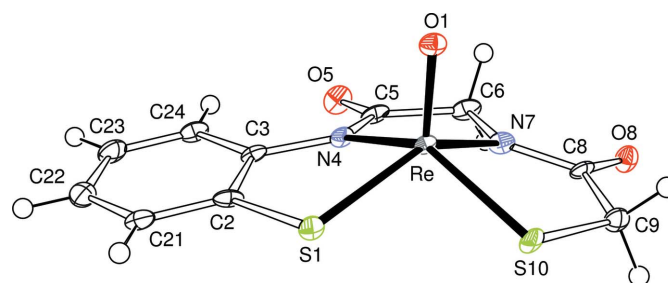


Figure 1

The structure of the complex anion. Displacement ellipsoids are drawn at the 30% probability level.

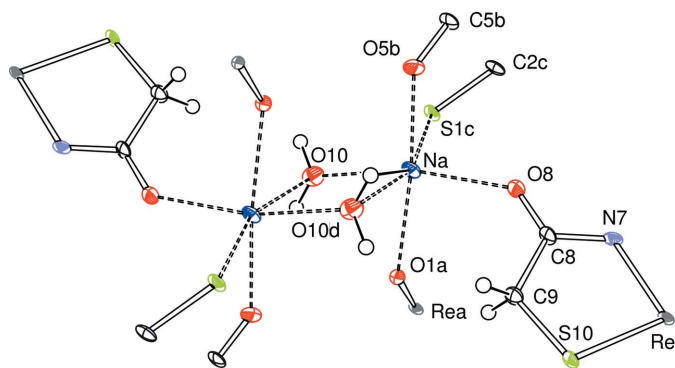


Figure 2

The environment of the Na^+ counter-ion [symmetry codes: (a) $1-x, 1-y, 1-z$; (b) $1-x, 2-y, 1-z$; (c) $x, y, z-1$; (d) $-x, 1-y, -z$ (also for unlabeled atoms)]. Displacement ellipsoids are drawn at the 30% probability level.

$111.2(2)^\circ$. The $\text{Re}=\text{O}$ bond length is normal for a mono-oxo complex. The $\text{Re}-\text{S}$ bond lengths are similar to those found in related compounds (Rao *et al.*, 1990; O'Neil *et al.*, 1994; Bell *et al.*, 1998; Hansen, Lipowska *et al.*, 1999; Hansen, Xu *et al.*, 1999; Friebe *et al.*, 2001; Lipowska *et al.*, 2002). The $\text{Re}-\text{N}$ bond lengths correspond to those observed for deprotonated amide N atoms (Bell *et al.*, 1998; Rao *et al.*, 1990).

Each Na^+ ion is surrounded octahedrally by one S and five O atoms, as shown in Fig. 2. A pair of Na^+ ions across the inversion center at $(0, 1/2, 0)$ is bridged by a pair of symmetry-equivalent water molecules. Approximately coplanar with this Na_2O_2 ring is found another $\text{Na}\cdots\text{O}$ contact with the amide atom O8. These units are assembled into a layer *via* long $\text{Na}\cdots\text{S}$ contacts. Cohesion between these layers involves $\text{Na}\cdots\text{O}$ contacts with an oxo ligand and with the other amide O5 atom. $\text{O}-\text{H}\cdots\text{S}$ hydrogen bonds (Table 2) involving the water molecule also contribute to the stability of the crystal structure.

Experimental

The compound was prepared as described earlier (Le Gal *et al.*, 2004). Crystals were obtained by slow evaporation of a solution in a 1:1 mixture of dichloromethane and methanol.

Crystal data

$\text{Na}[\text{Re}(\text{C}_{10}\text{H}_8\text{N}_2\text{O}_2\text{S}_2)\text{O}]\cdot\text{H}_2\text{O}$	$V = 663.02(2) \text{ \AA}^3$
$M_r = 495.51$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 2.482 \text{ Mg m}^{-3}$
$a = 7.9488(1) \text{ \AA}$	Cu $K\alpha$ radiation
$b = 9.5623(1) \text{ \AA}$	$\mu = 21.34 \text{ mm}^{-1}$
$c = 10.0320(1) \text{ \AA}$	$T = 100(2) \text{ K}$
$\alpha = 103.111(1)^\circ$	Block, red
$\beta = 111.146(1)^\circ$	$0.30 \times 0.12 \times 0.09 \text{ mm}$
$\gamma = 100.426(1)^\circ$	

Data collection

Bruker SMART 2K/Platform diffractometer	7982 measured reflections
ω scans	2524 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2504 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.043, T_{\max} = 0.147$	$R_{\text{int}} = 0.040$
	$\theta_{\text{max}} = 72.7^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.117$
 $S = 1.08$
 2524 reflections
 189 parameters
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0833P)^2 + 6.3732P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.004$
 $\Delta\rho_{\text{max}} = 6.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -2.90 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

$\text{Re}-\text{O1}$	1.706 (5)	$\text{Na}-\text{O5}^{\text{i}}$	2.261 (6)
$\text{Re}-\text{N7}$	1.984 (6)	$\text{Na}-\text{O10}^{\text{ii}}$	2.349 (6)
$\text{Re}-\text{N4}$	2.034 (6)	$\text{Na}-\text{O1}^{\text{iii}}$	2.448 (6)
$\text{Re}-\text{S10}$	2.2858 (17)	$\text{Na}-\text{O10}$	2.466 (7)
$\text{Re}-\text{S1}$	2.2969 (18)	$\text{Na}-\text{S1}^{\text{iv}}$	3.198 (3)
$\text{Na}-\text{O8}$	2.326 (6)		
$\text{O1}-\text{Re}-\text{N7}$	109.7 (3)	$\text{O5}^{\text{i}}-\text{Na}-\text{O1}^{\text{iii}}$	174.3 (2)
$\text{O1}-\text{Re}-\text{N4}$	111.2 (2)	$\text{O8}-\text{Na}-\text{O1}^{\text{iii}}$	91.3 (2)
$\text{N7}-\text{Re}-\text{N4}$	78.9 (3)	$\text{O10}^{\text{ii}}-\text{Na}-\text{O1}^{\text{iii}}$	80.2 (2)
$\text{O1}-\text{Re}-\text{S10}$	108.25 (18)	$\text{O5}^{\text{i}}-\text{Na}-\text{O10}$	102.0 (2)
$\text{N7}-\text{Re}-\text{S10}$	82.89 (19)	$\text{O8}-\text{Na}-\text{O10}$	160.5 (2)
$\text{N4}-\text{Re}-\text{S10}$	140.10 (18)	$\text{O10}^{\text{ii}}-\text{Na}-\text{O10}$	84.0 (2)
$\text{O1}-\text{Re}-\text{S1}$	108.18 (19)	$\text{O1}^{\text{iii}}-\text{Na}-\text{O10}$	72.7 (2)
$\text{N7}-\text{Re}-\text{S1}$	141.62 (19)	$\text{O5}^{\text{i}}-\text{Na}-\text{S1}^{\text{iv}}$	81.59 (17)
$\text{N4}-\text{Re}-\text{S1}$	81.94 (18)	$\text{O8}-\text{Na}-\text{S1}^{\text{iv}}$	98.47 (17)
$\text{S10}-\text{Re}-\text{S1}$	91.22 (6)	$\text{O10}^{\text{ii}}-\text{Na}-\text{S1}^{\text{iv}}$	157.0 (2)
$\text{O5}^{\text{i}}-\text{Na}-\text{O8}$	94.3 (2)	$\text{O1}^{\text{iii}}-\text{Na}-\text{S1}^{\text{iv}}$	98.61 (16)
$\text{O5}^{\text{i}}-\text{Na}-\text{O10}^{\text{ii}}$	97.3 (2)	$\text{O10}-\text{Na}-\text{S1}^{\text{iv}}$	73.76 (16)
$\text{O8}-\text{Na}-\text{O10}^{\text{ii}}$	104.6 (2)		

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x, -y+1, -z$; (iii) $-x+1, -y+1, -z+1$; (iv) $x, y, z-1$.

Table 2

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O10}-\text{H10A}\cdots\text{S10}^{\text{iv}}$	0.94 (10)	2.46 (10)	3.242 (6)	140 (8)
$\text{O10}-\text{H10B}\cdots\text{S1}^{\text{iii}}$	0.93 (15)	2.98 (15)	3.499 (6)	117 (11)

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

The H atoms of the organic ligand were positioned geometrically ($\text{C}-\text{H} = 0.95\text{--}0.99 \text{ \AA}$) and were included in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The water H atoms were refined freely. The highest residual electron density peak is located 0.97 \AA from the Re atom and the deepest hole is located 0.79 \AA also from the Re atom.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: UdmX (Maris, 2004).

We are grateful to the Natural Sciences and Engineering Research Council of Canada and the French Ministère de l'Éducation Nationale, de la Recherche et de la Technologie for financial support.

References

Addison, A. W., Rao, T. N., Reedijk, J., van Rijn, J. & Verschoor, G. C. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1349–1356.

- Allali, A., Cousinié, S., Gressier, M., Tessier, C., Beauchamp, A. L., Coulais, Y., Dartiguenave, M. & Benoist, É. (2006). *Inorg. Chim. Acta*, **359**, 2128–2134.
- Bell, R. A., McCarry, B. E. & Valliant, J. F. (1998). *Inorg. Chem.* **37**, 3517–3520.
- Bruker (1997). *SHELXTL*. Release 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (1999). *SAINT* (Release 6.06) and *SMART* (Release 5.059). Bruker AXS Inc., Madison, Wisconsin, USA.
- Friebe, M., Mahmood, A., Bolzati, C., Drews, A., Johannsen, B., Eisenhut, M., Kraemer, D., Davison, A. & Jones, A. G. (2001). *J. Med. Chem.* **44**, 3132–3140.
- Hansen, L., Lipowska, M., Meléndez, E., Xu, X., Hirota, S., Taylor, A. T. & Marzilli, L. G. (1999). *Inorg. Chem.* **38**, 5351–5358.
- Hansen, L., Xu, X., Lipowska, M., Taylor, T. Jr & Marzilli, L. G. (1999). *Inorg. Chem.* **38**, 2890–2897.
- Le Gal, J., Latapie, L., Gressier, M., Coulais, Y., Dartiguenave, M. & Benoist, É. (2004). *Org. Biomol. Chem.* **2**, 876–883.
- Le Gal, J., Tisato, F., Bandoli, G., Gressier, M., Jaud, J., Michaud, S., Dartiguenave, M. & Benoist, É. (2005). *Dalton Trans.* pp. 3800–3807.
- Lipowska, M., Hansen, L., Cini, R., Xu, X., Choi, H., Taylor, A. T. & Marzilli, L. G. (2002). *Inorg. Chim. Acta*, **339**, 327–340.
- Maris, T. (2004). *UdMX*. Université de Montréal, Canada.
- O’Neil, J. P., Wilson, S. R. & Katzenellenbogen, J. A. (1994). *Inorg. Chem.* **33**, 319–323.
- Rao, T. N., Adhikesavalu, D., Camerman, A. & Fritzberg, A. G. (1990). *J. Am. Chem. Soc.* **112**, 5798–5804.
- Sheldrick, G. M. (1996). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.