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# Tetraphenylphosphonium [2-(mercaptomethyl-carbonylamino)-*N*-(2-methylaminophenyl)-ethanamido(4-)- $\kappa^4$ S,N,N',N'']oxorhenate(V)

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## Key indicators

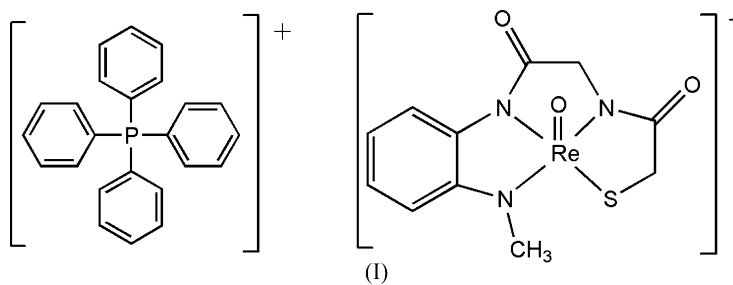
Single-crystal X-ray study  
 $T = 100$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å  
 $R$  factor = 0.035  
 $wR$  factor = 0.094  
Data-to-parameter ratio = 15.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $(\text{C}_{24}\text{H}_{20}\text{P})[\text{Re}(\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2\text{S})\text{O}]$ , is the  $[(\text{C}_6\text{H}_5)_4\text{P}]^+$  salt of a square-pyramidal  $[\text{ReOL}]^-$  complex anion [ $L$  is the deprotonated 2-(mercaptomethylcarbonylamino)- $N$ -(2-methylaminophenyl)ethanamide ligand], in which the  $\text{Re}=\text{O}$  bond is apical and the tetradentate ( $\text{N}-\text{N}-\text{N}-\text{S}$ )<sup>4-</sup> ligand fills the four basal sites. The presence of a methyl group on the terminal N-atom donor induces a small displacement of the coordinated S atom.

## Comment

The title compound, (I), and the corresponding non-methylated complex described in the previous paper (Le Gal *et al.*, 2007), were prepared as part of our ongoing project on the development of technetium and rhenium compounds for eventual application in nuclear medicine (Le Gal *et al.*, 2004, 2005). The present crystallographic study was undertaken to probe the influence of methylation of the terminal N donor on the geometry of the metal coordination environment.



The complex anion in (I), shown in Fig. 1, retains the square-pyramidal structure observed for the non-methylated compound. Selected distances and angles are listed in Table 1. Minor steric repulsion from the  $N$ -methyl substituent induces small changes in the  $\text{N}1-\text{Re}1-\text{S}10$  ( $+2.7^\circ$ ),  $\text{O}1-\text{Re}1-\text{S}10$  ( $-2.3^\circ$ ) and  $\text{N}4-\text{Re}1-\text{S}10$  ( $+2.8^\circ$ ) angles. The remaining angles around the Re centre differ by  $\leq 0.7^\circ$ . The  $\text{Re}1-\text{N}1$  distance [1.986 (3) Å] to the terminal deprotonated amine group is slightly greater than in the non-methylated compound [1.968 (2) Å]. This is likely due to the presence of the methyl substituent, since this terminal bond was found to be longer still (*ca* 2.020 Å) to mercaptoacetylglycylglycylaminobenzoic acids, where the  $N$ -aryl substituent is bulkier (Hansen *et al.*, 1992). In addition, the  $\text{Re}1-\text{S}10$  bond shows a significant, but small, difference [2.2999 (9) *versus* 2.2908 (7) Å], whereas the  $\text{Re}1-\text{N}7$  bond with one of the deprotonated amide donors is slightly shortened [1.992 (3) *versus* 2.013 (2) Å].

The distance from the Re atom to the basal  $\text{N}_3\text{S}$  plane is 0.7240 (15) Å and the square pyramid is regular, with the

O=Re–(N,S) angles lying in the narrow range 109.60 (14)–112.44 (13)°. The angles between the *trans* bonds are 136.76 (14) and 139.55 (8)°, corresponding to a very low trigonality index  $\tau$  of 0.05 (Addison *et al.*, 1984).

The [(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>P]<sup>+</sup> cation exhibits the expected tetrahedral geometry. Complex anions and [(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>P]<sup>+</sup> cations are packed in the unit cell with normal van der Waals contacts.

## Experimental

The title compound was prepared as described earlier (Le Gal *et al.*, 2005). Crystals were grown by slow evaporation of a solution of the compound in a 1:1 mixture of CH<sub>2</sub>Cl<sub>2</sub> and methanol.

### Crystal data

(C <sub>24</sub> H <sub>20</sub> P)[Re(C <sub>11</sub> H <sub>11</sub> N <sub>3</sub> O <sub>2</sub> S)O]	$V = 3144.62 (8) \text{ \AA}^3$
$M_r = 790.86$	$Z = 4$
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
$a = 13.7077 (2) \text{ \AA}$	$\mu = 8.99 \text{ mm}^{-1}$
$b = 12.5262 (2) \text{ \AA}$	$T = 100 (2) \text{ K}$
$c = 19.4526 (3) \text{ \AA}$	$0.50 \times 0.35 \times 0.22 \text{ mm}$
$\beta = 109.700 (1)^\circ$	

### Data collection

Bruker SMART 2K/PLATFORM diffractometer	20287 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	6047 independent reflections
$T_{\min} = 0.057$ , $T_{\max} = 0.138$	5633 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.045$

### Refinement

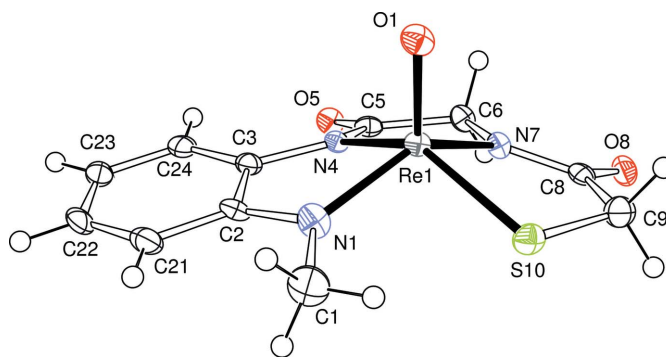
$R[F^2 > 2\sigma(F^2)] = 0.035$	398 parameters
$wR(F^2) = 0.094$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{\text{max}} = 1.67 \text{ e \AA}^{-3}$
6047 reflections	$\Delta\rho_{\text{min}} = -1.88 \text{ e \AA}^{-3}$

**Table 1**

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

Re1–O1	1.703 (3)	Re1–N4	2.011 (3)
Re1–N1	1.986 (3)	Re1–S10	2.2999 (9)
Re1–N7	1.992 (3)		
O1–Re1–N1	109.60 (14)	N4–Re1–N7	78.09 (11)
O1–Re1–N4	111.04 (11)	O1–Re1–S10	109.09 (9)
N1–Re1–N4	77.98 (13)	N1–Re1–S10	93.39 (10)
O1–Re1–N7	112.44 (13)	N4–Re1–S10	139.55 (8)
N1–Re1–N7	136.76 (14)	N7–Re1–S10	82.30 (8)

H atoms were positioned geometrically, with C–H = 0.95–0.99  $\text{\AA}$ , and were included in the refinement in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$ . The largest residual electron-



**Figure 1**

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

density peak and deepest hole are both located 0.81  $\text{\AA}$  from atom Re1. A final check for possible voids was performed using the VOID routine of *PLATON* (Spek, 2003).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; 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).

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