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The title compound, \(\text{[C}_{24}\text{H}_{20}\text{P}]\text{[Re(C}_{10}\text{H}_{9}\text{N}_{3}\text{O}_{2}\text{S})\text{O}]}\), contains well separated square-pyramidal \([\text{ORe}\text{L}]^-\) complex anions (\(\text{L}\) is the deprotonated \(\text{N-(2-aminophenyl)-2-(mercaptomethylcarbonylamino)ethanamide ligand}\)) and tetrahedral \([\text{[(C}_{6}\text{H}_{5})_{4}\text{P}]^+}\) cations. In the anion, the Re\(=\text{O}\) bond is oriented along the apical direction and the four basal sites are occupied by one S- and three N-atom donors of the tetradsentate \(\text{L}_4^\text{4-}\) ligand.

**Comment**

With the aim of developing specific technetium and rhenium compounds for eventual application in nuclear medicine, our research group has recently prepared a new family of semi-rigid tetradsentate ligands with \(\text{N}_2\text{S}_2\text{O}_2\text{N}_2\text{S}\) frameworks, and has studied their reactivity with \([\text{TcO}]^3^+\) and \([\text{ReO}]^3^+\) cores (Le Gal et al., 2004, 2005). In a previous paper (Le Gal et al., 2007), we described the structure of the Na\(^+\) salt of an oxorhenium(V) compound with the \(\text{N}_2\text{S}_2\) ligand \(\text{2-(mercaptomethylcarbonylamino)-N-(2-mercaptophenyl)-ethanamide}\). A related anionic complex was isolated as the title tetraphenylphosphonium salt, \((\text{I})\), with an \(\text{N}_3\text{S}\) ligand, where the thiol group bonded to the benzene ring has been replaced by an amine. The crystallographic study of this salt was undertaken to confirm the structure proposed from spectroscopic data.

A view of the complex anion of \((\text{I})\) is provided in Fig. 1. The Re atom adopts a square-pyramidal coordination geometry. The axial position is occupied by the oxo ligand, whereas the basal sites are filled by the tetradsentate (NNNS)\(^{4-}\) ligand. Selected distances and angles are listed in Table 1. The Re\(=\text{O}\) bond, the Re\(=\text{S}\) bond and the Re\(=\text{N}\) bonds to the deprotonated amide atoms N4 and N7 agree with those observed in complexes with the corresponding SNNS ligand (Le Gal et al., 2007) and other tetradsentate SNNS or NNNS ligands (Bell et al., 1998; Rao et al., 1990; Hansen et al., 1992; Wong et al., 1997; Lipowska, et al., 2002). The Re\(=\text{N}\) bond to the terminal deprotonated amine group N1 is significantly shorter than...
those observed in similar complexes with mercaptoacetylglucylglycylaminobenzoic acids (ca. 2.021 Å; Hansen et al., 1992); this may be ascribed to the presence of a large aryl substituent on this N donor in the latter case. The Re—N bond is ca. 0.18 Å shorter than those observed for non-deprotonated amine donors (Wong et al., 1997; Lipowska et al., 2002).

Since the angles between the trans bonds are equal [136.81 (10) and 136.79 (7)°], the trigonality index τ is 0.0 (Addison et al., 1984) and the environment of the metal is best described as square-pyramidal. This square pyramid is remarkably regular, the O-Re—(N,S) angles lying in the narrow range 110.27 (10)—111.82 (10)°. The distance from the Re atom to the basal N 3S plane is 0.7503 (12) Å.

The [(C6H5)4P]+ cation shows the expected tetrahedral geometry. Complex anions and [(C24H20P)Re(C10H9N3O2S)O]− cations are packed in the unit cell as well separated entities with normal van der Waals contacts. The terminal N1 donor lies 3.259 (3) Å from the Re—O O atom of an adjacent anion and weak N1—H···O1 hydrogen bonding may be present (geometric details are given in Table 2).

Experimental

Compound (I) was prepared as described earlier (Le Gal et al., 2005). Crystals were obtained by slow evaporation of a solution of the compound in a 1:1 CH2Cl2–methanol mixture. The authors 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.

Table 2

<table>
<thead>
<tr>
<th>Hydrogen-bond geometry (Å, °).</th>
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<tr>
<td>D—H···A</td>
</tr>
<tr>
<td>N1—H1···O1</td>
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</table>

Symmetry code: (i) x, −y + ½, z − ⅓.

H atoms were positioned geometrically, with C—H = 0.95–0.99 Å and N—H = 0.88 Å, and were included in the refinement in the riding-model approximation, with Uiso(H) = 1.2Ueq(parent). The maximum residual electron-density peak is located 0.88 Å from atom Re1 and the deepest hole is located 0.82 Å from the same atom. A final check for possible voids was performed using the VOID routine of PLATON (Spek, 2003).

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELX97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: UDX (Maris, 2004).

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References


Figure 1

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

Table 1

<table>
<thead>
<tr>
<th>Selected geometric parameters (Å, °).</th>
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<tbody>
<tr>
<td>Re1—O1</td>
</tr>
<tr>
<td>Re1—N1</td>
</tr>
<tr>
<td>Re1—N7</td>
</tr>
<tr>
<td>O1—Re1—N1</td>
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<td>O1—Re1—N4</td>
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