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Rhenium(I) and technetium(I) *fac*-M(NSO)(CO)$_3$ (M = Re, ^{99m}Tc) tricarbonyl complexes, with a tridentate NSO bifunctional agent: Synthesis, structural characterization, and radiochemistry

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ABSTRACT

The synthesis and structural characterization of the neutral rhenium complex *fac*-[Re(NSO)(CO)$_3$], **Re-1**, where (NSO) is a tridentate bifunctional chelating agent, 3-(carboxymethylthio)-3-(1*H*-imidazol-4-yl)propanoic acid (**1**), is presented. The complex crystallized from methanol–water and its structure was assigned by IR and ^{1}H, ^{13}C NMR spectroscopies and X-ray crystallography. Furthermore, the analogous technetium complex *fac*-[^{99m}Tc(NSO)(CO)$_3$], **^{99m}Tc-1**, was synthesized in high yield by reacting ligand **1** with the *fac*-[^{99m}Tc(OH$_2$)$_3$(CO)$_3$]$^+$ precursor for 30 min at 85 °C. The tracer complex was found to be more than 95% stable in the L-histidine challenge experiment. Our data indicate that the bifunctional NSO chelating agent **1** can be successfully applied for the development of potential ^{99m}Tc-radiopharmaceuticals.

1. Introduction

Technetium (^{99m}Tc) is the most significant radiometal in Nuclear Medicine and ^{99m}Tc-radiopharmaceuticals, routinely employed in Single Photon Emission Computerized Tomography (SPECT) diagnostic imaging [1–4]. Hexacoordinated ^{99m}Tc–tricarbonyl complexes of the *fac*-[^{99m}TcL(CO)$_3$] type are an attractive approach to the design of novel ^{99m}Tc-radiopharmaceuticals, because of their high stability and their convenient, high yield and high specific activity preparation in aqueous media [5]. Furthermore, the use of the beta-emitter ^{188}Re in therapeutic radiopharmaceuticals has been gaining considerable ground and the development of analogous to ^{99m}Tc complexes of the type *fac*-[^{188}ReL(CO)$_3$] could be employed for targeted radiotherapy. The ligand L should be preferably tridentate because it results in complexes with more favourable pharmacokinetics – compared to bidentate ones – and it may have a variety of donor atoms, like N, S, O [6]. The bifunctional strategy for the development of technetium and rhenium radiopharmaceuticals has become the most widely used method for producing well-defined technetium and rhenium labeled receptor ligands capable of highly specific in vivo localization in target tissues [7,8]. This strategy involves the development of a suitable bifunctional chelating agent (BFCA) which is used for the chelation of the radionuclide (^{99m}Tc or ^{188}Re) and the conjugation of the target-specific moiety. An ideal BFCA is that which is able to form stable and inert ^{99m}Tc or ^{188}Re complexes in high yield, at low concentration.

We have focused on the study of 3-(S-carboxymethylthio)-3-(1*H*-imidazol-4-yl)propanoic acid (**1**, Fig. 1), which is a natural metabolite of L-histidine [9], as a potential bifunctional chelating agent. This compound contains the N(π)-imidazolyl nitrogen, a thioether S, and a carboxylate O for charge-neutralizing purposes and can therefore act as a tridentate NSO ligand to generate neutral [M(NSO)(CO)$_3$] complexes with the *fac*-[M(CO)$_3$]$^+$ core (M = Re, ^{99m}Tc). Aromatic nitrogen atoms like the N(π)-pyridinyl or N(π)-imidazolyl are potent donors and a series of chelating agents used for the stabilization of the *fac*-[M(CO)$_3$]$^+$ core contain either of those [6,10–19]. Moreover, in addition to the NSO donor atom system, ligand **1** contains a second carboxylic group that can function as an anchor to conjugate amine containing biomolecules.

In the present work, we describe the synthesis and characterization of the products (Fig. 1) of the reaction of the [NEt$_4$]$_2$[Re(CO)$_3$Br$_3$] and [^{99m}Tc(CO)$_3$(H$_2$O)$_3$]$^+$ precursors with ligand **1**.

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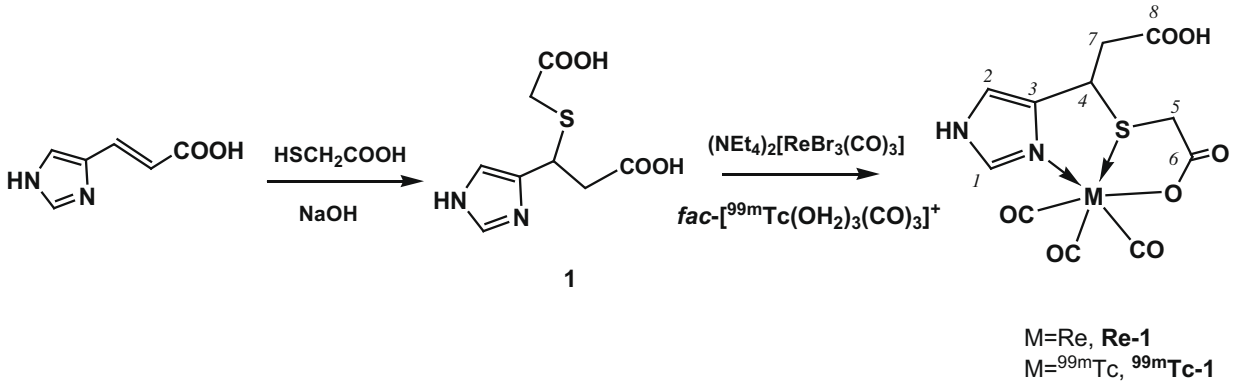

Fig. 1. Synthetic scheme of ligand **1** and **Re-1**, **^{99m}Tc-1** complexes.

2. Experimental

2.1. Materials and methods

All chemicals were reagent grade and were used as such unless otherwise noted. Rhenium was purchased from Aldrich as $Re_2(CO)_{10}$ and was converted to $(NEt_4)_2[ReBr_3(CO)_3]$ as previously reported [20].

For the ^{99m}Tc labeling a kit containing 5.5 mg $NaBH_4$, 4 mg Na_2CO_3 and 10 mg Na–K tartarate, was purged with CO gas prior to addition of $Na^{99m}TcO_4$, as described elsewhere [5].

Elemental analyses were performed on a Perkin–Elmer 2400 automated analyzer. IR spectra were recorded as KBr pellets on a Perkin–Elmer 1600 FT-IR spectrophotometer in the region 4000–500 cm^{-1}. ^{1}H NMR spectra were recorded on a Bruker 500 MHz Avance DRX spectrometer and are reported in Table 1. HPLC analysis was performed on a Waters 600 chromatography system coupled to both a Waters 2487 Dual λ Absorbance detector and a Gabi gamma detector from Raytest. Separations were achieved on a Nucleosil C18 (10 μm, 250 mm × 4 mm) column eluted with a binary gradient system at a 1 mL min^{-1} flow rate. Mobile phase A was methanol containing 0.1% trifluoroacetic acid and mobile phase B was water containing 0.1% trifluoroacetic acid. The elution profile was 0–1 min 100% B, followed by a linear gradient to 30% B in 10 min; this composition was held for 10 min more. After a column wash with 95% A for 5 min, the column was re-equilibrated by applying the initial conditions (100% B) for 15 min prior to the next injection.

2.2. Synthesis of 3-(S-carboxymethylthio)-3-(1H-imidazol-4-yl)propanoic acid (*1*)

Ligand **1** was synthesized by reaction of thioglycolic acid with *trans*-urocanic acid according to a literature procedure [9,21,22] with small modifications. *Trans*-urocanic acid (560 mg, 4 mmol) was dissolved in 40 mL methanol containing 2 mL NaOH 2 M. To this solution, thioglycolic acid (12 mmoL, 800 μL) was added and the mixture was refluxed overnight. The next day, the mixture was cooled and the pH was adjusted to 4 by addition of HCl 2 M. The mixture was subsequently condensed to afford a white solid which was recrystallized from a pH 4 aqueous solution. Yield: 460 mg (50%). ^{1}H NMR, δ (300 MHz, d_6-DMSO): 2.76–2.96 (m, 2H), 3.07–3.24 (m, 2H), 4.36 (tr, 1H), 6.98 (s, 1H), 7.65 (s, 1H). ^{13}C NMR, δ (300 MHz, d_6-DMSO): 32.71, 37.81, 39.15, 115.71, 135.2, 137.35, 171.44, 171.8. *Anal.* Calc. for $C_8H_{10}N_2O_4S$: C, 41.73; H, 4.38; N, 12.17. Found: C, 42.05; H, 4.68; N, 12.35%.

Table 1
^{1}H and ^{13}C NMR chemical shifts (ppm) for complex **Re-1** in DMSO-d_6 at 25 °C. The numbering of the atoms is shown in Fig. 1.

H-1	8.36	C-1	139.36
H-2	7.28	C-2	115.02
H-4	4.73	C-3	141.75
H-5	3.62, 3.42	C-4	46.13
H-7	2.82, 2.73	C-5	34.62
NH	13.17	C-6	177.80
		C-7	40.31
		C-8	170.50
		C≡O	196.00, 195.49, 193.75

2.3. Synthesis of fac-[Re(NSO)(CO)$_3$], *Re-1*

0.1 mmol (77 mg) of $(NEt_4)_2[ReBr_3(CO)_3]$ was reacted with 0.1 mmol (29 mg) 3-(carboxymethylthio)-3-(1*H*-imidazol-4-yl) propanoic acid in refluxing water, pH 6 for 3 h. The complex precipitated from water and was collected as a whitish solid. Crystals suitable for X-ray crystallography were obtained by slow evaporation from a methanol–water solution. Yield: 31 mg (62%), t_R (min) 15.60, IR (cm^{-1}, KBr): 2033, 1895, 1725, 1607, NMR data in Table 1, *Anal.* Calc. for $C_{11}H_9N_2O_7ReS$: C, 26.45; H, 1.82; N, 5.61. Found: C, 26.81; H, 2.05; N, 5.82%.

2.4. X-ray crystal structure determination of *Re-1*

Crystals of **Re-1** suitable for X-ray analysis were mounted in air on a Crystal Logic Dual Goniometer diffractometer using graphite monochromated Mo Kα radiation. Unit cell dimensions were determined by using the angular settings of 25 automatically centered reflections in the range $11 < 2\theta < 23°$ and they appear in Table 2. Intensity data were recorded using a θ–2θ scan. Three standard reflections monitored every 97 reflections showed less than 3% variation and no decay. Lorentz, polarization and psi-scan absorption corrections were applied using CRYSTAL LOGIC software. The structures were solved by direct methods using SHELXS-97 [23] and refined by full-matrix least squares techniques on F^2 using SHELXL-97 [24]. Further crystallographic details of **Re-1**: $2\theta_{max} = 50°$, scan speed 2.5°/min, scan range $1.6 + \alpha_1\alpha_2$ separation, reflections collected/unique/used 2535/2388 [$R_{int} = 0.0365$]/2388, 227 parameters refined, $[\Delta\rho]_{max}/[\Delta\rho]_{min} = 1.801/-1.131$ e/Å^3, $[\Delta/\sigma]_{max} = 0.002$, R_1/wR_2 (for all data) = 0.0353/0.0837. Hydrogen atoms were located by difference maps and were refined isotropically, except that of the imidazolic group which was introduced at calculated position as riding on bonded atom. All non-H atoms were refined anisotropically.

Table 2
Summary of crystal, intensity collection and refinement data.

	Re-1
Empirical formula	$C_{11}H_9N_2O_7ReS$
Formula weight	499.47
Temperature	298
Wavelength	Mo Kα 0.710730
Space group	$P2_1/c$
a (Å)	9.518(4)
b (Å)	10.390(4)
c (Å)	14.981(6)
β (°)	108.01(2)
V (Å^3)	1408.9(10)
Z	4
D_{calcd} (Mg m^{-3})	2.355
Absorption coefficient μ (mm^{-1})	8.810
F(0 0 0)	944
Goodness-of-fit (GOF) on F^2	1.077
R indices	R_1 = 0.0308[a]
	wR_2 = 0.0807[a]

[a] For 2152 reflections with $I > 2\sigma(I)$.

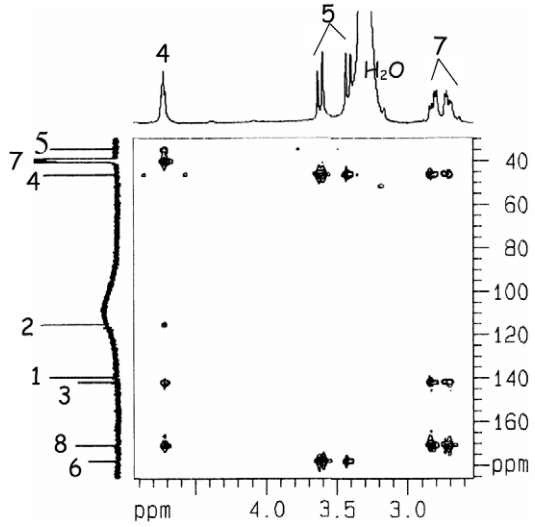

Fig. 2. ^{1}H–^{13}C long-range correlation spectrum (HMBC, range δ_H 4.9–2.5, range δ_C 185.9–29.1) of complex **Re-1** in DMSO-d$_6$ at 25 °C. The numbering of protons is shown in Fig. 1.

2.5. Synthesis of *fac*-[^{99m}Tc(NSO)(CO)$_3$], ^{99m}Tc-1

1 mL of an aqueous mixture of [^{99m}Tc(OH$_2$)$_3$(CO)$_3$]$^+$ (~0.5 GBq/mL) and 100 μg of **1** at pH 6.5 reacted at 85 °C for 30 min. The reaction mixture was analyzed by RP HPLC to estimate the yield and the complex was purified by isolating the radioactive peak from the column. t_R (min): 16.2. Yield > 90%.

Histidine challenge of ^{99m}**Tc-1***:* 0.2 mL of the reaction mixture of complex ^{99m}**Tc-1** was mixed with 0.8 mL solution of 1 mM histidine in 0.1 M potassium phosphate buffer solution, pH 7.4 and the mixture was incubated at 37 °C for 4 h. The mixture was analyzed by HPLC.

3. Results and discussion

3.1. Synthesis and characterization of complex *Re-1*

3-(Carboxymethylthio)-3-(1*H*-imidazol-4-yl)propanoic acid (**1**) provides two possible modes of complexation; the linear mode where the N(π)-imidazolyl nitrogen, the thioether S, and the carboxylate O of the thioglycolate moiety form the tridentate NSO donor atom system, and the tripodal mode, where the NSO system involves the coordination of the propionate carboxylate group instead (Fig. 1). However, HPLC analysis of the reaction mixture of ligand **1** with *fac*-(NEt$_4$)$_2$[ReBr$_3$(CO)$_3$] precursor showed the formation of a single product, under the conditions employed. The product was isolated as white crystalline solid and characterized by elemental analysis and spectroscopic methods. The NMR data confirmed the existence of a single product which was shown by X-ray crystallography to be the one with the linearly coordinated ligand (**Re-1** in Fig. 1). **Re-1** is soluble in DMSO, slightly soluble in methanol and ethanol and insoluble in water, and proved stable in the solid state and in solution for a period of months.

Infrared spectroscopy of the complex **Re-1** revealed the characteristic stretching bands of facially coordinated CO with ν(CO) at 2033 and 1895 cm^{-1}. Furthermore, the two bands at 1725 and 1607 cm^{-1} indicate the presence of one free carboxylate group and one coordinated, respectively.

^{1}H and ^{13}C chemical shift assignments for complex **Re-1** were based on ^{1}H–^{1}H and ^{1}H–^{13}C correlation spectra (Fig. 2) and are reported in Table 1. Upon coordination, downfield shifts (2–6 ppm) are noted for all carbons and protons (0.2–0.7 ppm) of **Re-1** compared to **1**, with the exception of the atoms of the free carboxyl chain that are affected to a lesser extend, as expected. The geminal protons on C-5 and C-7 are diastereotopic, due to the asymmetry of the complex, and appear at different chemical shifts.

3.2. Description of the structure

An ORTEP diagram of **Re-1** is given in Fig. 3a and selected bond distances and angles are listed in Table 3. The distorted octahedral environment of the Re atom in the structure of **Re-1** is defined by the three facially bound CO groups, and the NSO donor atom set of the tridentate ligand. The Re–carbonyl bond distances, 1.884(7)–1.927(8) Å, are consistent which those found in other Re–tricarbonyl complexes [25,26]. The Re–S and Re–O$_{carb}$ bond distances (2.479(2) and 2.162(4) Å, respectively) are consistent with those found in the analogous complex [Re(CO)$_3$L] (L = the monoanion of S-(carboxymethyl)-L-cysteine; Re–S = 2.469, 2.459 Å and Re–O$_{carb}$ = 2.148, 2.158 Å for the two crystallographically independent molecules) [26]. The Re–N$_{imid}$ bond distance (2.163(6) Å) in **Re-1** is significantly shorter than the Re–N$_{amine}$ bond length in [Re(CO)$_3$L] (2.244, 2.265 Å) [26] as expected for the sp^2 vs. sp^3 hybridization of the nitrogen atoms, respectively. There are two five-membered rings in the coordination sphere which are almost perpendicular to each other forming a dihedral angle of 83.3°. One of the five-membered rings is planar and is defined by the atoms Re, S, C6, C5, O5 (largest deviation ~0.08 Å for C6) and the second one, defined by the atoms Re, N1, C1, S, adopts the envelope configuration with C7 being 0.55 Å out of the best mean plane of the remaining four atoms. The protonated carboxylate entity of the tridentate ligand is hydrogen bonded to the uncoordinated oxygen atom of the second carboxylato moiety [O2···O4 (1 + x, 0.5 − y, 0.5 + z) = 2.592 Å, HO2···O4 = 1.438 Å, O2–HO2···O4 = 163.42°], and the imidazolic group is hydrogen bonded to the coordinated carboxylate oxygen atom O5 [N2···O5 (−x, 0.5 + y, 0.5 − z) = 2.769 Å, H2 N···O5 = 2.005 Å, N2–H2 N···O5 = 147.3°], thus forming a 2D network (Fig. 3b).

3.3. Radiochemistry

Complex *fac*-[^{99m}Tc(NSO)(CO)$_3$], ^{99m}**Tc-1** was synthesized in high yield (>90%) by the reaction of the *fac*-[^{99m}Tc(H$_2$O)$_3$(CO)$_3$]$^+$ precursor with ligand **1** at 85 °C for 30 min at pH 5–6. HPLC analysis of the reaction mixture showed the formation of a single product. The reaction yield was almost quantitative. The structure of

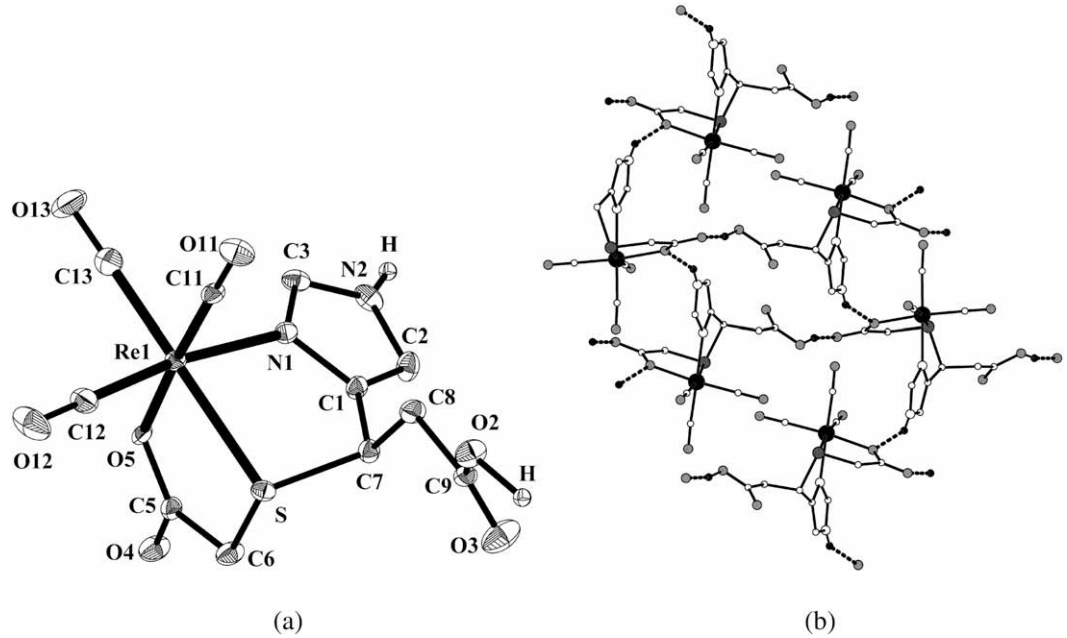

Fig. 3. (a) Labeled plot of the molecular structure of **Re-1**. (b) A small fragment of the 2D network in the structure of **Re-1** due to intermolecular hydrogen bonding interactions (dashed lines). Color code: Re, black; S, dark grey; O, medium grey; N, large white; C, small white; H, small black.

Table 3
Selected bond distances (Å) and angles (°) for **Re-1**.

Distances			
Re(1)–C(11)	1.886(7)	Re(1)–O(5)	2.162(4)
Re(1)–C(12)	1.911(8)	Re(1)–N(1)	2.163(6)
Re(1)–C(13)	1.928(8)	Re(1)–S	2.479(2)
Angles			
C(11)–Re(1)–C(12)	87.7(3)	C(13)–Re(1)–N(1)	96.6(3)
C(11)–Re(1)–C(13)	86.1(3)	O(5)–Re(1)–N(1)	80.4(2)
C(12)–Re(1)–C(13)	90.9(4)	C(11)–Re(1)–S	99.4(2)
C(11)–Re(1)–O(5)	175.2(2)	C(12)–Re(1)–S	92.9(3)
C(12)–Re(1)–O(5)	97.0(3)	C(13)–Re(1)–S	173.4(2)
C(13)–Re(1)–O(5)	94.4(3)	O(5)–Re(1)–S	79.8(1)
C(11)–Re(1)–N(1)	94.8(2)	N(1)–Re(1)–S	79.4(1)
C(12)–Re(1)–N(1)	172.2(3)		

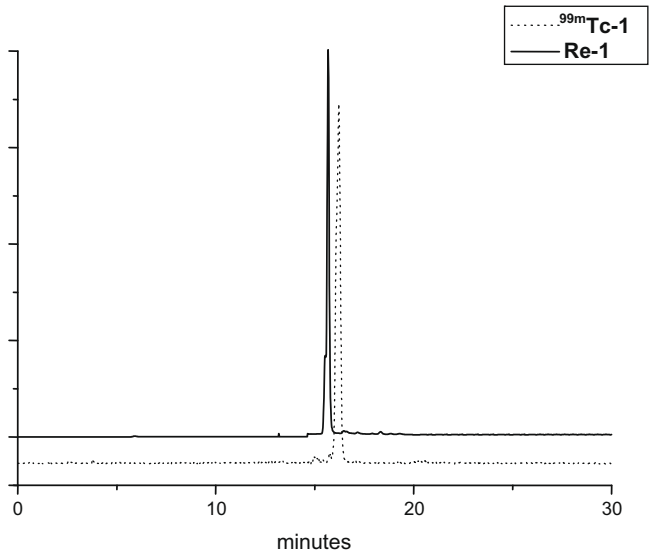

Fig. 4. Comparative high performance liquid chromatography (HPLC) chromatograms for complexes **Re-1** (carrier: photometric detection) and **^{99m}Tc-1** (tracer: radiometric detection).

the tracer complex **^{99m}Tc-1** was assigned by HPLC comparison of its retention time to that of the authentic well-characterized **Re-1**, by applying parallel radiometric and photometric detection. The retention times of the tracer complex **^{99m}Tc-1** and the **Re-1** were found to be t_R = 16.2 and 15.6 min, respectively (Fig. 4), providing a strong indication of their structural analogy. The tracer complex **^{99m}Tc-1** was about 100% stable for at least 4 h in the presence of 1 mM histidine in a 0.1 M potassium phosphate buffer at physiological pH 7.4 and 37 °C. This result indicates that the tracer complex will be stable against the endogenous histidine concentrations.

5. Conclusions

Ligand 3-(carboxymethylthio)-3-(1*H*-imidazol-4-yl)propanoic acid (**1**), coordinates in tridentate fashion with the rhenium(I)–tricarbonyl metal fragment with formation of a neutral [Re(NSO)$(CO)_3$] complex in high yield, leaving the propionate carboxylate group free. The formation of only one complex demonstrates that ligand **1**, coordinates preferably by the linear mode and formation of two five-membered chelate rings (Fig. 1). Apparently this type of coordination renders higher stability to the complex compared to formation of three rings (two five-membered and one seven-membered) expected from the non-linear mode, as also reported in the literature [26]. The analogous tracer complex *fac*-[^{99m}Tc(NSO)$(CO)_3$] is also formed in high yield and is stable in high histidine concentrations. The presence of a free carboxylate group in these metal chelates can be used to further anchor a bioactive moiety, e.g., via the formation of an amide bond, targeting thus specific biological substrates. Therefore, this bifunctional chelating agent can be applied for the development of target-specific ^{99m}Tc and $^{186/188}$Re radiopharmaceuticals for imaging and therapeutic applications, respectively.

Supplementary data

CCDC 738387 contains the supplementary crystallographic data for **Re-1**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge

Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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