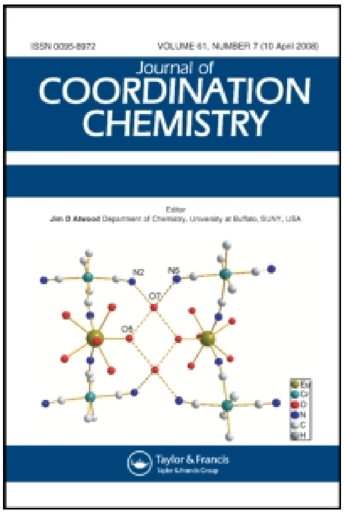

Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Synthesis and structure of oxorhenium(V) complexes containing a terdentate imidazole ligand. A route to mixed '3 + 2' complexes

Thomas I. A. Gerber[a]; Peter Mayer[b]; Zenixole R. Tshentu[a]
[a] Department of Chemistry, Nelson Mandela Metropolitan University, 6000 Port Elizabeth, South Africa [b] Department of Chemistry, Ludwig-Maximilians University, D-81377 München, Germany

To cite this Article Gerber, Thomas I. A. , Mayer, Peter and Tshentu, Zenixole R.(2005) 'Synthesis and structure of oxorhenium(V) complexes containing a terdentate imidazole ligand. A route to mixed '3 + 2' complexes', Journal of Coordination Chemistry, 58: 11, 947 — 953

To link to this Article: DOI: 10.1080/00958970500068917

URL: http://dx.doi.org/10.1080/00958970500068917

Journal of Coordination Chemistry
Vol. 58, No. 11, 20 July 2005, 947–953

Synthesis and structure of oxorhenium(V) complexes containing a terdentate imidazole ligand. A route to mixed '3 + 2' complexes

THOMAS I. A. GERBER*†, PETER MAYER‡ and ZENIXOLE R. TSHENTU†

†Department of Chemistry, Nelson Mandela Metropolitan University,
6000 Port Elizabeth, South Africa
‡Department of Chemistry, Ludwig-Maximilians University, D-81377 München, Germany

(*Received 30 November 2004; in final form 11 February 2005*)

The neutral mononuclear complex [ReOBr$_2$(ami)] (**1**) [Hami = 2-(1-ethanolaminomethyl)-1-methylimidazole] was prepared by reaction of (*n*-Bu$_4$N)[ReOBr$_4$(OPPh$_3$)] with an equimolar amount of Hami in acetonitrile. Further reaction of **1** with disodium oxalate (Na$_2$ox) in methanol yielded the mixed didentate–terdentate complex [ReO(ox)(ami)] (**2**). These compounds were characterized by spectroscopy and X-ray crystallography. Both complexes have distorted octahedral geometry with the alcoholate oxygen of ami$^-$ coordinated *trans* to the oxo group. In **1** the Br–Re–Br angle equals 89.91(3)° and in **2** the oxalate has a bite angle of 81.0(2)°.

Keywords: Oxorhenium(V); Crystal structure; Mixed didentate/terdentate

1. Introduction

In order to advance the coordination chemistry of rhenium(V) and to synthesize novel compounds of the metal for possible application as radiopharmaceuticals, we have become interested in using multidentate ligands containing the imidazole group as chelators. One approach to the development of stable rhenium(V) complexes has been the '3 + 1' concept of ligand permutation [1]. This concept is based on the ligation of a dinegative tridentate chelate in combination with a monodentate uninegative thiolate to produce a square-pyramidal oxorhenium(V) complex [2]. However, these '3 + 1' complexes were found to be relatively unstable *in vitro* and *in vivo* [3]. Kinetic stability was improved somewhat by '3 + 2' six-coordinated oxo-rhenium(V) complexes [4].

We have recently shown that, under an argon atmosphere, the reaction of the N,N-donor bidentate ligand 2-(1-ethylaminomethyl)-1-methylimidazole (emi) with

---

*Corresponding author. Email: thomas.gerber@upe.ac.za

Journal of Coordination Chemistry
ISSN 0095-8972 print: ISSN 1029-0389 online
DOI: 10.1080/00958970500068917

trans-[$ReOCl_3(PPh_3)_2$] led to the isolation of the cationic complex [ReOCl(OEt)(emi)(PPh_3)]$^+$ [5]. However, in air the oxo-bridged binuclear complex [(μ-O) {$ReOCl_2$(emi)}$_2$] was formed [6]. With the N,O-donor bidentate ligand 2-(hydroxymethyl)-1-methylimidazole (Hmi), the reaction led to the compound [$ReOCl_2$(mi) (PPh_3)] [7].

In this study, the two bidentate emi and Hmi ligands have been 'combined' in the N,N,O-donor terdentate 2-(1-ethanolaminomethyl)-1-methylimidazole (Hami), the reaction of which with (*n*-Bu_4N)[$ReOBr_4(OPPh_3)$] in air led to the isolation of the neutral monomer [$ReOBr_2$(ami)] (**1**). Further reaction of **1** with sodium oxalate (Na_2ox) in methanol gave the '3 + 2' complex [ReO(ox)(ami)] (**2**) in good yield.

2. Experimental

(*n*-Bu_4N)[$ReOBr_4(OPPh_3)$] was prepared by a literature procedure [8]. The ligand 2-(1-ethanolaminomethyl)-1-methylimidazole (Hami) was synthesized by reaction of 2-chloromethyl-1-methylimidazole and 2-aminoethanol [9]. Scientific instrumentation used is the same as reported elsewhere [5]. Infrared spectra were obtained using KBr disks and ^{1}H NMR spectra were run in d_6-DMSO.

2.1. *Synthesis*

2.1.1. [$ReOBr_2$(ami)] (1). (*n*-Bu_4N)[$ReOBr_4(OPPh_3)$] (100 mg, 96 µmol) was dissolved in 10 cm^3 of acetonitrile and an equimolar amount of Hami (15 mg) dissolved in 5 cm^3 of acetonitrile was added dropwise with stirring at room temperature. The color of the mixture changed from pink to green. The solution was then heated to reflux for 30 min, and after cooling to room temperature, a blue precipitate was collected by filtration. It was washed with water and acetone, and dried under vacuum. Slow evaporation of the filtrate at room temperature deposited blue platelets that were suitable for X-ray analysis. Yield 68% (33.7 mg); m.p. 281°C. Anal. Calc. for $C_7H_{12}N_3O_2Br_2Re$(%): C, 16.29; H, 2.34; N, 8.14. Found: C, 16.51; H, 2.42; N, 8.22. Infrared: ν(Re = O) 945(s); ν(N–H) 3220(m); ν(Re–N) 521(m), 571(m); ν(Re–O) 432(w). ^{1}H NMR: 9.42 (br s, 1H, NH); 7.83 (s, 1H, H^1); 7.79 (s, 1H, H^2); 4.63 (s, 2H, C^5H_2); 4.00 (s, 3H, NCH_3); 3.78 (t, 2H, C^7H_2); 3.21 (t, 2H, C^6H_2).

2.1.2. [ReO(ox)(ami)] (2). A mixture of 30 mg of **1** (58 µmol) and 31 mg of disodium oxalate (232 µmol) in 20 cm^3 of methanol was heated under reflux for 2 h. During this time the solution changed color from blue to purple. After cooling to room temperature, the solution was filtered and allowed to stand in a closed vessel. After 2 days dark purple crystals were obtained, and these were collected by filtration and dried in an oven at 50°C. Yield 74% (19 mg); m.p. 268°C. Anal. Calc. for $C_9H_{12}N_3O_6Re$ (%): C, 24.32; H, 2.72; N, 9.46. Found: C, 24.46; H, 2.83; N, 9.63. Infrared: ν(Re = O) 953(s); ν(N–H) 3167(m); ν(Re–N) 525(m), 575(s); ν(Re–O) 438(w), 459(w). ^{1}H NMR: 9.84 (br s, 1H, NH); 7.64 (s, 1H, H^1); 7.18 (s, 1H, H^2); 4.61 (s, 2H, C^5H_2); 4.15 (t, 2H, C^7H_2); 3.89 (s, 3H, NCH_3); 3.11 (t, 2H, C^6H_2).

2.2. *X-ray crystallography*

X-ray diffraction studies on crystals of **1** and **2** were performed at 200(2) K using a Nonius Kappa CCD diffractometer with graphite-monochromated Mo Kα

Table 1. Crystal data and structure refinement for **1** and **2**.

	1	**2**
Chemical formula	$C_7H_{12}N_3O_2Br_2Re$	$C_9H_{12}N_3O_6Re$
Formula weight	516.20	444.42
Temperature (K)	200(2)	200(2)
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/c$
Unit cell dimensions (Å,°)	$a = 7.1922(2)$	7.7176(2)
	$b = 7.2655(2)$	12.4945(3)
	$c = 12.2472(4)$	12.9546(4)
	$\alpha = 74.243(2)$	–
	$\beta = 86.472(1)$	97.734(1)
	$\gamma = 72.167(1)$	–
Volume (Å)	586.17(3)	1237.82(6)
Z	2	4
Density (calc.) (Mg/m^3)	2.925	2.385
Absorption coefficient (mm^{-1})	17.166	9.845
$F(000)$	472	840
Crystal size (mm)	$0.02 \times 0.13 \times 0.18$	$0.04 \times 0.05 \times 0.08$
θ range for data collection (°)	3.4–25.0	3.3–25.0
Index ranges	$-8 \leq h \leq 8$; $-8 \leq k \leq 8$; $-14 \leq l \leq 14$	$-9 \leq h \leq 9$; $-14 \leq k \leq 14$; $-15 \leq l \leq 15$
Reflections measured	6720	13 692
Independent/observed reflections	2030/1780	2182/1737
Data/restraints/parameters	2030/0/136	2182/0/172
Goodness-of-fit on F^2	1.07	1.05
Final R indices [$I > 2\sigma(I)$]	0.0359, $wR2 = 0.0883$	0.0296, $wR2 = 0.0523$
Largest diff. peak and hole (e/Å^3)	1.85, −3.06	1.08, −1.37

radiation ($\lambda = 0.71073$ Å). Further details are given in table 1, with selected bond lengths and angles in table 2. The structures were solved by direct methods. Nonhydrogen atoms were refined with anisotripic displacement parameters. Structural refinements were made by the full-matrix least-squares method on F^2 using the program SHELXL-97 [10].

3. Results and discussion

3.1. *Synthesis*

The more commonly used oxorhenium(V)-halide starting material (n-Bu_4N)[$ReOCl_4$] is moisture sensitive, so we have chosen (n-Bu_4N)[$ReOBr_4(OPPh_3)$] as the starting complex because of its stability and the ease of substitution of its bromide and $OPPh_3$ ligands. Its simple reaction with an equimolar amount of Hami in acetonitrile gave the blue complex [$ReOBr_2$(ami)] (**1**) as product. Further reaction of **1** with disodium oxalate (Na_2ox) in methanol gave as product the purple '3 + 2' complex [ReO(ox)(ami)] (**2**). Both complexes are diamagnetic (formally d^2) and are nonelectrolytes in DMF. They are weakly soluble, but stable, in polar solvents such as chloroform, DMSO and DMF.

3.2. *Spectroscopic characterization*

In the infrared spectra, the Re = O stretching frequencies of **1** and **2** appear as sharp strong bands at 945 and 953 cm^{-1}, respectively, which fall in the typical region of

Table 2. Selected bond lengths (Å) and angles (°) for **1** and **2**.

1		**2**	
Re–O(2)	1.698(6)	Re–O(6)	1.690(4)
Re–O(1)	1.907(5)	Re–O(1)	1.909(4)
Re–Br(1)	2.503(1)	Re–O(2)	2.034(4)
Re–Br(2)	2.596(1)	Re–O(4)	2.069(4)
Re–N(1)	2.082(7)	Re–N(1)	2.087(5)
Re–N(3)	2.199(7)	Re–N(3)	2.188(4)
O(1)–C(7)	1.43(1)	O(1)–C(7)	1.413(7)
C(1)–N(1)	1.39(1)	C(1)–N(1)	1.387(7)
C(3)–N(1)	1.31(1)	C(3)–N(1)	1.336(7)
C(1)–C(2)	1.32(1)	C(1)–C(2)	1.352(8)
C(4)–N(2)	1.46(1)	C(4)–N(2)	1.459(7)
C(3)–N(2)	1.34(1)	O(2)–C(8)	1.314(7)
C(2)–N(2)	1.39(1)	O(3)–C(8)	1.206(7)
C(3)–C(5)	1.49(1)	O(5)–C(9)	1.203(7)
C(6)–N(3)	1.49(1)	O(4)–C(9)	1.295(7)
O(2)–Re–O(1)	164.1(3)	O(6)–Re–O(1)	164.9(2)
Br(1)–Re–Br(2)	89.91(3)	O(2)–Re–O(4)	81.0(2)
N(1)–Re–N(3)	79.1(3)	N(1)–Re–N(3)	77.7(2)
O(1)–Re–N(3)	75.7(3)	O(1)–Re–N(3)	78.2(2)
N(1)–Re–Br(2)	173.4(2)	N(1)–Re–O(4)	172.5(2)
N(3)–Re–Br(1)	166.5(2)	N(3)–Re–O(2)	169.3(2)
Re–O(1)–C(7)	125.0(5)	Re–O(1)–C(7)	122.0(3)
C(3)–C(5)–N(3)	110.6(7)	C(3)–C(5)–N(3)	109.2(4)
C(5)–N(3)–C(6)	113.6(7)	C(5)–N(3)–C(6)	114.4(4)
C(1)–N(1)–C(3)	107.7(7)	C(1)–N(1)–C(3)	106.5(4)
C(2)–N(2)–C(3)	106.9(7)	C(2)–N(2)–C(3)	108.1(4)
O(2)–Re–Br(1)	103.5(2)	O(6)–Re–O(2)	103.8(2)
O(2)–Re–Br(2)	88.9(2)	O(6)–Re–O(4)	96.2(2)
O(2)–Re–N(1)	94.1(3)	O(6)–Re–N(1)	90.7(2)

940–970 cm^{-1} that is normally observed for complexes with a hard alcoxy donor atom *trans* to the Re$=$O moiety [11,12]. A single prominent band around 3200 cm^{-1} is assigned to ν(N(3)–H), proving that the secondary amino nitrogens remain protonated in **1** and **2**. The coordination of the deprotonated alcoxy oxygen O(1) of Hami in both complexes is reflected in ν(C–O) at 1170 and 1157 cm^{-1}, respectively. A strong band at 1208 cm^{-1} in the spectrum of **2** is assigned to ν(C–O) of the oxalate ligand. Two bands around 520 and 570 cm^{-1} in both complexes are ascribed to ν(Re–N), the latter value to ν(Re–N(1)).

^{1}H-NMR spectra of **1** and **2** clearly establish the presence of the chelate ami. The ring protons of ami are shifted markedly upfield in complex **2** relative to **1**. Protons attached to the atoms C(5), C(6) and C(7) give rise to the expected singlet–triplet–triplet pattern. Broad singlets around δ 9.6 ppm, integrating for one proton each, in the spectra of both **1** and **2** indicate the coordinated NH moiety, and thus the coordination of ami as a terdentate monoanionic chelate.

3.3. *Description of the structures*

The structure of **1** is illustrated in figure 1. It consists of a discrete, monomeric and neutral oxorhenium(V) complex [$ReOBr_2$(ami)] packed with no intermolecular contacts shorter than the sum of the van der Waals radii. The coordination geometry around

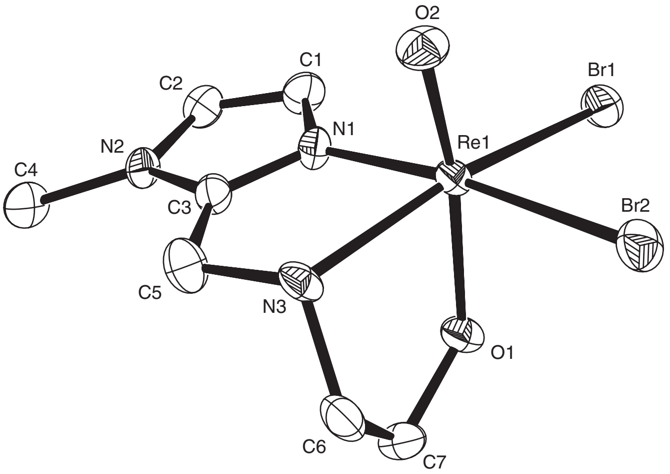

Figure 1. An ORTEP view of [$ReOBr_2$(ami)] (**1**) showing the atom labeling scheme and 50% probability ellipsoids.

rhenium is highly distorted octahedral; the two nitrogen atoms of the terdentate uninegative N(1)N(3)O(1)-donor ligand lie on the equatorial plane, along with the two *cis*-bromides, while the alcoholate O(1) is *trans* to the O(2) oxo atom. The equatorial plane is strictly planar, and the rhenium atom is displaced from this plane by 0.165 Å towards the oxo oxygen atom. This displacement leads to the nonorthogonal angles O(2)–Re–Br(1) = 103.5(2), O(2)–Re–N(1) = 94.1(3), O(2)–Re–N(3) = 89.2(3) and O(2)–Re–Br(2) = 88.9(2)°. The distortion results in a nonlinear O(1)–Re–O(2) axis of 164.1(3)°, accomplished by Br(1)–Re–N(3) and Br(2)–Re–N(1) angles of 166.5(2) and 173.4(2)°, respectively. The two 'bite' angles of ami, N(1)–Re–N(3) = 79.1(3) and O(1)–Re–N(3) = 75.7(3)°, contribute considerably to this distortion. The Re atom is 1.126 Å from the Br(1)Br(2)O(2) plane and 1.343 Å from the N(1)N(3)O(1) plane, the angle between the two triangular faces being 8.24°. The interligand angles in the equatorial plane depart slightly from the ideal value of 90° (from 89.9 to 95.1°). The imidazole ring is planar, as can be expected from an aromatic system, with torsion angles C(1)N(1)C(3)N(2) = 0.1(9) and C(2)N(2)C(3)N(1) = 0.4(9)°. The C(1)–C(2) and C(3)–N(1) bonds are double bonds at 1.32(1) and 1.31(1) Å, respectively. The bond angle around C(5) is 110.6(7)°, and the N(1)C(3)C(5)N(3) torsion angle equals 4(1)°. The axial Re–O(1) bond [1.907(5) Å] is longer than those found for Re–O (ethoxide) bonds, and indicates significant double bond character [5,13]. Another indication of its double bond character is that the Re–O(1)–C(7) angle is splayed to 125.0(5)° when no apparent intramolecular nonbonding contacts exist. Bond lengths within the inner core show no unusual features, being within the range expected from comparison with other six coordinate monooxorhenium(V) complexes containing the $O_2N_2X_2$ (X = Cl, Br) donor set, and are not discussed further here [11,14,15]. Only the Re–N distances deserve comment. The Re–N(1) distance [2.082(7) Å] is markedly shorter than the Re–N(3) bond [2.199(7) Å], showing the effect of the π-acidity of the imidazole ring.

In the mononuclear complex **2** (figure 2) the coordination geometry around the rhenium(V) is distorted octahedral; the two nitrogens of the terdentate ami ligand

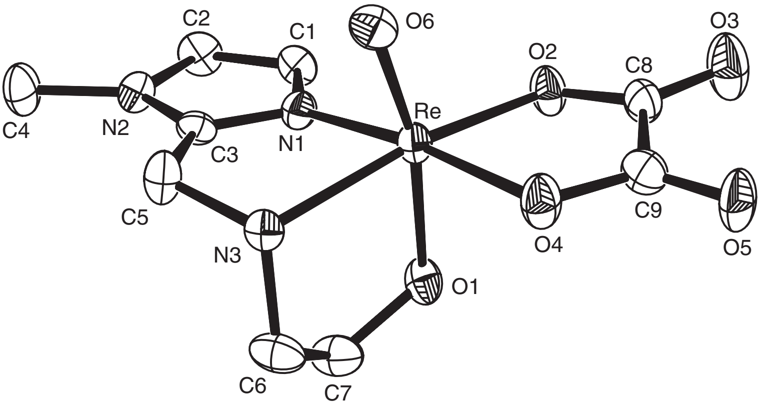

Figure 2. Molecular structure and atom numbering scheme for [ReO(ox)(ami)] (**2**). Hydrogen atoms have been omitted for clarity.

and the two oxalate oxygen atoms lie on the equatorial plane, with the alcoholate O(1) *trans* to the O(6) oxo atom. In the crystal the molecules are packed individually and interact via normal van der Waals contacts. The O(6)–Re–O(1) axis is nonlinear [164.9(2)°], and the rhenium atom is displaced from the mean equatorial plane by 0.163 Å towards the oxo oxygen atom, resulting in the nonorthogonal angles O(6)–Re–O(2) = 103.8(2), O(6)–Re–O(4) = 96.3(2), O(6)–Re–N(1) = 90.7(2) and O(6)–Re–N(3) = 86.7(2)°. Contrary to what was observed in **1**, the 'bite' angle O(1)–Re–N(3) [78.2(2)°] is larger than the N(1)–Re–N(3) one [77.7(2)°]. The bite angle of the oxalate is 81.0(2)°, considerably less than the Br(1)–Re–Br(2) angle of 89.91(3)° in **1**. In the O_4N_2 polyhedron the Re atom is 1.004 Å from the O(2)O(4)O(6) plane and 1.361 Å from the N(1)N(3)O(1) plane, the dihedral angle being 4.70°. There is a greater deviation from planarity of the imidazole ring in **2** than in **1**, with the C(1)N(1)C(3)N(2) and C(2)N(2)C(3)N(1) torsion angles equal to −1.9(6) and 1.0(6)°, respectively. There is a delocalized double bond over the N(1)–C(3)–N(2) part of the ring, with bonds N(2)–C(3) = 1.342(7) and N(1)–C(3) = 1.336(7) Å. The bond angle C(3)–C(5)–N(3) = 109.2(4) and the N(1)–C(3)–C(5)–N(3) torsion angle equals 15.6(7)°. The axial Re–O(6) [1.690(4) Å] and Re–O(1) [1.909(4) Å] bond lengths are nearly identical to those in **1**. As in **1**, the Re–N(1) distance [2.087(5) Å] is markedly shorter than Re–N(3) [2.188(4) Å], with the result that the two Re–O(oxalate) distances are significantly different [Re–O(2) = 2.034(4) and Re–O(4) = 2.069(4) Å]. The dinegative bidentate coordination mode of the oxalate ligand is manifested by the C(9)–O(5) and C(8)–O(3) double bonds [average 1.205(7) Å] and the C(9)–O(4) and C(8)–O(2) single bonds [average 1.305(7) Å].

A previous study with a number of terdentate O,N,N-donor Schiff base ligands gave the products *trans*-[$MOCl_2$(ONN)] (M = Re, Tc) [16]. A crystal structure analysis of a technetium analog showed that the central imino nitrogen of these Schiff bases is located *trans* to the oxo group. Because of the resultant *trans* arrangement of the two chlorides, it is intimated that the preparation of oxorhenium(V) complexes with 'mixed' di- and terdentate chelates by the substitution of the chlorides by dibasic ligands would be unlikely.

We have previously isolated the complex [$ReOCl_2(L)$], where L^- is an ONN-donor ligand containing a phenolate oxygen, secondary amino nitrogen and a pyridyl nitrogen as donor atoms (HL = 2-[[{2-(2-pyridinyl)ethyl}amino]methyl]phenol) [14]. A crystal structure determination showed that the two chlorides are in *cis* positions, with the phenolate oxygen occupying the site *trans* to the oxo oxygen. In our opinion [$ReOCl_2(L)$] was an ideal starting complex to prepare new complexes with 'mixed' didentate OO-donor and terdentate ONN-donor ligands by simple substitution of the *cis* chlorides. However, this was only successful for the synthesis of [ReO(eg)(L)] (H_2eg = ethylene glycol), and unsuccessful for [ReO(cat)(L)] and [ReO(ox)(L)] (H_2cat = 1,2-dihydroxybenzene). The latter two complexes were isolated from the one-pot reaction of $[ReOCl_4]^-$ with HL and H_2cat/H_2ox [17].

Supplementary data

CCDC-253774 (for **1**) and CCDC-253775 (for **2**) contain the crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033; email: deposit@ccdc.cam.ac.uk].

References

[1] X. Chen, F.J. Femia, J.W. Babich, J. Zubieta, *Inorg. Chim. Acta*, **307**, 154 (2000).
[2] F.J. Femia, X. Chen, K.P. Maresca, T.M. Shoup, J.W. Babich, J. Zubieta, *Inorg. Chim. Acta*, **306**, 30 (2000).
[3] B. Nock, T. Maina, D. Yannoukakos, I.C. Pirmettis, M.S. Papadopoulos, R. Chiotellis, *J. Med. Chem.*, **42**, 1066 (1999).
[4] X. Chen, F.J. Femia, J.W. Babich, J. Zubieta, *Inorg. Chim. Acta*, **307**, 149 (2000).
[5] T.I.A. Gerber, Z.R. Tshentu, P. Mayer, *J. Coord. Chem.*, **56**, 1357 (2003).
[6] T.I.A. Gerber, E. Hosten, Z.R. Tshentu, P. Mayer, E. Pérez-Carreño, S. Garcia-Granda, J.G.H. du Preez, *J. Coord. Chem.*, **56**, 1063 (2003).
[7] T.I.A. Gerber, Z.R. Tshentu, S. Garcia-Granda, P. Mayer, *J. Coord. Chem.*, **56**, 1093 (2003).
[8] K.P. Maresca, F.J. Femia, G.H. Bonavia, J.W. Babich, J. Zubieta, *Inorg. Chim. Acta*, **297**, 98 (2000).
[9] Z.R. Tshentu, MSc thesis, Univesity of Port Elizabeth, South Africa (2002).
[10] G.M. Sheldrick, *SHELX-97*: *Programs for Structure Solution and Refinement*, University of Göttingen, Germany (1997).
[11] A. Abrahams, G. Bandoli, S. Gatto, T.I.A. Gerber, J.G.H. du Preez, *J. Coord. Chem.*, **42**, 303 (1997).
[12] H.J. Banbery, F. McQuillan, T.A. Hamor, C.J. Jones, J.A. McCleverty, *J. Chem. Soc.*, *Dalton Trans.*, 1405 (1989).
[13] T.I.A. Gerber, D. Luzipo, P. Mayer, *J. Coord. Chem.*, **56**, 1549 (2003).
[14] G. Bandoli, T.I.A. Gerber, R. Jacobs, J.G.H. du Preez, *Inorg. Chem.*, **33**, 178 (1994).
[15] T.I.A. Gerber, H.J. Kemp, J.G.H. du Preez, G. Bandoli, *J. Coord. Chem.*, **28**, 329 (1993).
[16] F. Tisato, F. Refosco, A. Moresco, G. Bandoli, U. Mazzi, M. Nicolini, *J. Chem. Soc.*, *Dalton Trans.*, 2225 (1990).
[17] A. Abrahams, G. Bandoli, S. Gatto, T.I.A. Gerber, J.G.H. du Preez, *J. Coord. Chem.*, **43**, 297 (1998).