This thesis investigates the synthesis and characterization of rhenium complexes containing oxygen, nitrogen and thiol ligands. The first part describes the chemistry of the nonradioactive rhenium labeling of four somatostatin analogues. Confirmation for the formation of the four rhenium-cyclized somatostatin analogues was accomplished with LC-MS. Purification was performed by analytical HPLC.

The LC-MS data showed the formation of two cyclic rhenium products with different retention times and mass ionization patterns, which suggest the formation of isomers with different rhenium binding structures. The retention time comparison studies showed that by acetylation of the peptide N-terminus, a product with increased hydrophilicity, and possibly a structure more similar to the disulfide, was formed as the major product.

The second part describes the synthesis and characterization of three rhenium complexes, two of them with ethylenediamine type ligands, in which the rhenium is in +5 oxidation state, and the third complex with the 2-aminoethanethiolate ligand and a phosphine ligand, in which the rhenium is in the +3 oxidation state.

The acid dissociation constants (pKa2 values) for the co-ordinated oxo groups of rhenium complexed ethylenediamine type ligands supported the observation that functionalization of ethylenediamine type ligands decreased the pKa values of the oxo group of the Re(V) complexes rather than increasing them.