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COORDINATION CHEMISTRY REVIEWS

Yttrium 1995

Zoe Pikramenou *

University of Edinburgh, Dept. of Chemistry, West Mains Road, Edinburgh EH9 3JJ, U.K.

Contents

1. Introduction

This review covers the coordination chemistry of yttrium for 1995. Although an SCI search exhibited 705 papers referring to yttrium, a lot of the work was centred around the properties of yttrium compounds in materials science and medical applications. The literature on superconductivity of yttrium complexes is broad and selected examples of appropriate compounds have been covered. This review does not cover organometallic complexes although, a few organometallic complexes could not be avoided, due to their mixed ligand nature featuring coordinatively interesting ligands. The literature was searched using both the Cambridge Crystallographic Data Base (CCDB) and the SCI. Crystal structures shown were redrawn using structural coordinate files from the CCDB.

2. Yttrium(III)

2.1. Complexes with halide ligands

The reaction of Y_2O_3 and Li_2CO_3 with NH_4F to produce $LiYF_4$ was studied by thermogravimetric and X-ray diffraction methods [1]. Fluorination of yttrium oxide

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* Corresponding author. E-mail: pikramenou@ed.ac.uk

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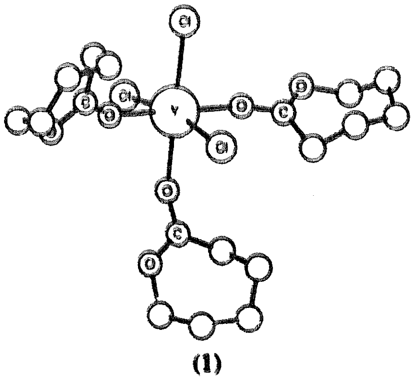
gives first $YF_3 \cdot 1.5NH_3$ which decomposes at 300-380°C to YF_3 and NH_3. In the ternary system NH_4F-Li_2CO_3-Y_2O_3, the resulting mixture of simple yttrium and lithium fluorides is converted to $LiYF_4$ at high temperatures. An investigation of the thermal decomposition of ammonium yttrium chlorides, $(NH_4)_3YCl_6$ [2] and iodides $(NH_4)_3YI_6$ [3] has been reported.

The solid state reaction of stoichiometric amounts of yttrium metal, yttrium iodide, yttrium nitride and carbon in closed Ta capsules at 1150 K gave $Y_7I_{12}C_2N$ [4]. The olive green crystals have been analysed by X-ray crystallography. A study of the polytypism and phase width of Y_2Br_2C which crystallises in two modifications has been reported [5]. The two forms are temperature polytypes with a transition temperature of 1400 K.

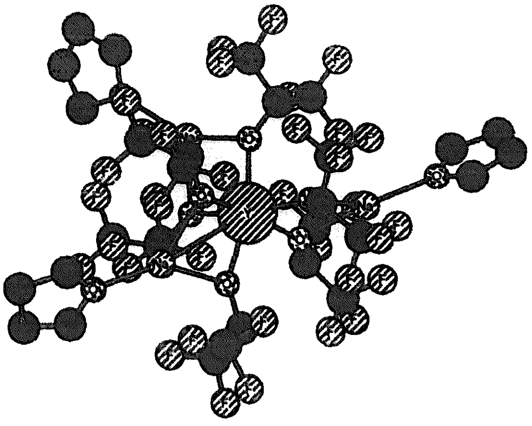
An extensive study of the structural diversity in solvated lanthanoid halide complexes has been reported [6]. Crystallization of lanthanoid trichlorides from neat ϵ-caprolactone ($C_6H_{10}O_2$) and caprolactone/THF mixtures has revealed a very diverse structural chemistry for solvated lanthanoid trichloride complexes. The products do not follow a particular pattern in terms of ease of formation, crystallization, size of metal or nature of the coordinating ligand. The crystal structure of the yttrium complex $YCl_3(C_6H_{10}O_2)_3$ (**1**) is reported and compared with other structures of lanthanoid complexes; their implications in lanthanoid chemistry is discussed. The six-coordinate yttrium complex (**1**) shows only slight distortion from regular octahedral geometry; the three chloride ligands adopt a meridional geometry which is like the T-shaped arrangement of the halides in pentagonal bipyramidal $LnCl_3L_4$ complexes.



(1)

2.2. Complexes with oxygen donor ligands

Yttrium alkoxides have been of interest as metal precursors for MOCVD synthesis of high-T_c superconductors. Metathesis reactions between YCl_3 and sodium fluoroalkoxides offered mixed metal species such as $Na_3Y(hfip)_6(THF)_3$ (**2**) and $Na_2Y(hftb)_5(THF)_3$ (**3**) and $Na_2Y(tftb)_5(THF)$ where hfip = $OCH(CF_3)_2$, hftb = $OCMe(CF_3)_2$ and tftb = $OCMe_2(CF_3)$ [7]. Their synthesis and characterisation by elemental analysis, FT-IR, mass spectrometry, ^1H, $^{13}C\{1H\}$ and ^{19}F NMR spectroscopies, and X-ray crystallographic analysis for (**2**) and (**3**) have been reported.



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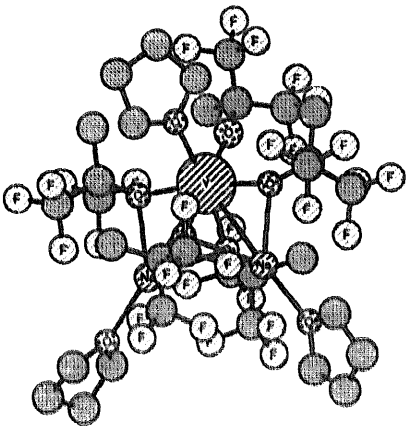
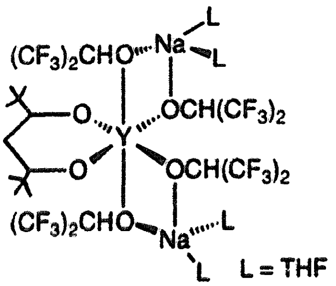


(**3**)

The formation of mixed metal Na–Y species takes place even in the case where the sodium reactant is in a smaller amount than the stoichiometric quantity. The steric demand of the alkoxide groups (OR_f) is claimed to be the imposing factor of the control of complex formation. The NMR spectroscopic data confirm the presence of THF molecules in the metal coordination sphere. A common feature of the crystal structures is the distorted octahedral environment around the yttrium centre, the sodium atoms being connected as $Na(\mu\text{-}OR_f)_2$ units. The sodium atom retains a high coordination number without polymerisation, ensuring volatility as well as stability in the vapour phase. The THF ligands are labile in solution in the presence of other polar solvents and in the vapour phase. Volatile yttrium-sodium fluoroisopropoxide derivatives have also been synthesised by Labrize *et al* [8]. The syntheses and characterization of the mixed metal species (**2**) and $YNa_2[OCH(CF_3)](thd)(THF)_4$ (**4**) were reported (thdH = 2,2,6,6-tetramethylheptane-3,5-dione). Reaction of $Y[OCH(CF_3)_2](THF)_3$ with thdH yields the complex $Y[OCH(CF_3)_2](thd)_2$ (**5**). The complexes are characterised by ^1H, ^{13}C and ^{19}F

NMR and IR spectroscopies, mass spectrometry and elemental analyses. The spectroscopic data indicate a dimeric structure for (**5**).



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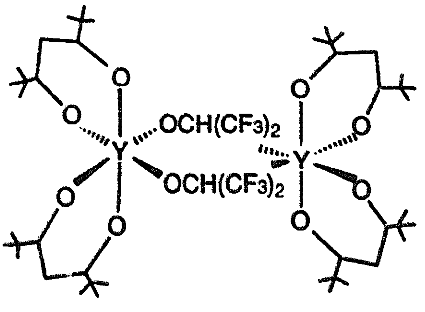


(5)

A review of the employment of metal alkoxides and β-diketonates as molecular precursors to metal oxides and their implication in materials science has been reported [9]. The synthetic routes and appropriate choice of alkoxide and/or ancillary ligands for the control of the properties of the resulting oxides are accounted. Strategies that govern the formation of mixed-metal species and the control of the stoichiometry between metals are also discussed. The structure of potassium yttrium cyclotetraphosphate is reported [10]. The structure contains the cyclotetraphosphate ring anion $[P_4O_{12}]^{4-}$. The rings are linked by YO_8 polyhedra to form a three dimensional framework. This disposition leaves intersecting tunnels in which the K^+ ions can reside.

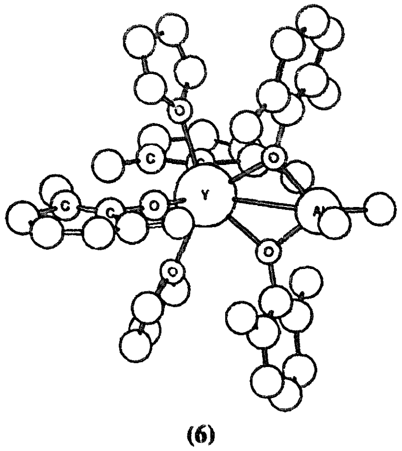
Heterometallic isopropoxides of yttrium(III) and lanthanoids(III) have been prepared for the development of versatile precursors for mixed metal oxides [11]. Reaction of $YCl_3 \cdot 3Pr^iOH$ with $KSn_2(OPr^i)_9$ in benzene gave the complexes $[YCl_{3-x}\{Sn_2(OPr^i)_9\}_x]$, x = 2 or 3. The complexes were characterised by IR and ^1H, ^{13}C and ^{119}Sn NMR spectroscopies. The NMR spectroscopic data agree with the tridentate bonding mode of nonaisopropoxodistannate $[(Pr^iO)_3Sn(\mu\text{-}OPr^i)_3Sn(OPr^i)_3]^-$.

Mixed metal complexes including yttrium or lanthanoids and aluminium ions with the 2,6 phenoxide ligand (ArO) have been synthesised [12]. The $[(ArO)_2(THF)_2Y(\mu\text{-}OAr)_2AlR_2]$ [R = Me or Et] complexes have been prepared by reaction of $Y_2(OAr)_6\text{-}(THF)_2$ with Al_2Me_6 or $AlEt_3$ respectively. The complex (**6**) (R = Me) has distorted octahedral and tetrahedral coordination environments around the lanthanoid and aluminium centres, respectively.

The structure refinement of $Y_2Ru_2O_7$ by neutron powder diffraction is reported [13].

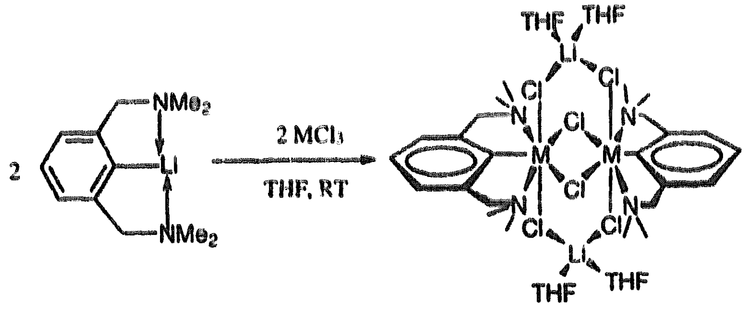
2.3. Complexes with nitrogen donor ligands

New complexes of lutetium and yttrium with the monoanionic terdentate ligand $[2,6\text{-}(Me_2NCH_2)_2C_6H_3]^-$ (abbreviated to NCN) have been synthesised [14]. Reaction of the corresponding MCl_3 (M = Y, Lu) with one equivalent of (NCN)Li afforded the complexes $[(NCN)MCl_2(\mu\text{-}Cl)(\mu\text{-}Li(THF)_2)]_2$ (**7**). The terdentate



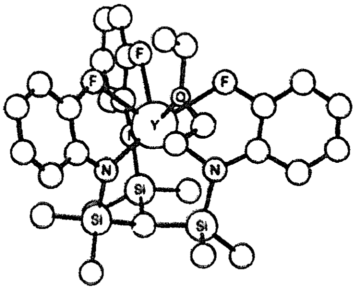
(6)

ligand is bound in a *mer*-fashion. The NMR spectroscopic data suggest that the two complexes are isostructural. The X-ray crystal structure of the Lu(III) complex has only been obtained. Each metal centre is heptacoordinate. The M(μ-Cl)$_2$M unit is flat and it is twisted 21.1° with respect to the aromatic plane of the aryldiamine ligand. The two methyl groups of each nitrogen and the two protons of each benzyl group are non-equivalent. The variable temperature ^{1}H NMR spectroscopic data are consistent with this argument. The NMe$_2$ resonances appear as a singlet at room temperature which splits to two singlets when the solution is cooled to 245 K. The ^{13}C NMR spectra of the yttrium complex show additional features due to coupling to ^{89}Y. The data indicate an equilibrium between the two species [(NCN)YCl$_3$]$_2^{2-}$ and [(NCN)YCl$_2$(μ-Cl)(μ-Li(THF)$_2$] hich becomes slow at low temperatures.



(7)

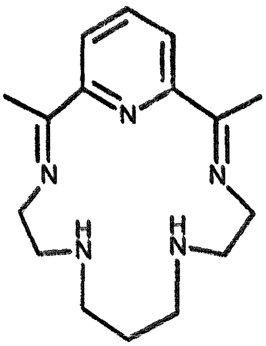
Reaction of the lithium salt of the tripodal ligand HC[SiMe$_2$NH(2-FC$_6$H$_4$)]$_3$ with YCl$_3$ yields the seven-coordinate yttrium complex {HC{SiMe$_2$NH(2-FC$_6$H$_4$)}$_3$}Y(OEt$_2$) (**8**) which has been structurally characterised by X-ray crystallography [15]. The ^{1}H, ^{13}C, ^{29}Si and ^{19}F NMR spectroscopic data at 295 K are consistent with the three-fold symmetry of the compound. However, at lower temperatures they indicate a structure lower than three fold molecular symmetry. The coordination of the seven donor atoms is established by the X-ray structure analysis and it involves the three amido atoms of the tripodal ligand as well as an oxygen atom of the diethyl ether molecule. The recent advances in the chemistry of yttrium and lanthanoid pyrazolyborate complexes have been reviewed [16]. The paper describes the structural and solution behaviour of the complexes.



(8)

Lanthanoid complexes with the ligand (^{t}Bu)N=CHCH=N(^{t}Bu) (dad) bound as a dianion are synthesised [17]. The yttrium complex is isolated as pale yellow crystals. The NMR spectroscopic data show one set of signals for the dad ligand, indicative for a highly symmetric complex. However the crystal structure data of the Lu(III) analogue show that two different types of complex exist. Low temperature NMR spectroscopy was employed to elucidate the process of interconversion of the two isomers and the energy barrier for the process was estimated.

Template synthesis of macrocyclic Schiff base complexes of yttrium have been reported. The reaction of 2,6-diacetylpyridine with 3,3′-diaminodipropylamine in the presence of yttrium nitrate produces the 28-membered N_8 [2+2] cyclocondensation product [18]. The complex is characterised by IR and NMR spectroscopies, elemental and thermogravimetric analysis. In a similar fashion, [1+1] Schiff base cyclocondensation of 2,6-diacetylpyridine with 3,7-diazanonane-1,9-diamine in the presence of yttrium perchlorate gave the 16-membered N_5 macrocyclic complex of ligand (9) [19]. The ^{1}H NMR spectrum of the complex exhibits three well resolved triplets in a ratio of 1:1:1 attributed to the two methylene groups of the $=NCH_2CH_2N$ chain and the methylene group of the propylene bridge to the nitrogen. The complex was also characterised by MS and elemental analysis.



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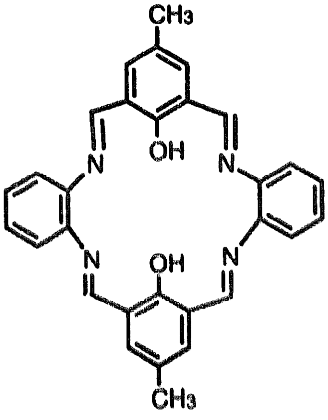
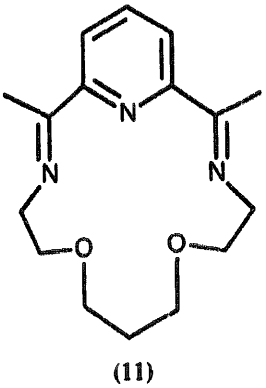
2.4. Complexes with mixed oxygen and nitrogen donor ligands

Yttrium(III) and lanthanoid complexes of the 18-membered dioxatetraaza macrocyclic ligand (**10**) have been prepared by template synthesis [20]. Schiff base condensation of 2,6-diformyl-4-methylphenol with 1,2-diaminobenzene in the presence of the yttrium(III) nitrate in acetonitrile gives the crystalline complex in 60.9% yield. The complex is characterized by IR spectroscopy, FAB MS and UV–VIS spectroscopy. Absence of the template ion leads to the formation of a different macrocycle that does not bind metal ions when it is pre-formed. The IR spectroscopic data of the complex indicate that there is not a hydrogen bond between the OH groups in the macrocycle and that a nitrate counter-ion is coordinated in a didentate fashion to the metal ion. Thermogravimetric analysis indicates the high stability of the coordinated macrocycle which is stable up to 320°C for a series of lanthanoid complexes. Although macrocycle (**10**) has two ionizable protons, it acts as neutral species. The coordination environment of the metal ion is satisfied by the four nitrogens and one oxygen of the macrocycle, the chelating nitrate ion and a water molecule. The ability of this macrocycle to bind a series of lanthanoids is attributed to its flexibility to adapt its size to the demands of the metal ion.

A 15-membered Schiff base macrocyclic ligand complex was also synthesized by



(**10**)

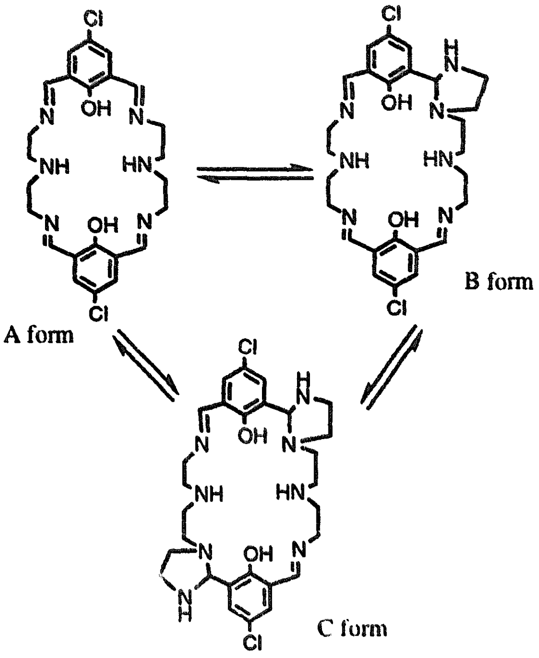


(**11**)

template synthesis [19]. Cyclocondensation of 2,6-diacetylpyridine with 3,6-dioxaoctane-1,8-diamine in the presence of yttrium chloride gives the $[YL(H_2O)_2]Cl_3 \cdot H_2O$ complex of macrocycle L where L = (**11**); this has been characterized by IR and ^1H NMR spectroscopies, UV–VIS, and MS data, elemental and thermogravimetric analyses. The C=N stretching frequency at 1630 cm^{-1} is indicative of the Schiff base condensation. The C–O–C stretching mode at 1110 cm^{-1} and the ring breathing mode at 1010 cm^{-1} are characteristic of the coordinating oxygen atoms and the pyridine nitrogen, respectively. The two different binding modes of the water molecules is shown not only by the IR spectroscopic data but also by the thermogravimetric analysis results. The singlet for the methylene protons of the ethylene bridge in the ^1H NMR spectrum agrees with the rest of the data and indicates the planarity of the macrocycle. Interestingly, the complex does not form when yttrium nitrate is employed. The authors have attributed the influence of the counter-ion to the coordinating effect of the nitrate as opposed to the chloride. The nitrate coordination may result in an increased ionic radius of the yttrium(III) centre and the metal ion does not fit well in the macrocyclic cavity. This 15-membered planar macrocycle seems to stabilize yttrium(III) in the uncommon coordination number of seven by the equatorial donor set of the macrocycle and two water molecules in the axial positions.

An investigation of the interconversion of two enantiomeric forms of macrocyclic Schiff base yttrium and lanthanoid complexes gives a correlation of the results of the solution studies and crystal structures [21]. The ligand, H_2L, (**12**) has been previously reported and the interconversion of the three isomeric forms is shown schematically. The complexes were prepared following literature procedures and crystals of the yttrium complex $[Y(H_2L)(NO_3)_2](NO_3)$ (**13**) were grown from a dimethylformamide/diethyl ether solution. The crystal structure was determined by X-ray crystallography. The yttrium atom is nine-coordinate with five donor atoms of the macrocycle which possesses a type B structure with the metal ion occupying only one chamber of the macrocycle. A 2D-heterocorrelated reverse experiment [^{13}C-^1H] HSQC allowed the detection of the ^{13}C signals that could not be obtained with a normal experiment due to the low solubility of the complex. The low temperature results (243 K) suggest the contracted form of the complex as it is in the crystal structure. The NMR spectroscopic results agree with those of the more soluble, isostructural La(III) complex. The variable temperature NMR spectroscopic studies clearly give evidence for a dynamic process involving C–N bond activation and a hydrogen transfer mechanism to result in the ring contraction and expansion.

The synthesis and crystal structure of the yttrium triethanolamine complex $[Y(TEA)_2](ClO_4)_3 \cdot 3C_5H_5N$ (**14**) was reported [22]. Excess triethanolamine reacts with a methanolic solution of $Y(ClO_4)_3 \cdot 6H_2O$ at room temperature for 15 hours. Crystals were obtained by layering pyridine onto an ether solution of the complex. The complex was characterised by IR and ^1H and ^{13}C NMR spectroscopies. The solution data are not conclusive for the binding of the pyridine to the complex, whether it is metal coordinated or hydrogen bonded to the OH of the ligand. The crystal structure (**14**) shows the yttrium ion to be octa-coordinated by the two ligating triethanolamine molecules. The pyridine molecules form hydrogen bonds



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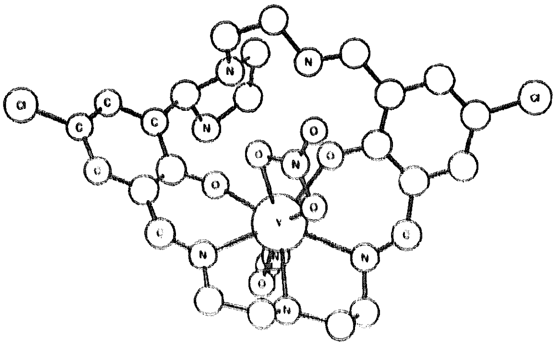
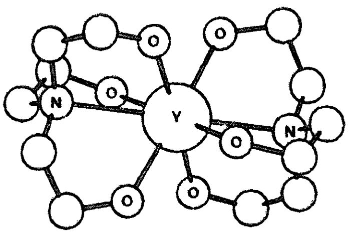


(13)

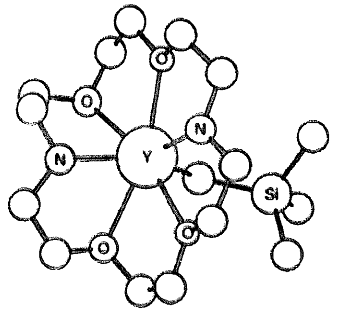
with the OH protons of the unprimed side of the molecule and the oxygen of each perchlorate forms a hydrogen bond with the OH proton of the primed side of the molecule. This hydrogen bonding pattern in the molecule may be responsible for the lengthening of the Y–N′ and Y–O′ bonds relative to the Y–N and Y–O bonds respectively.

A series of chromium(III)-lanthanoid(III) complexes have been synthesised and structurally characterised for an understanding of the interactions between the ions in the same crystal lattice [23]. Isolation of the yttrium complex $[Cr(NH_2)_2sar]$-$[Y(dipic)_3] \cdot 8H_2O$ (sar = 3,6,10,13,16,19- hexaazabicyclo[6.6.6]icosane and dipic = pyridine-2,6-dicarboxylate) and its crystal structure are reported. The deprotonated 4,13 diaza-18-crown-6 (DAC) has been used as an ancillary ligand in yttrium alkyl complexes. Synthesis of $Y(DAC)(CH_2SiMe_3)$, its X-ray crystal



(14)

structure and its thermal stability have been reported [24]. The X-ray crystal structure (**15**) features a seven-coordinate yttrium centre exhibiting no agostic interactions with the alkyl group.



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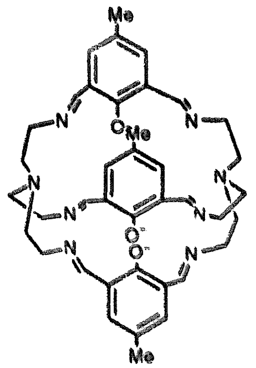
The cryptate complexes of ligand (**16**) with yttrium and lanthanoids have been synthesised and characterised by NMR spectroscopy, mass spectrometry and their structures are analysed by X-ray crystallography [25]. The yttrium complex $[\mathrm{Y}(\mathit{16})(\mathrm{H_2O})]^{3+}\,3\mathrm{ClO_4^-}\cdot 2\mathrm{EtOH}\cdot 0.5\mathrm{MeOH}$ (**17**) is isomorphous with the Gd and Tb one. The metal ion is eight-coordinate, being bonded to three imino-nitrogens and three oxygen donors from one end of the cryptand as well as to the bridgehead nitrogen and one water molecule. The kinetic stability of the complexes is evident over a period of several days.

The reactions of $\mathrm{Ln(hfacac)_3}$, where Ln = Y or lanthanoids and



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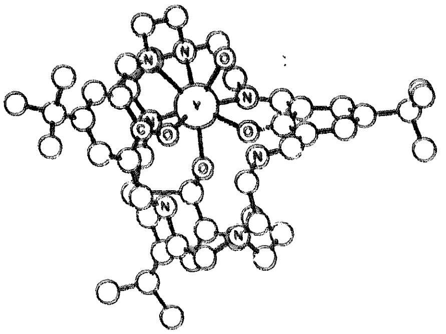

(17)

hfacacH $=CF_3C(O)CH_2C(O)CF_3$, with the bdmapH ligand and $Cu(OCH_3)_2$ or $Cu_2(O_2CCH_3)_4(H_2O)_2$ have been investigated (bdmapH = 1,3-bis(dimethylamino)-2-propanol) [26]. The YCu(bdmapH)$_2$(hfacac)$_2$(O_2CCF_3)L complex was isolated and characterised, L^{2-} = 2-methyl-2,4,6-tris(trifluoromethyl)-1,3-dioxane-4,6-diolato. The magnetic properties of the complex have been examined.

2.5. Complexes with sulfur, selenium or tellurium donor ligands

The structure and properties of the ternary yttrium sulfides, CaY_2S_4, SrY_2S_4 and BaY_2S_4 have been described [27]. CaY_2S_4 crystallises in the orthorhombic [Yb_3S_4]-type structure whereas SrY_2S_4 and BaY_2S_4 adopt the orthorhombic [$CaFe_2O_4$] structure. The properties of these compounds indicate that they are potentially useful as infrared window materials.

Homoleptic trivalent selenolates of lanthanum and yttrium have been isolated from the reaction of $M[N(SiMe_3)_2]_3$ (M = La, Y) with three equivalents of $HSeSi(SiMe_3)_3$ in hexanes [28]. A dimeric structure is proposed for the yttrium compound based on the variable temperature NMR spectroscopic data $\{Y[SeSi(SiMe_3)_3]_2[\mu\text{-}SeSi(SiMe_3)_3]\}_2$. Complete characterisation of the compounds by IR and ^{1}H, ^{13}C{^{1}H}, ^{77}Se{1H} 2D spectroscopies and elemental analysis. Tellurolate species have also been prepared and addition of DMPE (1,2-bis-(dimethylphosphino)ethane) following the former procedure gave the yttrium complex $Y[TeSi(SiMe_3)]_3(DMPE)_2$ which was characterised by multinuclear and variable temperature NMR spectroscopy [28]. The X-ray crystal structure of the lanthanum tellurolate derivative has been determined.

2.6. Complexes with mixed sulfur and oxygen donor ligands

Reaction of sodium sulfo-salicylate with lanthanum and yttrium perchlorates afforded the complex $[Na_3YLa_2(C_7H_3SO_6)_4\cdot 26H_2O]$ [29]. The crystal structure has been determined by single crystal X-ray diffractometry.

2.7. Complexes with mixed sulfur and nitrogen donor ligands

Mixed ligand complexes of lanthanoids and yttrium with 1,10 phenanthroline (phen) and 4-(3,5-diphenyl-2-pyrazolin-1yl)benzene sulfonate = DPBS (x) with the formula $[M(phen)_2(DPBS)_2]\cdot DPBS$ have been synthesised [30]. Reaction of $M(phen)_2(ClO_4)_3$ with HDBPS in methanol yields the complexes which are characterised by elemental analyses, molar conductances, magnetic measurements, thermogravimetric analyses, XPS, IR and UV–VIS spectral studies. The data indicate an octahedral structure where phen is *trans* to another phen in the equatorial position and two DPBS ligands are *trans* to each other in the axial positions. The yttrium(III) complex was obtained in 64% yield. Another series of mixed ligand complexes of yttrium and lanthanoids features bipyridyl and bis(phenylsulfinyl)propane (bphfsp) as ligands [31]. The analytical, spectroscopic and magnetic data indicate a formula of $[Y(bphsp)_3(bpy)(ClO_4)](ClO_4)_2$. A strong Lewis acid based on yttrium was prepared by mixture of yttrium nitrate with zirconyl nitrate (16:84 ratio) with aqueous ammonia [32]. Treatment with sulfuric acid gave a compound with composition 82.6 Zr, 15.6 Y, 1.8 S%.

3. Superconductivity

Superconducting powders with the composition of $(Y_{1-x}Eu_x)Ba_2(Cu_{1-y}Fe_y)_4O_8$ have been prepared by a simple sol-gel method [33]. Studies on the $(Y_{1-x}Eu_x)Ba_2Cu_4O_8$ system highlight the importance of doping with iron and the effect on the properties. The crystal structure of $YBaCu_2(CO_3)O$-$5.5 \pm$ delta oxycarbonate has been reported [34]. A series of yttrium based compounds $La_{1-x}Sr_xYCu_2O_6$ ($x = 0$ to 0.5) has been prepared [35]. The crystal structure and cation distribution are determined by Rietveld analysis of X-ray powder diffraction data.

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