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COORDINATION CHEMISTRY REVIEWS

4. Yttrium 1993

Catherine E. Housecroft

CONTENTS

INTRODUCTION

This article surveys the coordination chemistry of yttrium for the year 1993 and is similar in format to the 1992 review in this series [1]. The literature has been searched by using *Current Contents* and the Cambridge Crystallographic Data Base. Structural figures have been redrawn using coordinates taken from the Cambridge Crystallographic Data Base, implemented through the ETH, Zürich [2].

The review is not wholly comprehensive; the aim is, rather, to give a broad survey of the area for the year, highlighting novel compounds, compounds that have been structurally characterized, and results of particular significance. In general, organometallic complexes are excluded from the survey, although where a complex contains a ligand of interest to the coordination chemist, it has been described.

A review entitled 'zero oxidation state of compounds of scandium, yttrium and the lanthanides' has appeared [3].

Of general interest is an article which details the application of CP/MAS ^{89}Y NMR spectroscopy for the characterization of solid state yttrium compounds. Data can be obtained over a short period of time, and a wide range of complexes has been studied including $[YX_3(thf)_3]$ (X = Cl, Br, I), YF_3, YCl_3, Y_2O_3, $YCl_3.6H_2O$, $Y(OAc)_3.nH_2O$ (n = 3, 4), $Y_2(SO_4)_3.8H_2O$, $Y(NO_3)_3.6H_2O$, $[Cp_3Y(thf)]$, $[\{Cp_2YCl\}_2]$ and several alkoxides. The chemical shift range is about 1000 ppm [4]. Gas-phase reactions of methanol and Y^+ (and Sc^+) ions have been studied using FT-ion cyclotron resonance mass spectrometry. The formation of the ions $[YOH]^+$, $[YOMe]^+$, $[YO]^+$,

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$[YOCH_2]^{2+}$ and $[YH_2]^+$ is observed, and further reaction leads to $[Y(OMe)_2(MeOH)_n]^+$ for $n = 0$–3. Estimates of Y–O bond energies have been made [5].

4.1 YTTRIUM(III)

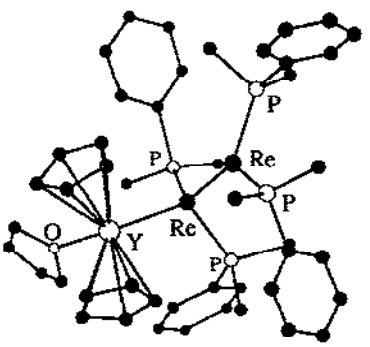
4.1.1 Extraction

Acidic organophosphonates containing hydrophobic groups have been prepared and their effectiveness with respect to the extraction of Y^{3+} (and Ho^{3+} and Er^{3+}) ions from aqueous acidic chloride solution has been investigated. The extraction equilibrium constants and the separation factors have been found to exceed those of commercially used extraction agents [6].

4.1.2 Complexes with halide and hydride donors

Physicochemical techniques have been used to study the binary systems containing YI_3 and alkali metal iodides. The YI_3/NaI system is of the eutectic type; $7NaI.3YI_3$ forms and decomposes at 410°C. Combinations of YI_3 and KI or RbI lead to $3KI.YI_3$ and $3Rbi.YI_3$, and these melt at 650 and 630°C respectively [7].

The reaction between $[YCl_3(thf)_{3.5}]$ and LiL where HL is $PhCH\{N(SiMe_3)_2\}_2$ leads to the formation of the complex $[L_2YCl]$. This proves to be a good precursor to a range of yttrium(III) alkyls including $[L_2YCH(SiMe_3)_2]$, which reacts with H_2 (3 atm) to yield an air sensitive compound identified as the dimer $[\{L_2Y(\mu\text{-}H)\}_2]$. In the ^1H NMR spectrum of $[\{L_2Y(\mu\text{-}H)\}_2]$, the hydride signal shows coupling of $J_{YH} = 27.6$ Hz. Reactions of $[L_2YCH(SiMe_3)_2]$ and $[\{L_2Y(\mu\text{-}H)\}_2]$ with alkynes have been studied [8]. Heterometallic polyhydrides of yttrium(III) result from the reaction of $[Cp_2YMe(thf)]$ with $[ReH_7(PPh_3)_2]$ and $[ReH_5(PMe_2Ph)_3]$. Crystallographic data confirm that the products are $[Cp_2Y(thf)H_6Re(PPh_3)_2]$ and $[Cp_2Y(thf)H_4Re(PMe_2Ph)_3]$ — both possess bridging hydride ligands. Treating $[Cp_2YMe(thf)]$ with $[Re_2H_8(PMe_2Ph)_4]$ leads to the complex $[Cp_2Y(thf)H_7Re_2(PMe_2Ph)_4]$ (**1**) which exhibits an open L-shaped YRe_2-core. Compound (**1**) is



Hydrides not shown

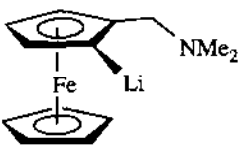
(**1**)

fluxional in solution; it reacts with CO and CO_2 but not with ethene. The new compounds have been characterized by ^1H and ^{31}P NMR and IR spectroscopies (in addition to the X-ray diffraction studies). In the ^1H NMR spectra, both ^1H-^{89}Y and ^1H-^{31}P couplings are observed [9].

4.1.3 Complexes with nitrogen donor ligands

The reaction of $[Cp^*YCl_2].KCl.1.8thf$ and LiL, (2), leads to the brown-red complex [Cp*YLCl] which has been characterized by IR, UV-VIS and ^1H and ^{13}C NMR spectroscopies. A structure for [Cp*YLCl] has been proposed, with the L^- ligand binding to the yttrium(III) centre as a *C,N*-chelate. The UV-VIS spectrum of [Cp*YLCl] (thf) exhibits bands with λ_{max} = 22 050 cm^{-1} (lg ε = 1.96, assigned to the $^1A_{1g} \rightarrow {}^1E_{1g}$ transition), 29 700 cm^{-1} (lg ε = 2.62) and 30 050 cm^{-1} (lg ε = 2.65). The same authors have also described the related complex $[Cp_2YL]$ [10,11].

The synthesis of the yttrium(III) complex $[\{MeN(CH_2CH_2C_5H_4)_2YCl\}_2]$ has been reported. Characterization by mass spectrometry, and IR and ^1H NMR spectroscopies leads to the proposal of structure (3) [12].



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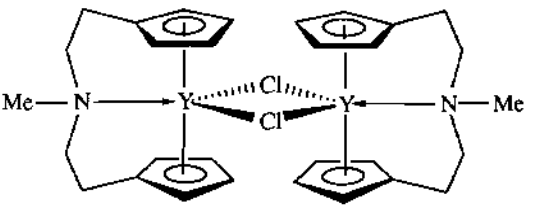


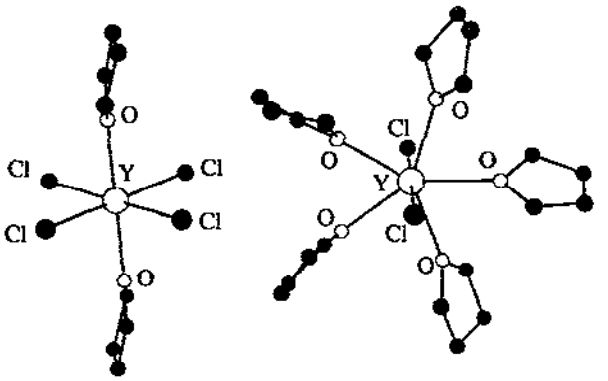
(3)

4.1.4 Complexes with oxygen donor ligands

Phase transitions in the system yttrium acidic oxalate trihydrate have been studied using a method involving Eu^{3+} luminescence; a phase transition is observed at 328 K. Crystal field parameters have been determined [13].

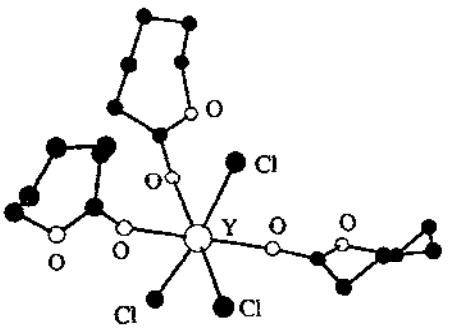
It has been possible to obtain single crystals of $[NH_4]_2[Y(NO_3)_5]$ from a solution of the sesquioxide in a melt of ammonium nitrate; excess ammonium nitrate was sublimed. The single crystal structure of the related compound $[NH_4]_2[Tm(NO_3)_5]$ has been determined; the Tm(III) centre is coordinated by five didentate nitrate ions [14]. The enthalpies of solution of $Y(NO_3)_3.nH_2O$ have been determined in water (at 298.15 K). There is a linear relationship between $\Delta H_{soln.}$ and the water content (n). The result for a series of compounds (yttrium(III) and lanthanide nitrates) have been discussed in terms of the lattice energies and the hydration structure of the metal(III) ions in aqueous solution [15].

The solid state structure of $[YCl_2(thf)_5][YCl_4(thf)_2]$ (4) has been reported. The cation contains a pentagonal bipyramidal yttrium(III) centre with the thf ligands residing in the equatorial plane. In the octahedral anion, the chloride ligands are mutually *trans* [16].



(4)

Yttrium(III) chloride reacts with ε-caprolactone (L) to give the complex $[YL_3Cl_3]$ (5) which crystallizes as a solvated species. A *mer*-configuration has been established by X-ray diffraction methods, and the compound is an example of a 6-coordinate yttrium(III) centre; each ligand L is monodentate with Y–O distances in the range 2.269(2)-2.296(3)Å [17].



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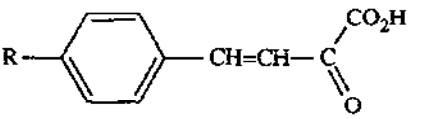
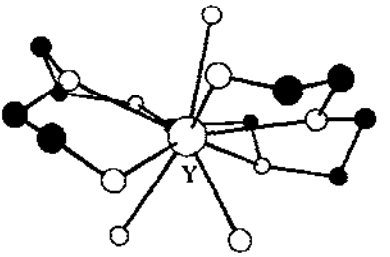


R = NMe_2 or OMe

(6)

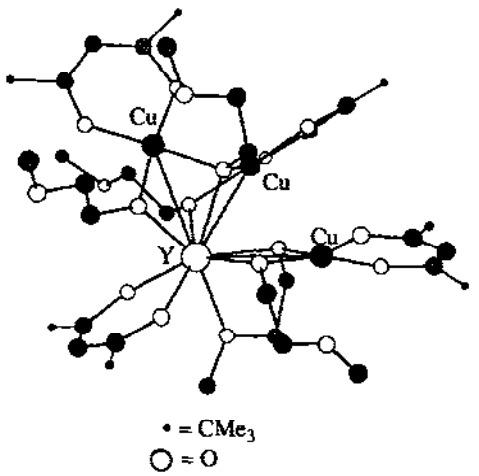
The preparation and properties of $[YL_3.nH_2O]$ (HL = 3-methoxybenzoic acid; n = 0-4) have been detailed [18]. The compounds YL_3, for HL = (6), have been prepared and studied by TG and DTA methods [19,20].

An investigation concerning polyether coordination to yttrium(III) and lanthanoid ions has focused upon differences in complex formation involving 18-crown-6 and pentaethylene glycol. For yttrium(III), only the reaction with the acyclic ligand has been studied, and the pentaethyleneglycol complex $[Y(H_2O)L]Cl_3.H_2O$ in which the cation is (7) has been prepared its crystal structure determined. The yttrium(III) centre is 9-coordinate; the geometry is that of a tricapped trigonal prism with the *O*-donor atoms of the glycol ligand occupying alternating prism and capping sites. Complexes of the same type, namely $[M(H_2O)L]Cl_3.H_2O$ have also been obtained for M = Sm-Lu, but for M = La-Pr, the products have the general formula $[MCl_2(H_2O)L]Cl.H_2O$ [21].



(7)

The reaction between $[\{Y(OCH_2CH_2OMe)_3\}_{10}]$, $[\{LCu(OCH_2CH_2OMe)\}_4]$ and $[\{L'Cu(OCH_2CH_2OMe)\}_4]$ where L = 2,2,6,6-tetramethyl-3,5-heptanedione and L' = 1,1,1,5,5,5-hexafluoroacetylacetone leads to the heterometallic complexes $[L_4Cu_3Y(OCH_2CH_2OMe)_5]$ (8), $[\{L'_2CuY(OCH_2CH_2OMe)_3\}_2]$ and $[L_2L'_2Cu_2Y(OCH_2CH_2OMe)_3]$. The new compounds have been characterized by elemental analysis, IR and electronic spectroscopies and X-ray analysis. Solution studies indicate that there is some tendency for the break up of the assemblies that are present in the solid state [22].



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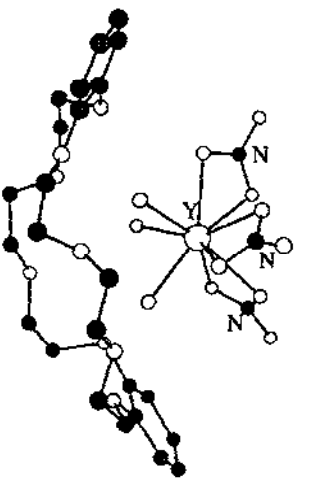
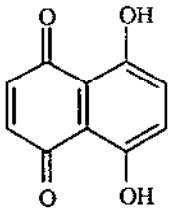


(9)

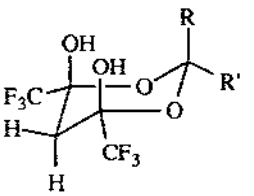
Complexes involving macrocyclic ether ligands include $[Y(NO_3)_3(H_2O)_3$(dibenzo-24-crown-8)] (9) which has been synthesized from Y_2O_3. Characterizations by IR spectroscopy and molar conductance measurements are complemented by a single crystal X-ray diffraction study. The yttrium(III) centre is 9-coordinate (tricapped trigonal prism) with three didentate nitrate ions and three water molecules comprising the inner-coordination sphere. The cyclic ether is hydrogen-bonded to the coordinated water molecules. The average $Y\text{-}O_{nitrate}$ and $Y\text{-}O_{water}$ distances are 2.426(4) and 2.344(4)Å respectively. The mismatch in sizes between the yttrium(III) ion and the macrocyclic cavity has been suggested as a reason for the preferential coordination to the nitrate ions

[23]. Complex formation between yttrium(III) nitrate and dibenzo-30-crown-10 has also been investigated [24].

The acid-base characteristics of ligand (**10**) in water-ethanol mixtures and the composition and molar absorptivities of complexes formed between yttrium(III) and (**10**) have been studied. The work also reports determinations of stability constants for these species [25].



(**10**)



R = CH_3 or CF_3
R' = CF_3 or CH_3

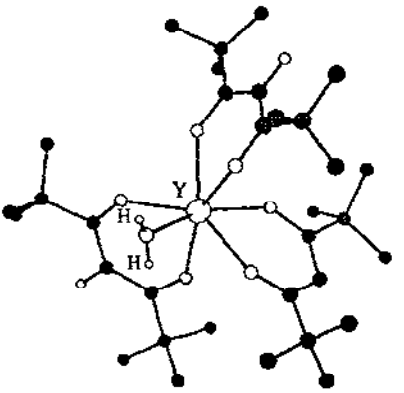
(**11**)

Yttrium(III) acetylacetonate has been prepared and variable temperature ^1H and ^{89}Y NMR spectroscopic studies show that the complex is a tetramer in solution. Attempts to crystallize the compound from hexane produced, instead, the hydrolysis product $[Y_4(\mu_3\text{-}OH)_2(\mu\text{-}acac)_6(acac)_4)]$. Its structure has been determined; the complex is centrosymmetric and each yttrium centre is 8-coordinate. Other methods of producing $[Y_4(\mu_3\text{-}OH)_2(\mu\text{-}acac)_6(acac)_4)]$ have been detailed — for example, vacuum thermolysis of solid $Y(acac)_3.3H_2O$. After recrystallization from benzene, the product obtained is $[Y_4(OH)_4(acac)_{10}].4C_6H_6$, the solid state structure of which has been elucidated [26].

The conjugate base of ligand (**11**), H_2L', is formed during the reaction of $[Y(Hfacac)_3]$, $Cu(OMe)_2$ and HL [HL = $CH(OH)(NMe_2)_2$] in thf by the cycloaddition of Hfacac (Hfacac = $CF_3C(O)CH_2C(O)CH_3$) and 1,1,1-trifluoro-2,2-propandiol; the latter is the hydration product of trifluoroacetone. The product is $[YCu(HL)_2(Hfacac)_2(O_2CCF_3)L']$ and details of the structure have been confirmed by the results of an X-ray diffraction analysis. The formation of $[L']^{2-}$ is unusual indeed but atempts to isolate the uncoordinated ligand have failed [27]. Complex formation between yttrium(III) and oximes, β-diketonates and polyaminopolycarboxylates has been studied and the results compared with those for lanthanoid(III) ions. Despite the fact that the ionic radius of Y^{3+} is similar to that Ho^{3+}, the complexation constants for the yttrium(III) complexes are similar to those of *early* lanthanoid(III) complexes. The relevance of the study to developing uses of yttrium(III) in organic reaction environments is discussed [28].

The combination of $^tBuC(O)CH_2C(O)^tBu$ (HL), yttrium(III) oxo isopropoxide (both in toluene) and the product of the reaction of metallic barium and $^iPrOH/EtOH$ leads to the formation of the first dimetallic barium yttrium oxo alkoxide — $[Y_4Ba_2(\mu_6\text{-}O)(\mu_3\text{-}OEt)_8L_6]$. Structural data show that the metal atoms form an octahedral cage around the oxygen centre; ethoxide ligands are in face-capping sites whilst each $[^tBuC(O)CHC(O)^tBu]^-$ ligand is chelating to a metal centre. The presence of ethoxide but not isopropoxide ligands is attributed to steric effects. Thermogravimetric analysis of the new alkoxide compound reveal loss of eight ethanol molecules between 100 and 200°C, loss of four HL molecules over the range 200-300°C and loss of the final two diketones between 300 and 400°C [29]. The reaction of $^tBuC(O)CH_2C(O)^tBu$ (HL) with yttrium(III)

isopropoxide leads, after sublimation of the initial product, to $[YL_3]$. Both the products have been characterized by IR and ^1H, ^{13}C and ^{89}Y NMR spectroscopies, mass spectrometry and TG and DT analyses. The anhydrous β-diketonate complex can also be prepared from yttrium(III) nitrate although $[YL_3(H_2O)]$ is the first product in the synthesis. Both $[YL_3]$ and $[YL_3(H_2O)]$ (**12**) have been crystallographically characterized. In the former, the Y(III) centre is confirmed as being 6-coordinate with Y-O distances in the range 2.128 to 2.298Å. In (**12**), there is hydrogen bonding between the coordinated H_2O molecule and the ketonic oxygen atoms, and this leads to dimer formation. For the 7-coordinate yttrium(III) centre, Y-O distances for the L^- ligands lie in the range 2.207 to 2.337Å with $Y\text{-}O_{water} = 2.334(9)$Å [30].



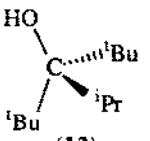
(**12**) O = O

Treatment of $[YL_3(H_2O)]_\infty$ (HL = 2,2,6,6-tetramethyl-3,5-heptanedione) with triglyme leads to the formation of $[\{YL_3\}_2(triglyme)]$ — the elimination of water is noteworthy. The product has been characterized by mass spectrometry and ^1H and ^{13}C NMR spectroscopies as well as X-ray analysis. The triglyme ligand bridges between the two metal centres, each of which is 8-coordinate with a distorted square antiprismatic geometry of *O*-donor atoms. The $Y\text{-}O_{diketonate}$ distances are less than the $Y\text{-}O_{triglyme}$ bond lengths [31].

Bulky alkoxide ligands have attracted some attention. The reaction of alcohol (**13**), HL, with $[Y\{N(SiMe_3)_2\}_3]$ in hexane (25°C) yields $[YL_3]$; it is a monomeric complex which sublimes at 160°C (10^{-3} mbar). Mass spectrometric and IR spectroscopic data are presented. Related monomeric and dimeric rare earth alkoxides are also described in this work [32]. Hermann *et al* have also investigated the reaction between $[Y\{N(SiMe_3)_2\}_3]$ and $EtOCH_2C^iPr_2OH$. The product is complex (**14**), characterized by mass spectrometry and IR and ^{13}C and ^{89}Y NMR spectroscopies; the ^{89}Y NMR spectrum exhibits a signal at δ +277.1 (308K in C_6D_6). Compound (**14**) sublimes at 95°C at 10^{-3} mbar. The alkoxide ligand is described as having a 'clinching' habit [33].

The reactions of $[Y\{N(SiMe_3)_2\}_3]$ with R_2CHOH, RMe_2COH and R_2MeCOH (R = CF_3) lead to a wide range of complexes — the exact nature of the product is dependent upon solvent. All the products have been characterized by elemental analysis, IR and multi-nuclear NMR spectroscopies, and mass spectrometry. Examples of ^{89}Y NMR chemical shifts are δ +93.66 for $[Y(OCMe_2R)_3(thf)_{2.5}]$, δ +66.44 for $[Y(OCMeR_2)_3(thf)_3]$ and δ +78.63 for the complex

[$Y(OCMeR_2)_3$(diglyme)]. The latter compound sublimes in tact whereas other compounds tend to undergo ligand-loss during the process. The crystal structures of [$Y(OCMeR_2)_3(thf)_3$] and [$Y(OCMeR_2)_3$(diglyme)] have been determined. In each, the yttrium(III) centre is octahedral with a *fac*-configuration [34].



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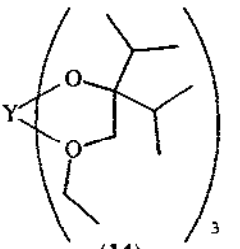
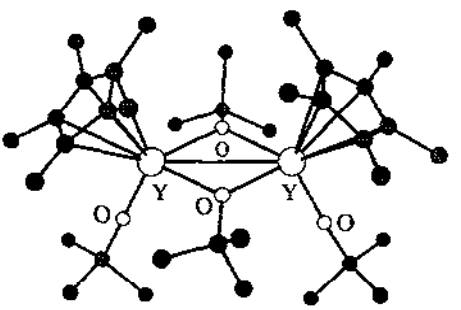


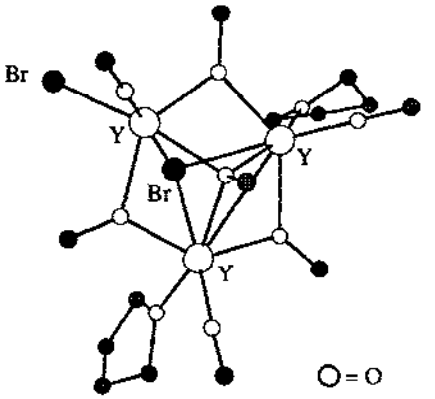
(14)

Reacting sodium or potassium cyclopentadienides with $[Y_3(O^tBu)_7Cl_2(thf)_2]$ leads to the formation of the alkoxy-bridged complexes $[(\eta^5\text{-}C_5R_5)_2Y_2(\mu\text{-}O^tBu)_2(O^tBu)_2]$ (R = H or Me, or R_5 = H_4Me or H_4SiMe_3). Three of the four complexes have been structurally characterized — (**15**) has R = Me, and the *cis* arrangement of the Cp rings is a common feature of all the compounds. An alternative route to these dimers is the reaction of $[YCl_3(thf)_n]$ with NaO^tBu and MC_5R_5 (M = K or Na) in toluene under reflux. A related indenyl complex has also been studied. Further reactions of these systems have been investigated and products include $[(^tBuO)Y(\mu\text{-}O^tBu)_3Li_5(\mu_3\text{-}O^tBu)_2(thf)(\mu\text{-}OCH_2CH_2OMe)_2]$ and $[(\eta^5\text{-}C_5H_4SiMe_3)_2Y(\mu\text{-}O^tBu)_2Li(thf)_2]$, both of which have been the subjects of X-ray analysis [35].



(15)

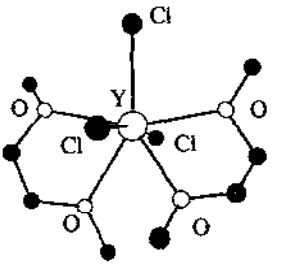
The reaction of yttrium trichloride and ten equivalents of NaO^tBu in thf leads to the formation of $[YNa_8(O^tBu)_{10}Cl]$. The yield is high, although the reaction conditions are critical. Structural analysis reveals that the YNa_8 core possesses a capped square antiprismatic geometry with the yttrium atom in the capping site. The chloride ion resides at the centre of the nonametal cavity (Y–Cl = 2.915(4)Å). Eight *tert*-butoxide ligands are in μ_3-bonding modes, one is terminally attached to the yttrium centre (Y–O = 2.044(7)Å), and one occupies a μ_4-position interacting solely with the sodium centres. The hydroxy-compound $[YNa_8(O^tBu)_{10}(OH)]$ has also been prepared and characterized. The structure is similar to that of the chloro analogue with the OH^- group interstitially sited. The results of ^1H NMR spectroscopic studies are presented [36].



Me groups from CMe_3 substituents omitted for clarity.

(16)

Several yttrium(III) alkoxides — $[Y_3(O^iPr)_9(HO^iPr)_2]$, $[Y_3(O^iPr)_7X_2(thf)_2]$ (X = Cl or Br) and $[Y_3(O^iPr)_3(AlMe_3)_2(thf)]$ — have been included within a wider ^{89}Y NMR spectroscopic study (see introduction). The crystal structure of $[Y_3(O^iPr)_7Br_2(thf)_2]$ (**16**) has been determined; the yttrium atoms are in a triangular array supported by a μ_3-bromide, a μ_3-isopropoxide ligand and three μ-isopropoxides. The second bromide is terminally attached to one metal centre [4]. Reactions between $[KAl(O^iPr)_4]$ and $YCl_3.3^iPrOH$ lead to the formation of $[\{YCl_2\{Al(O^iPr)_4\}.^3/_2{}^iPrOH\}_2]$ and $[\{Y\{Al(O^iPr)_4\}_2(\mu\text{-}Cl)(^iPrOH)\}_2]$, prolonged evacuation of which gives $[\{YCl\{Al(O^iPr)_4\}(\mu\text{-}Cl)\}_2]$ and $[\{Y\{Al(O^iPr)_4\}_2(\mu\text{-}Cl)\}_2]$. These in turn have been used as precursors for the compounds $[L_{3-x}Y\{Al(O^iPr)_4\}_x]$ (L = O^iPr or $Zr_2(O^iPr)_9$; x = 1 or 2). The new alkoxides have been characterized by elemental analysis, molecular weight determination and IR and ^1H, ^{13}C, ^{27}Al and ^{89}Y NMR spectroscopies [37].



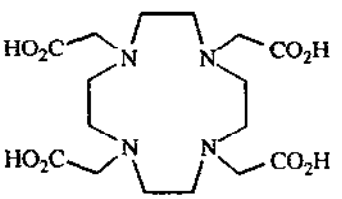
(17)

Further alkoxide chemistry includes the reaction of $AlMe_3$ with $[Y_3(O^tBu)_7Cl_2(thf)_2]$ which yields $[(^tBuO)(thf)Y\{(\mu\text{-}O^tBu)(\mu\text{-}Me)AlMe_2\}_2]$, $[(^tBuO)(thf)_2ClY\{(\mu\text{-}O^tBu)(\mu\text{-}Me)AlMe_2\}]$ and $[Y\{(\mu\text{-}O^tBu)(\mu\text{-}Me)AlMe_2\}_3]$. Proton NMR spectroscopic results are complemented by X-ray analyses. Octahedral yttrium(III) centres are a feature of all three complexes. $[Y\{(\mu\text{-}O^tBu)(\mu\text{-}Me)AlMe_2\}_3]$ may be considered to be a tris-$AlMe_3$ adduct of $[Y(O^tBu)_3]$, and similar descriptions are appropriate for the other species. A further compound to be described is $[YCl_3(dme)_2]$ (**17**),

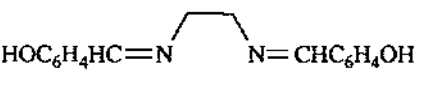
formed after recrystallization attempts from thf/dme solution. This possesses a 7-coordinate yttrium(III) centre in which two chloride ligands are axially sited. Treatment of $[Y_3(O^tBu)_7Cl_2(thf)_2]$ with $LiAlMe_4$ yields the complex $[\{(thf)_3Y(\mu_3\text{-}Cl)_3\}Y_3(\mu_3\text{-}O^tBu)(\mu\text{-}O^tBu)_3(O^tBu)_3(\mu_4\text{-}O)]$, the structure of which may be considered in terms of a $YCl_3(thf)_3$ unit capping a triangular array of yttrium atoms, the latter being supported by edge-bridging and one μ_3-butoxide ligands. The centre of the tetra-yttrium cavity holds an oxide ligand [38].

4.1.5 *Complexes with mixed donor atom ligands*

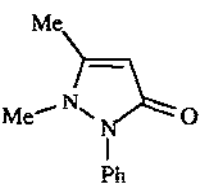
The preparation, thermal decomposition studies and structural investigations of the solid state complexes $H[Y(edta)].nH_2O$ have been reported [39]. Ligand (**18**), H_4L, reacts with freshly prepared yttrium(III) hydroxide (from YCl_3 and NaOH at pH 8.5-9.0) to yield white crystals of $Na[YL(H_2O)].4H_2O$. Structural data confirm that the ligand binds using an N_4O_4-donor set (Y-O = 2.316-2.327Å, Y-N = 2.633-2.666Å), the Y(III) centre being 9-coordinate overall with the water molecule occupying the ninth site ($Y\text{-}O_{water}$ = 2.435(3)Å) [40].



(**18**)



(**19**)



(**20**)

The preparation and characterization (IR and ESR spectroscopies, thermal analysis, magnetic susceptibility and electrical conductance measurements) of the yttrium(III) complex $[YL(HL)BiCl_3]$, H_2L = (**19**), have been detailed. The salicylideniminate ligands are *N,O*-donors [41].

Complex formation involving the Schiff's base 4-*N*-(4'-antipyrylmethylidene)amino-antipyrine, L, [antipyrine = (**20**)] has been a subject of study. Among a series of compounds reported is $[YL_2(NO_3)_2]NO_3$ which has been characterized by elemental and thermogravimetric analyses, electrical conductance measurements, and IR, UV-VIS and ^1H NMR spectroscopies. In the series of complexes as a whole, the UV-VIS spectroscopic results show that the $n\rightarrow\pi^*$ band of the ligand (free ligand 348 nm) is red-shifted to 345-342 nm, whilst the $\pi\rightarrow\pi^*$ band is shifted to 227-222 nm (free ligand 247 nm). Ligand L is proposed to be tridentate, bonding through the two carbonyl *O*-donor atoms and one azomethine *N*-donor atom. The nitrate ions are proposed as being monodentate [42].

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