Coordination Chemistry Reviews, 124 (1993) 51–62

3. Yttrium

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CONTENTS

INTRODUCTION

Previously, a review of the coordination chemistry of yttrium has not been included in this series. However, its inclusion here provides a useful comparison to the coordination chemistry of its Periodic group member scandium, the coordination chemistry of which was surveyed for 1990 in Chapter 2 of this volume. There is an obvious relevance to the solid state chemistry of high T_c superconductors. No attempt has been here to cover the latter topic but the reader is guided to several relevant review articles which have appeared during 1990: a review of electronic conductors including high temperature superconductors [1], an overview of crystallographic and microstructural aspects of $YBa_2Cu_3O_7$ [2], and a review of and the theory of oxygen ordering in high T_c superconductors [3].

This review is organised according to the oxidation state of yttrium and according to the donor atoms involved in complexation. I should like to acknowledge assistance from the Cambridge Crystallographic Data Base who have allowed access to coordinates for structures **(5)**, **(6)**, **(9)**, and **(14)**-**(18)**.

3.1 YTTRIUM (III)

3.1.1 Complexes with halide and pseudohalide ligands

When Y_2O_3 is heated with an excess of solid ammonium chloride, YOCl is formed which upon dissolution in 2M HCl produces $[YCl_2(H_2O)_6]Cl$. Structural characterisation of this complex

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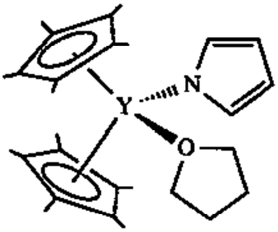
confirms a square antiprismatic coordination sphere with D_{4d} symmetry. Important distances are Y-Cl = 2.740 (1) Å and Y-O = 2.333 (3), 2.360 (3) and 2.361 (3) Å [4]. The reaction of yttrium(III) iodide and powdered yttrium with impure Y_2O_3 is described in Section 3.2 [5]

The two hydrates $Y(NCS)_3.4.5H_2O$ and $Y(NCS)_3.2.5H_2O$ have been prepared by careful dehydration of the hexahydrate. All the complexes have been structurally characterised. Thermochemical data ($\Delta H_{solution}$, ΔH°_f, $\Delta H^{\circ}_{dehydration}$, and lattice energies) have been determined [6].

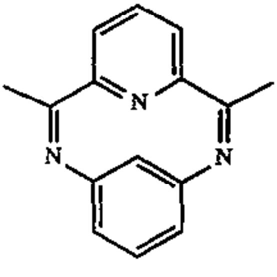
3.1.2 *Complexes with nitrogen donor ligands*

The reaction of $Na[NC_4H_4]$ with $Cp^*_2YCl(thf)$ yields the pyrrolyl complex (**1**) as colourless crystals. The complex has been characterised by ^1H and ^{13}C NMR spectroscopy and the analogous complex $(\eta^5\text{-}Cp)_2LuL(thf)$ has been characterised by X-ray crystallography [7].

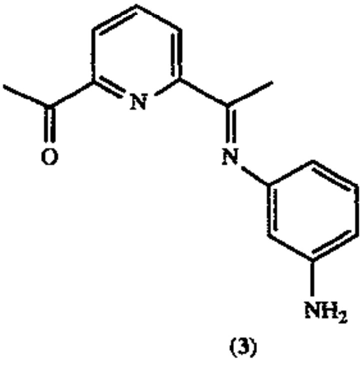


(**1**)

A series of papers has appeared detailing the coordination of $[HB\{C_3N_2H_3\}_3]^-$ to yttrium(III) and lanthanides. Tridentate $[HB\{C_3N_2H_3\}_3]^-$ is present in $Y(HB\{C_3N_2H_3\}_3)_2L$ where L^- = benzoate, acetate or tropolonate [8], $Y(HB\{C_3N_2H_3\}_3)(PhC(O)CHC(O)Me)$ [9], $Y(HB\{C_3N_2H_3\}_3)(PhC(O)CHC(O)Ph)$ [9], $Y(HB\{C_3N_2H_3\}_3)_2(O_2C\text{-}C_6H_4\text{-}4\text{-}R)$ (R = NMe_2 or NO_2) [10] and dimeric $[Y(HB\{C_3N_2H_3\}_3)(O_2C\text{-}C_6H_4\text{-}4\text{-}^tBu)_2]_2$ [10]. These compounds will be discussed further in Section 3.1.4. A phenanthroline containing yttrium(III) complex [11] will also be described in Section 3.1.4.



(**2**)



(**3**)

It has been reported that the complex $YL(NO_3)_3.3H_2O$ for L = (2) is obtained from the template condensation of 2,6-diacetylpyridine and *m*-phenylenediamine in the presence of Y(III) ions. Infrared, UV-vis, ^{1}H NMR and mass spectroscopic data have been reported to support this formulation [12]. However, a [2+2] product might seem to be more likely. In the absence of yttrium(III), the reaction leads to the template agent (3) [12].

The lanthanoid(III) phthalocyanine complexes Ln(Pc)(OAc) are sensitisers for the photoreduction of Methyl Viologen chloride ($MVCl_2$) in methanol for Ln = Y as well as Sm, Gd, Yb or Lu with irradiation in the visible region. The absorbance due to Y(Pc)(OAc) at $\lambda = 670$ nm decreases upon the addition of $MVCl_2$; isosbestic points are at 652 and 681 nm. An absorbance attributed to an adduct has been observed. The reaction pathway shown in Scheme I has been proposed; L = triethanolamine and $k_q = 4.5 \times 10^9$ $M^{-1}s^{-1}$ for the quenching step which generates the radical [13].

$$\mathrm{Y(Pc)(OAc)} + \mathrm{MV}^{2+} \overset{K}{\rightleftharpoons} [\mathrm{Y(Pc)(OAc)}{-}{-}{-}{-}\mathrm{MV}^{2+}]$$

$$\begin{array}{rcl}
\mathrm{Y(Pc)(OAc)} & \xrightarrow{h\nu} & {*^1}\mathrm{Y(Pc)(OAc)} \\
{*^1}\mathrm{Y(Pc)(OAc)} & \longrightarrow & {*^3}\mathrm{Y(Pc)(OAc)} \\
{*^3}\mathrm{Y(Pc)(OAc)} & \longrightarrow & \mathrm{Y(Pc)(OAc)} \\
{*^3}\mathrm{Y(Pc)(OAc)} + \mathrm{MV}^{2+} & \xrightarrow{k_q} & \mathrm{Y(Pc)(OAc)}^{\cdot+} + \mathrm{MV}^{\cdot+} \\
\mathrm{Y(Pc)(OAc)}^{\cdot+} + \mathrm{L} & \longrightarrow & \mathrm{Y(Pc)(OAc)} + \mathrm{L}^{\cdot+}
\end{array}$$

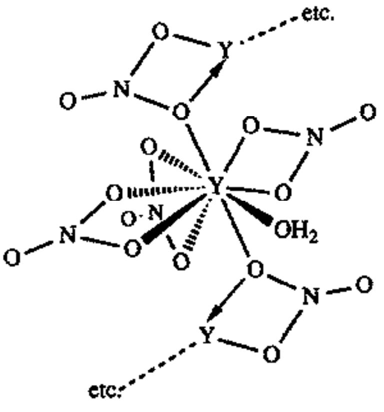
Scheme I

3.1.3 *Complexes with phosphorus donor ligands*

Metal complexes with neutral phosphorus donors have been reviewed and a section surveying yttrium compounds has been included [14].

3.1.4 *Complexes with oxygen donor ligands*

The hygroscopic nitrate $Y(NO_3)_3.H_2O$, (4), has been prepared by the thermal decomposition of the hexahydrate and has been the subject of a single-crystal X-ray diffraction study. The yttrium(III) atom in (4) is 9-coordinate and is sited in an irregular coordination sphere. Each coordination polyhedron is linked to the next via common nitrate-*O* donor atoms. This generates continuous chains running parallel to the crystallographic *c* axis [15]. The thermal decompostion of the hydrated yttrium(III) selenite $Y_2(OH)(SeO_3)_{2.5}.3H_2O$ has been a subject of investigation [16].



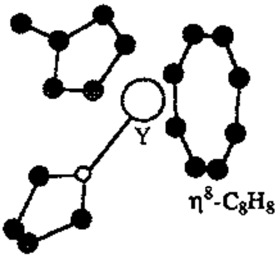
Distances:

Y–O(H$_2$O) = 2.312 (9)Å

Y–O(nitrate) range from
2.312 (9) to 2.468 (7) Å

(4)

Complexes of yttrium(III) exhibiting coordinated thf ligands include (η^8-cyclooctatetraenyl)(η^5-methylcyclopentadienyl)(tetrahydrofuran)yttrium(III), **(5)**, which has been crystallographically characterised [17] and complex **(1)** described in Section 3.1.2 [7]. The complex $Cp_2YMe(thf)$ reacts with $Re_2H_8(PMe_2Ph)_4$ in thf at room temperature to give $Cp_2Y(thf)Re_2H_7(PMe_2Ph)_4$, **(6)**. This heterometallic species has been characterised by X-ray diffraction; the Re-Re distance is 2.576 (1) Å. One Re-Y edge is open (4.186 Å) while the other Re-Y distance is 3.090 (2) Å and this is consistent with the presence of bridging hydride ligands; in the $^1H\{^{31}P\}$ NMR spectrum, coupling of J_{YH} = 7.2 Hz is observed. When **(6)** is dissolved in toluene, $Cp_2YRe_2H_7(PMe_2Ph)_4$ is formed [18].



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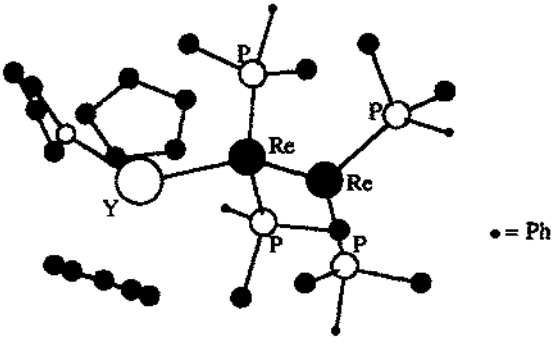
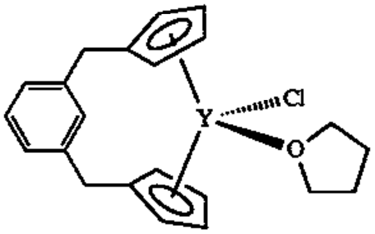


(6)

Complex **(7)**, Y(L)Cl(thf), is prepared from Na_2L and YCl_3 in thf at –50°C. The complex **(7a)** where L^{2-} is the 1,4-analogue of the xylylene ligand in **(7)** has also been reported. Both yttrium(III) complexes have been characterised by infrared, ^1H NMR, and mass spectroscopy. Results of XPS studies show that the binding energy of the metal atom is 159.4 eV compared to 159.8 eV in Cp_2YCl [19].



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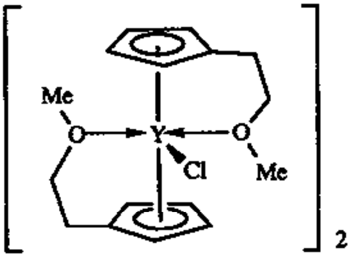
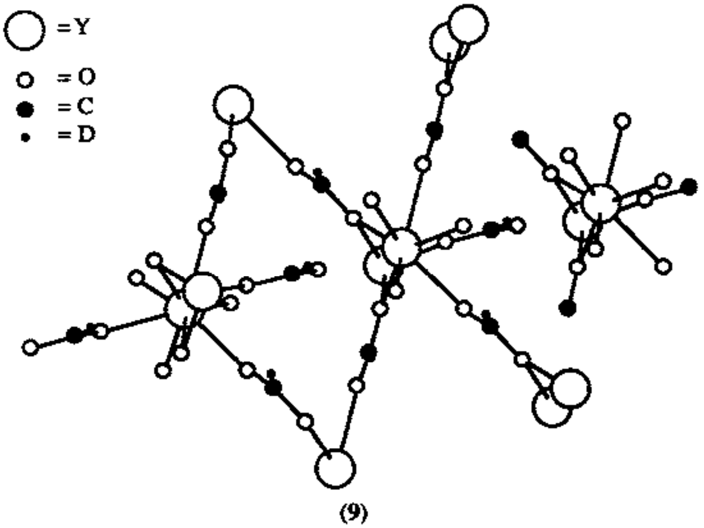


(8)

Reaction of $[CH_3OCH_2CH_2\text{-}\eta^5\text{-}C_5H_4]Na$ with YCl_3 in thf at ambient temperatures yields the dimeric complex (8). Infrared, mass, ^1H and ^{13}C NMR spectroscopic and XPS data have been reported; the IR spectral XPS results indicate that each ether-functionalised cyclopentadienyl ligand is indeed coordinated to the yttrium(III) centre through the *O*-donor atom and this has been confirmed by a preliminary X-ray diffraction study [20].

Yttrium(III) oxide is important as an additive in new ceramic materials and striving to obtain Y_2O_3 of a uniform (fine) particle size has led to a study of the thermal decomposition of $[NH_4][Y(C_2O_4)_2].H_2O$. The rapid addition of an aqueous solution of ammonia to a highly acidic solution of $Y_2(C_2O_4)_3$ gives $[NH_4][Y(C_2O_4)_2]$, the particle size of which is dependent upon the exact conditions (eg. addition time) of component combination. A mechanism for the precipitation has been discussed. Thermal decomposition of $[NH_4][Y(C_2O_4)_2].H_2O$ the particle size of which is controlled and uniform provides a route to finely powdered Y_2O_3, the particle size of which is again uniform. Preparing Y_2O_3 in this manner is advantageous; obtaining good quality (with respect to particle size) fine crystals of the oxide on an industrial scale by the thermal decomposition of $Y_2(C_2O_4)_3$ is difficult [21]. Further discussion of the preparation of $[NH_4][Y(C_2O_4)_2].H_2O$ on an industrial scale has also appeared [22].



(9)

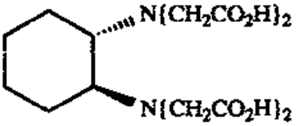
A high resolution neutron powder diffraction study of anhydrous $Y(O_2CD)_3$, **(9)**, has been carried out. The yttrium(III) atom is in a tricapped trigonal prismatic coordination polyhedron with $3m$ symmetry. Pertinent distances are $Y\text{-}O_{prism}$ = 2.505 (7) and 2.465 (3) Å and $Y\text{-}O_{prism\text{-}cap}$ = 2.465 (3) Å [23].

Treatment of aqueous $YCl_3.6H_2O$ with $K[HB\{C_3N_2H_3\}_3]$ and 2-hydroxy-2,4,6-*cyclo*-heptatrien-1-one leads to a yellow precipitate which has been identified as $Y(HB\{C_3N_2H_3\}_3)_2L$ (L^- = tropolonate). Infrared and ^1H NMR spectroscopic data have been reported. The related complexes $Y(HB\{C_3N_2H_3\}_3)_2L$ where L^- = benzoate or acetate have also been prepared. Crystallographic data are available for the $Yb(HB\{C_3N_2H_3\}_3)_2L$ (L^- = tropolonate or benzoate); the metal is in a distorted square antiprismatic environment [8]. The yttrium(III) complexes $Y(HB\{C_3N_2H_3\}_3)L$ in which HL = $PhC(O)CH_2C(O)Me$ or $PhC(O)CH_2C(O)Ph$ have been synthesised and spectroscopically characterised. $Y(HB\{C_3N_2H_3\}_3)(PhC(O)CHC(O)Me)$ is a colourless complex with m. pt. = 232-234°C; $Y(HB\{C_3N_2H_3\}_3)(PhC(O)CHC(O)Ph)$ is yellow, m. pt. = 285-290°C [9]. The monomeric compounds $Y(HB\{C_3N_2H_3\}_3)_2(O_2C\text{-}C_6H_4\text{-}4\text{-}R)$ (R = NMe_2 or NO_2) and the dimer $[Y(HB\{C_3N_2H_3\}_3)(O_2C\text{-}C_6H_4\text{-}4\text{-}{}^tBu)_2]_2$ have also been prepared and characterised by spectroscopic methods, elemental analysis and molecular weight determination. Reasons for the differences in the observed stoichiometry of Y:benzoate for R = NMe_2 or NO_2 versus R = ^tBu have been assessed. The differences cannot easily be explained in terms of either electronic or steric factors but it is considered possible that the hydrophobic nature of the ^{t}butyl group may make the complex $[Y(HB\{C_3N_2H_3\}_3)(O_2C\text{-}C_6H_4\text{-}4\text{-}{}^tBu)_2]_2$ particularly insoluble thereby causing precipitation of this dimeric complex during the early stages of the reaction [10].

For the dicarboxylic acids $H_2L = HO_2C(CH_2)_nCO_2H$ (n = 1, 2, 3), recrystallisation of $Y(OH)_3$ in a solution of H_2L leads to the complexes $Y_2L_3.7H_2O$. For n = 4–8, complexes $Y_2L_3.xH_2O$ are obtained from the reaction of YCl_3 with $[NH_4]_2[L]$. Upon heating, the compounds containing ligands with n = 1, 2, 3, 7, or 8 lose all the water of crystallisation; for n = 4, 5, or 6, only some water is lost and mono- or dihydrates are formed prior to decomposition to Y_2O_3 [24]. Treatment of $Y(OH)_3$ with $CH_3(CH_2)_7CO_2H$ yields $Y(O_2C(CH_2)_7CH_3)_3$ which is highly soluble in organic solvents [25]. Yttrium(III) and lanthanide complexes of 3,4-dinitrobenzoate have been synthesised. Solubilities in water at 298 K are all of the same order of magnitude, i.e. 10^{-3} mol dm^{-3}. Infrared spectroscopic data for both hydrated and dehydrated forms of the species have been reported; dehydration occurs without isomerisation from the nitro- to nitrito-form of the ligand. The thermal decomposition of the complexes has been investigated over a temperature range of 273 to 573 K; above 573 K, all the nitrobenzoates decompose explosively [26].

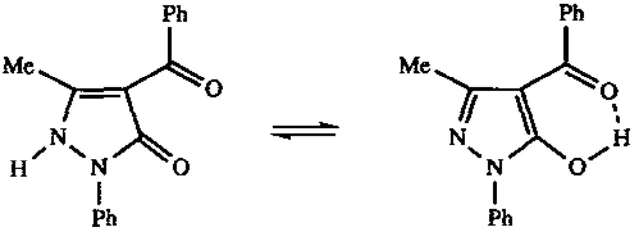
The calcium(II)-like behaviour of Y(III) ions has been employed in modelling calcium-binding proteins with the advantage that yttrium has an NMR active nucleus. A range of carboxylate complexes has been prepared in an initial study; these include use of the carboxylic acids $N\{CH_2CO_2H\}_3$, $N\{CH_2CH_2CO_2H\}_3$, $HOCH_2CH_2N\{CH_2CO_2H\}_2$, $\{HO_2CCH_2\}_2NCH_2CH_2OCH_2CH_2OCH_2CH_2N\{CH_2CO_2H\}_2$, $\{HO_2CCH_2\}_2NCH(Me)CH_2N\{CH_2CO_2H\}_2$, H_4edta, and **(11)**. ^{89}Y NMR spectroscopic shifts for these complexes have been measured. Results for these known complexes have then been compared with those obtained for the complex of Y(III) ions with pike parvalbumin; parvalbumin is a protypical calcium-binding protein in muscle.

It has been concluded that ^{89}Y NMR spectral characteristics may be used to distinguish between quite similar binding sites and that Y(III) appears to be a good probe for Ca(II) [27].

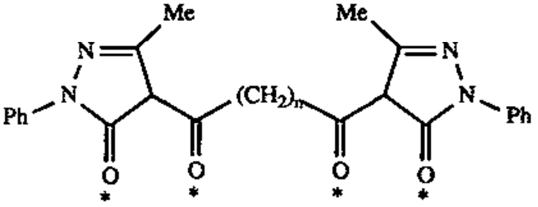


(11)

The complex $YL_2(NO_3)_3$ where L = (**12**) has been prepared from the reaction of L with $Y(NO_3)_3$ in ethanol under conditions of reflux. The product is yellow and has been characterised by elemental analysis, a cryoscopic molecular weight determination, molar conductivity measurements, and infrared spectroscopy. The infrared spectrum of the complex exhibits absorptions at 1620, 1640, and 408 cm^{-1} assigned to the C=O(ring), C=O(side-chain), and Y-O bonds respectively. Ligand (**12**) is proposed to function as a bidentate donor via both carbonyl groups. For the free ligand L, the N-H absorption appears at 3140 cm^{-1} and this is absent in the infrared spectrum of the complex implying that in complexing to the yttrium(III) centre, the ligand is transformed from a keto- to enol-tautomer. Related complexes of La, Pr, Nd, Sm, Eu, Gd, Tb, and Dy have also been reported [28].



keto-enol (**12**)



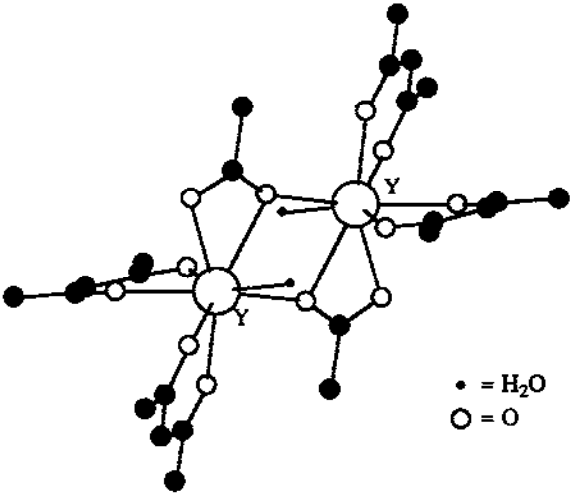
* = coordination site

(13)

A mixed phenanthroline/diketonate complex of yttrium(III) has been prepared. The ligand H_2L, (**13**), is dissolved in alcohol by adding ammonia and adjusting the pH to 5-6 by the addition of dilute nitric acid. Upon the addition of an ethanol solution of phen and $Y(NO_3)_3$ such that the ratio of (**13**) : phen : Y(III) = 1.5 : 1.0 : 1.05, a complex is precipitated that has been formulated as $Y_2L_3(phen)_2$. It is characterised in its infrared spectrum by a carbonyl absorption at 1532 cm^{-1}

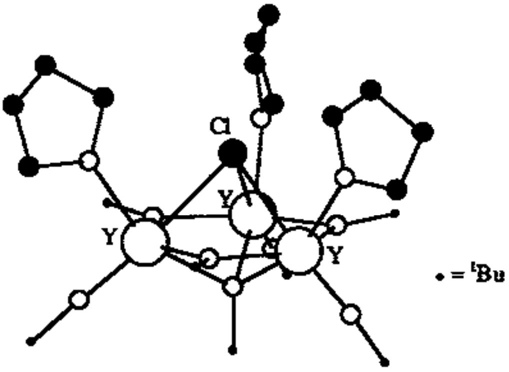
compared to 1554 cm^{-1} observed for the free ligand (**13**). From this and from UV-vis and ^1H NMR spectroscopy and from the results of thermal analysis, it is proposed that the two Y(III) centres are bridged by the L^{2-} ligands, each being coordinated via an O-O donor set to each yttrium atom. Each metal atom is eight coordinate within an O_6N_2-coordination sphere [11].

A number of publications reporting the preparation and characterisation of yttrium alkoxide and aryloxide complexes adds to the information available in this area. Interest in these compounds arises from the involvement of yttrium oxides in materials, for example in high T_c superconductors. Two methods of preparing $Y(OAr)_3$ have been described in *Inorganic Synthesis* [29]. The treatment of YCl_3 with $[Li(\mu\text{-}OAr)(OEt_2)]_2$ ($OAr = O\text{-}C_6H_2\text{-}2,6\text{-}{}^tBu_2\text{-}4\text{-}R$; R = Me, ^tBu) in thf under reflux gives $Y(OAr)_3$. A second route is the reaction of $Y\{N(SiMe_3)_2\}_3$ with ArOH. These aryloxides are air and moisture sensitive, but are thermally stable under inert conditions. $Y(O\text{-}C_6H_2\text{-}2,6\text{-}{}^tBu_2\text{-}4\text{-}Me)_3$ is a white solid (m.pt. = 178-180°C) and is soluble in hydrocarbon solvents; cryoscopic data indicate a monomeric species. The structure of $Y(O\text{-}C_6H_2\text{-}2,6\text{-}{}^tBu_2\text{-}4\text{-}Me)_3$ has previously been confirmed crystallographically and this X-ray determination represents the first proven example of an yttrium(III) aryloxide [30]. $Y(O\text{-}C_6H_2\text{-}2,4,6\text{-}{}^tBu_3)_3$ is also an air and moisture sensitive white solid (m. pt. = 140-142°C) [29]. The silyloxy complex $[Y(OSiPh_3)_3(thf)_3]$.thf has been synthesised and structurally characterised; it exhibits an octahedrally coordinated Y(III) ion with a *fac*-arrangement of ligands. The complex is isostructural with its cerium(III) and praseodymium(III) analogues [31].



(14)

During attempts to prepare $M(O^iPr)_3$ (M = Sc, Y, In, or Yb), the complex $M_5(O^iPr)_5(\mu_5\text{-}O)(\mu_3\text{-}O^iPr)_4(\mu_2\text{-}O^iPr)_4$, (structure (**11**) in the preceding chapter of this volume) has been isolated. For M = Y, the complex is isolated as the product of the reaction of yttrium powder with isopropanol in the presence of $HgCl_2$ catalyst under conditions of reflux [32]. The reaction of $Y_5O(O^iPr)_5$ with acetylacetone under anhydrous conditions leads to the dinuclear complex $Y_2(\mu_2\text{-}OAc)_2(acac)_4(H_2O)_2$, (**14**). The complex has been characterised by ^1H and ^{13}C NMR spectroscopy and by X-ray diffraction. It is a centrosymmetric dimer; each yttrium centre is 8-coordinate with a distorted square antiprismatic environment. The Y-O distances range from 2.271 (5) to 2.505 (5) Å and the Y------Y separation is 4.054 (1) Å. The stability of **(14)** in solution is limited unless additional acetylacetone is present [33].



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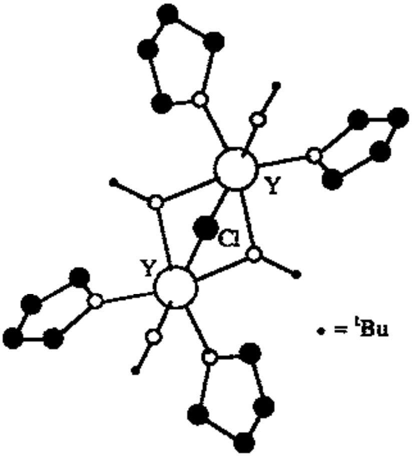

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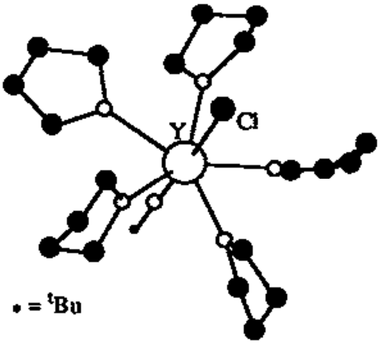
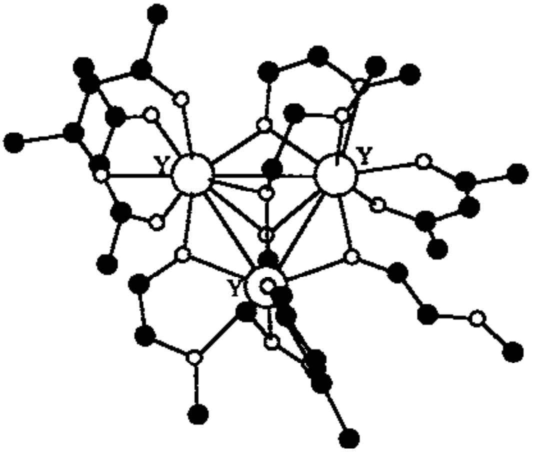


(17)

The cationic alkoxide complexes $[Y_3(O^tBu)_7Cl(thf)_3]^+$, **(15)**, $[Y_2(O^tBu)_4Cl(thf)_4]^+$, **(16)**, and $[Y(O^tBu)Cl(thf)_5]^+$, **(17)**, have been synthesised [34]. They are members of a new class of complex and each has been structurally characterised by X-ray diffraction. Complexes **(15)** and **(16)** are prepared as the tetraphenylborate salts by the reaction of $Ag[BPh_4]$ with $Y_3(O^tBu)_7Cl_2(thf)_2$, **(15)** being favoured at a reaction temperature of 0°C. When $Y_3(O^tBu)_7Cl_2(thf)_2$ is treated with $Na[BPh_4]$, [**(17)**]$[BPh_4]$ is produced. This complex is formed in high yield by the reaction of YCl_3 with $Na[O^tBu]$ and $Na[BPh_4]$. In cation **(15)**, the Y-O distances within the triangular framework range from 2.27 (1)-2.31 (1) Å while for the μ_3-O^tBu group, Y-O = 2.38 (1), 2.36 (1), and 2.35 (1) Å. The dinuclear species **(16)** possesses a C_2 axis and the Y-O_{bridge} distances are 2.24 (2) ad 2.26

(2) Å. In cation **(17)**, the Y(III) atom is within a pentagonal bipyramidal coordination sphere with the five thf ligands occupying the equatorial sites; the average Y-O_{thf} distance is 2.41 (1) Å.

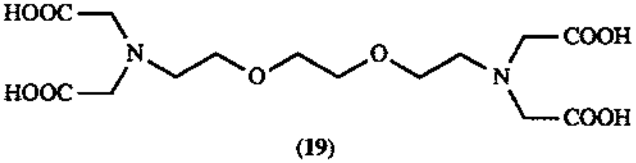
Treating $\{Y(OC_2H_4OMe)_3\}_{10}$ with 30 equivalents of $Cu(acac)_2$ leads not to a heteronuclear species but to $Y_3(\mu_3,\eta^2\text{-}OC_2H_4OMe)_2(\mu_2,\eta^2\text{-}OC_2H_4OMe)_2(\mu_2,\eta^1\text{-}OC_2H_4OMe)(acac)_4$ **(18)**. The diamagnetic complex **(18)** has been characterised by infrared spectroscopy and by X-ray diffraction. Pertinent distances are av. Y-$O_{(\mu_3\text{-}L)}$ = 2.47 Å, av. Y-$O_{(\mu_2\text{-}L)}$ = 2.26 Å, av. Y-$O_{(Me)}$ = 2.545 Å, and av. Y-$O_{(acac)}$ = 2.304 Å [35].



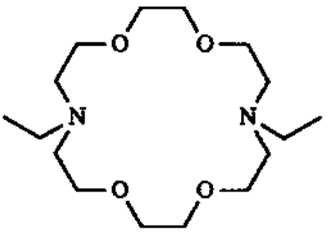
(18)

3.1.5 *Complexes with mixed nitrogen-oxygen donor ligands*

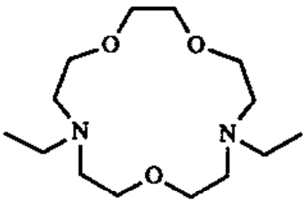
The interactions of the carboxylic acids $N\{CH_2CO_2H\}_3$, $N\{CH_2CH_2CO_2H\}_3$, $HOCH_2CH_2N\{CH_2CO_2H\}_2$, $\{HO_2CCH_2\}_2NCH_2\text{-}CH_2OCH_2CH_2OCH_2CH_2N\{CH_2CO_2H\}_2$, $\{HO_2CCH_2\}_2NCH(Me)CH_2N\{CH_2CO_2H\}_2$, H_4edta, and **(11)** with yttriumIII) ions have been described in Section 3.1.4 [27]. In related work, complexes of Y(III) ions with ligands **(19)**, **(20)**, and **(21)** have been prepared and studied. In each case, a 1:1 complex has been characterised. For the macrocyclic ligands, it has been shown that in Y**(21)**, all the donor atoms of the ligand are involved in bonding to the metal ion. In Y**(20)**, the ligand is fluxional and this property has been interpreted in terms of a mismatch between large macrocyclic-cavity and relatively small metal ion. Ligand **(20)** undergoes a process in which it wraps and unwraps itself around the metal ion. The study emphasises the importance of matching the radius of the metal atom with the hole-size of the macrocyclic ligand [36].



(19)



(20)



(21)

3.2 YTTRIUM (II)

The carbide $Y_{10}I_{13}C_2$ is formed at 800°C by the reaction of yttrium(III) iodide and powdered yttrium with impure Y_2O_3. The source of the carbon is perceived as arising from impurities in the yttrium oxide. This phase has not been obtained by using more obvious sources of carbon. The structure of $Y_{10}I_{13}C_2$ consists of centred $Y_6I_{12}C$- and empty Y_6I_8-type clusters which are condensed via edge sharing. The resultant double and single chains are cross-linked by iodine atoms [5].

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