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Structures and catalytic properties of complexes of *bis*(diphenylphino)methane dioxide with scandium and lanthanide trifluoromethane sulfonates

John Fawcett [a], Andrew W.G. Platt [b,*]

[a] Department of Chemistry, The University of Leicester, Leicester LE1 7RH, UK
[b] School of Sciences, Staffordshire University, College Road, Stoke-on-Trent ST4 2DE, UK

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Abstract

Complexes of scandium and lanthanide triflates with *bis*(diphenylphosphino)methane dioxide ($Ph_2P(O)CH_2P(O)Ph_2$=L) have been prepared and have the general formulae $Sc(OTf)_3L_3$, $La(OTf)_3L_41.5H_2O$ and $Ln(OTf)_3L_3H_2O3.5H_2O$. The structures show that the phosphine oxides act as bidentate chelates, and whilst triflate ions are not coordinated to the metals some hydrogen bond with the methylene protons in the ligand. The solution properties have been investigated using electrospray mass spectrometry and ^{31}P NMR spectroscopy. The catalytic acylation of anisole has been investigated for the ytterbium and scandium complexes with the latter showing no activity.

Keywords: Catalytic properties; Complexes; Bis(diphenylphino)methane dioxide

1. Introduction

The catalytic properties of scandium and lanthanide triflates have been extensively studied in a variety of aqueous and non-aqueous solvents [1–8]. The Diels–Alder reactions of ytterbium coordination complexes with polyethers have been examined [9] and more recently chiral coordination complexes of scandium triflate have been shown to catalyse enantioselective Michael addition reactions [10], and aldol reactions in aqueous media [11]. Chiral coordination complexes of lanthanides, including triflate complexes have been recently reviewed [12] and chiral lanthanum complexes have shown enantiomeric excesses of over 80% in the addition of RLi and silylcyanation of aldehydes, respectively [13,14].

We recently reported the preparation, structures and catalytic behaviour of scandium and lanthanide triflate complexes with triphenylphosphine oxide [15]. Whilst catalysis proceeded at a slower rate compared with the uncomplexed salts, there were some marginal increases in the selectivity. We thus felt it of interest to extend this study to complexes containing chelating phosphine oxide ligands. These should show interesting differences in structures compared with corresponding triphenylphosphine oxide complexes, by constraining the donor set into bidentate ligands. They should also bind more strongly to the metal ions and thus restrict access of substrates to the metal in catalytic reactions, thereby favouring the least sterically demanding catalytic pathway.

2. Results and discussion

The complexes differ from those of the triphenylphosphine oxide reported previously in that the triflate ion is expelled from the primary coordination sphere of the metal. This is expected in view of the stronger coordinating ability of the bidentate ligands. The overall coordination number appears to be related to the ionic radius of the metal. Thus the small Sc^{3+} ion is six co-

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* Corresponding author. Tel.: +44-1782-29-4784; fax: +44-1782-74-5506.
E-mail address: a.platt@staffor.qc.uk (A.W.G. Platt).

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ordinate whilst Lu^{3+} has a coordination number of 7. Although only poor quality crystals of the lanthanum complex were obtained they show that the complex contains $[LaL_4]^{3+}$ with a coordination number of 8 at the metal. The analytical data for the La complex agree well with an anhydrous composition and it is possible that the hydrated crystals formed during recrystallisation are not representative of the bulk sample.

2.1. Solid state structures

The single crystal X-ray structures of the cations in $Sc(OTf)_3L_3$, $Lu(OTf)_3L_34.5H_2O$ and $La(OTf)_3$-$L_41.5H_2O$ are shown in Figs. 1–3, respectively. Selected bond lengths and angles are shown in Table 1 and details of the data collection and refinement in Table 2. All show the ligand acting as a bidentate chelate with the triflate ion not coordinated to the metal. The scandium ion is in an approximately octahedral environment whilst the lutetium is coordinated in a distorted pentagonal bipyramidal manner with the water occupying one of the equatorial sites. For the lanthanum complex, crystals from different crystallisation attempts all gave poor quality data. The final R_1 value (0.12) appears to be related only to disorder of the triflate ions as the metal complex ion refines as expected with relatively low anisotropic displacement values. Partial occupancy water molecules in the lattice equate to three molecules in the unit cell. The coordination geometry about the La ion is a distorted square antiprism.

The higher coordination numbers in the lutetium and lanthanum complexes simply reflect their larger ionic radii. The Sc–O and Lu–O distances are marginally

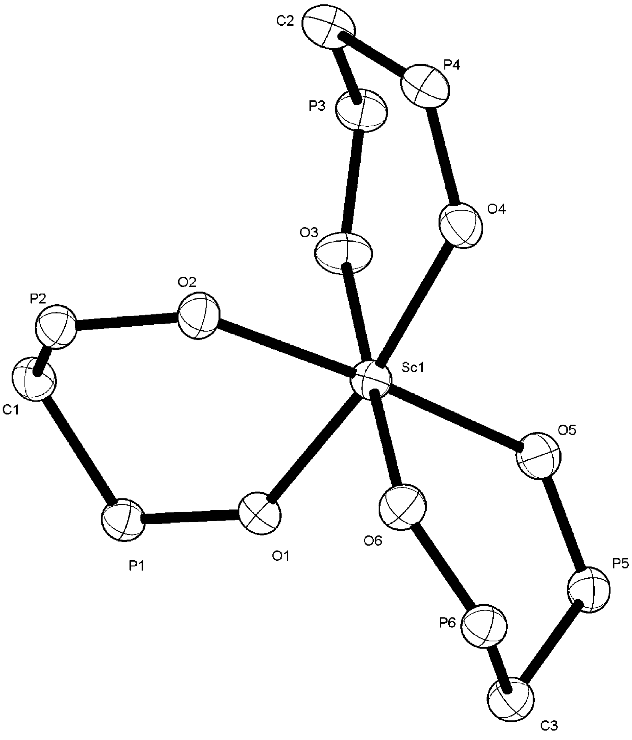


Fig. 1. The environment of the Sc atom in the $[ScL_3]^{3+}$ cation showing the atom label scheme and 50% displacement ellipsoids. The phenyl rings, H atoms and triflate ions have been omitted for clarity.

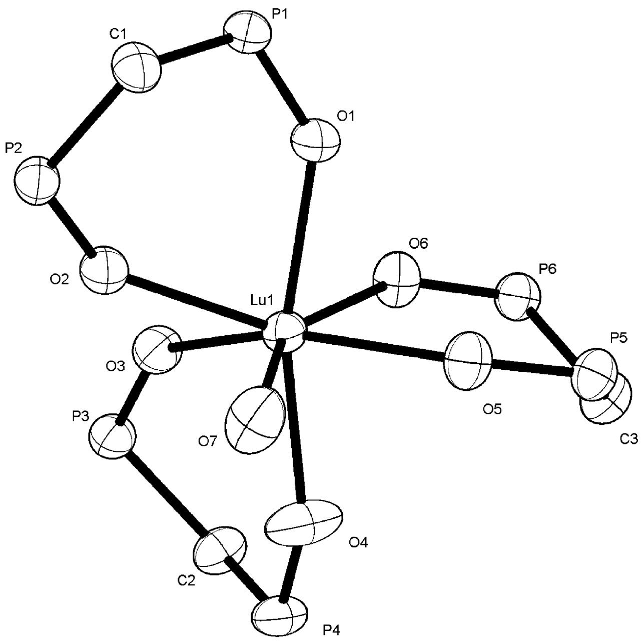


Fig. 2. The environment of the Lu atom in the $[LuL_3{\cdot}H_2O]^{3+}$ cation showing the atom label scheme and 50% displacement ellipsoids. The phenyl rings, H atoms, triflate ions and lattice waters have been omitted for clarity.

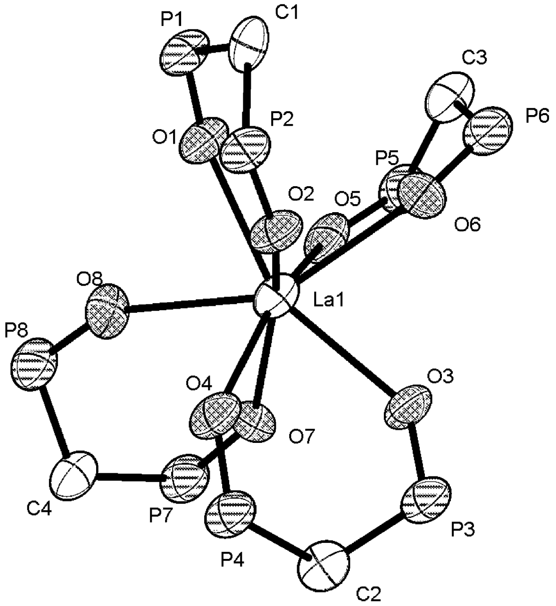


Fig. 3. The environment of the La atom in the $[LaL_4]^{3+}$ cation showing the atom label scheme and 50% displacement ellipsoids. The phenyl rings, H atoms, triflate ions and lattice waters have been omitted for clarity.

Table 1
Selected bond lengths (Å) and angles (°) for $Sc(OTf)_3L_3$, $Lu(OTf)_3L_3H_2O \cdot 3.5H_2O$ and $La(OTf)_3L_4 \cdot 1.5H_2O$

	Sc	Lu	La
Bond lengths			
M(1)–O(6)	2.0723(14)	2.282(3)	2.576(8)
M(1)–O(4)	2.0849(15)	2.254(3)	2.464(9)
M(1)–O(3)	2.0868(15)	2.270(2)	2.513(8)
M(1)–O(5)	2.0881(14)	2.249(3)	2.468(9)
M(1)–O(2)	2.1035(14)	2.281(3)	2.489(8)
M(1)–O(1)	2.1056(14)	2.248(2)	2.490(8)
M(1)–O(7)		2.294(3)	2.488(8)
M(1)–O(8)			2.483(8)
P(1)–O(1)	1.5095(14)	1.516(3)	1.471(9)
P(2)–O(2)	1.5133(15)	1.504(3)	1.497(8)
P(3)–O(3)	1.5108(15)	1.506(3)	1.498(9)
P(4)–O(4)	1.5086(15)	1.508(3)	1.519(9)
P(5)–O(5)	1.5097(15)	1.506(3)	1.509(9)
P(6)–O(6)	1.5121(15)	1.507(3)	1.485(9)
P(7)–O(7)			1.517(9)
P(8)–O(8)			1.510(8)
Bond angles			
O(6)–M(1)–O(4)	94.93(6)	89.69(11)	128.1(3)
O(6)–M(1)–O(3)	178.52(6)	76.80(9)	74.5(3)
O(4)–M(1)–O(3)	85.30(6)	77.16(10)	74.9(3)
O(6)–M(1)–O(5)	85.62(6)	78.70(9)	70.9(3)
O(4)–M(1)–O(5)	95.70(6)	82.12(11)	153.3(3)
O(3)–M(1)–O(5)	92.91(6)	147.77(9)	96.3(3)
O(6)–M(1)–O(2)	91.60(6)	132.08(9)	71.3(3)
O(4)–M(1)–O(2)	87.17(6)	117.55(11)	72.1(3)
O(3)–M(1)–O(2)	89.87(6)	72.63(9)	96.7(3)
O(5)–M(1)–O(2)	176.16(6)	139.49(9)	134.5(3)
O(6)–M(1)–O(1)	92.58(6)	79.76(10)	80.2(3)
O(4)–M(1)–O(1)	170.69(6)	165.23(12)	120.9(3)
O(3)–M(1)–O(1)	87.34(6)	109.97(10)	154.6(3)
O(5)–M(1)–O(1)	90.36(6)	85.68(10)	77.3(3)
O(2)–M(1)–O(1)	87.12(6)	77.20(9)	72.4(3)
P(1)–O(1)–M(1)	136.14(9)	139.79(15)	147.7(5)
P(2)–O(2)–M(1)	137.18(9)	146.18(16)	142.2(5)
P(3)–O(3)–M(1)	134.33(9)	142.48(16)	139.4(5)
P(4)–O(4)–M(1)	133.98(9)	146.41(18)	143.0(5)
P(5)–O(5)–M(1)	134.60(9)	142.21(16)	145.7(5)
P(6)–O(6)–M(1)	133.69(9)	141.81(16)	145.9(5)
P(7)–O(7)–M(1)			147.0(5)
P(8)–O(8)–M(1)			144.7(5)
P(1)–C(1)–P(2)	115.14(11)	111.28(19)	112.9(7)
P(3)–C(2)–P(4)	112.49(12)	111.9(2)	116.5(7)
P(6)–C(3)–P(5)	111.89(11)	113.6(2)	114.2(7)
P(7)–C(4)–P(8)			115.7(7)

longer than in the corresponding Ph_3PO complexes [15] as a result of the increased steric effects on constraining the donor set into the chelate ring. One of the triflate ions hydrogen bonds to one of the methylene hydrogens of the ligands whilst the other two triflates act as ‘free’ ions. The close contacts of 2.373 Å in the scandium and 2.571 Å in the lutetium complex are below the sum of the Van der Waals radii for oxygen and hydrogen (2.72 Å). It is interesting to compare the structures of the chelate rings with similar *tris*-chelated ion, $[NaL_3]^+Br^- \cdot 3H_2O$ [16]. Here the six membered ring adopts a classical chair form. In our complexes, however, the ring is flattened in the MO_2P_2 region, the difference between the two is illustrated schematically in Fig. 4. We attribute this to increased electrostatic repulsion between the metal ion and the positively charged phosphorus centres. Thus the Na···P distance of 3.69 Å in the NaBr complex reflects a balance between the stability of the ring conformation and the electrostatic repulsion between Na^+ and the P atoms, the relatively low magnitude of the charges allowing the conformational stability of the ring to outweigh the repulsions. For the Sc and Lu complexes the magnitude of the charge and, in the case of scandium, the smaller ionic radius, increase these repulsions, forcing the ring to adopt a configuration which increases the M···P separation. Molecular modelling calculations carried out on the $[ScL]^{3+}$ fragment give the same configuration for the chelate ring, with a calculated angle of 144.6° between the mean ScO_2P_2 plane and the P_2C plane compared with a measured angle of 129.6°. Similarly the measured bond lengths between scandium and oxygen are slightly longer than the calculated values, and the O–Sc–O angles are all narrower than calculated. All these differences are probably due to the increased steric strain imposed by the presence of three ligands actual structure. Unfortunately we are unable to carry out ab initio calculations on larger structures such as $[ScL_3]^{3+}$. Molecular mechanics calculations of interatomic distances give values in reasonable agreement with those found in the crystal structure, but tending to slightly overestimate the Sc–O distance at an average of 2.117 Å compared with a range of 2.0706(14)–2.1034(14) Å in the structure. The complete structure generated by these calculations, however, fails to give the observed conformation of the ring, predicting a chair conformation instead.

2.2. *Solution properties and catalytic activity*

The speciation of the complexes in solution has been analysed using electrospray mass spectrometry [17]. The results for representative complexes in acetone are shown in Table 3 and indicate that the *tris*-chelate structure is retained to a large extent in this solvent, the base peak being represented by ML_3 based cations in all cases. Ligand redistribution occurs to a much smaller extent than in complexes with triphenylphosphine oxide, as would be expected for more stable chelated structures. The behaviour in methanol is different with ligand redistribution reactions giving relatively higher proportions of LnL_4 and LnL_2 containing cations. One possible explanation for this observation is that dissociation of phosphine oxide may be promoted by hydrogen bonding with methanol, thus favouring redistribution reactions. Hydrogen bonding interactions of PO groups with water [18] and alcohols [19] are well

Table 2
Crystal data and structure refinement data for $[ScL_3][OTf]_3$, $[LuL_3H_2O][OTf]_3 3.5H_2O$ and $[LaL_4][OTf]_3 1.5H_2O$

Empirical formula	$C_{78}H_{66}F_9O_{15}P_6S_3Sc$	$C_{78}H_{75}F_9LuO_{19.50}P_6S_3$	$C_{103}H_{91}F_9LaO_{18.5}P_8S_3$
Formula weight	1741.27	1952.35	2305.72
Temperature (K)	180(2)	180(2)	150(2)
Crystal system	orthorhombic	triclinic	triclinic
Space group	$P2(1)2(1)2(1)$	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions			
a (Å)	17.9701(6)	13.8163(5)	15.842(4)
b (Å)	20.9565(8)	15.7120(5)	17.821(5)
c (Å)	21.1338(8)	21.3191(7)	20.511(6)
α (°)	90	83.0820(10)	86.442(4)
β (°)	90	72.6360(10)	82.380(4)
γ (°)	90	88.4040(10)	66.435(4)
V (Å^3)	7958.8(5)	4384.8(3)	5261(2)
Z	4	2	2
D_{calc} (Mg m^{-3})	1.453	1.479	1.438
Absorption coefficient (mm^{-1})	0.377	1.391	0.668
$F(000)$	3576	1978	2326
Crystal size (mm)	0.32 × 0.23 × 0.17	0.19 × 0.17 × 0.13	0.38 × 0.14 × 0.05
θ Range for data collection (°)	1.37–27.0	1.01–26.00	1.25–25.00
Index ranges	$-22 \leq h \leq 22$; $-26 \leq k \leq 26$; $-26 \leq l \leq 26$	$-17 \leq h \leq 16$; $-19 \leq k \leq 19$; $-26 \leq l \leq 26$	$-18 \leq h \leq 18$; $-21 \leq k \leq 21$; $-24 \leq l \leq 24$
Reflections collected	66 907	34 480	33 542
Independent reflections	17 365 [$R_{int} = 0.0427$]	17 072 [$R_{int} = 0.0183$]	18 172 [$R_{int} = 0.1788$]
Completeness (%)	100	99.1	98.2
Maximum and minimum transmission	0.94 and 0.89	0.89 and 0.83	0.894 and 0.639
Data/restraints/parameters	17 365/0/1009	17 072/0/1132	18 172/0/1285
Goodness-of-fit on F^2	0.942	1.073	0.933
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0336$, $wR_2 = 0.0651$	$R_1 = 0.0379$, $wR_2 = 0.1051$	$R_1 = 0.1201$, $wR_2 = 0.2588$
R indices (all data)	$R_1 = 0.0448$, $wR_2 = 0.0673$	$R_1 = 0.0405$, $wR_2 = 0.1066$	$R_1 = 0.2093$, $wR_2 = 0.3160$
Absolute structure parameter	$-0.011(19)$		
Largest difference peak and hole (e Å^{-3})	0.430 and -0.315	1.570 and -0.690	2.291 and -1.743

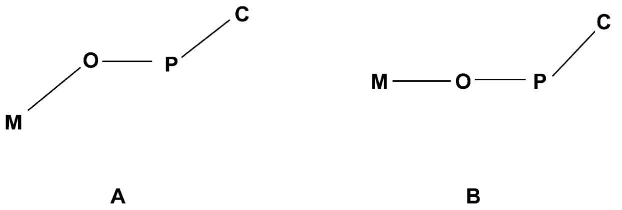


Fig. 4. Schematic representations of the ring conformation change in $[NaL_3]^+$ (A) and $[ScL_3]^{3+}$, $[LaL_4]^{3+}$ and $[LuL_3H_2O]^{3+}$ (B).

established in the solid state. The spectra for the lutetium complex illustrate the typical differences between the two solvents and are shown in Fig. 5. The peak at $m/z = 644$ is present in the spectra of all complexes, and occurs in the spectrum of the free ligand itself. In all spectra this peak is broad and has no resolved isotope peaks, and thus probably arises from a multiply charged ion. We assign this as $[L_3+Na+NH_4]^{2+}$ on the basis of the presence of both ammonium

Table 3
Positive ion electrospray mass spectral data [a] for $M(OTf)_3L_3$ in acetone

	La [b]	Dy	Yb	Lu	Sc
ML_3^{3+c}	462.4(100)	470.7(100)	473.9(55)	474.5(100)	431.0(100)
$ML_2(OTf)^{2+}$	560.0(5)	572.5(40)	577.3(20)	578.0(< 5)	513.2(60)
ML_4^{3+}	601.4(25)	609.3(50)	612.7(25)	613.2(< 5)	
$ML_3\text{–}H^{2+}$	693.2(30)	705.4(< 5)	710.7(5)	711.3(25)	
$ML_3(OTf)^{2+}$	768.1(50)	780.6(60)	785.7(100)	786.1(50)	721.5(50)
$ML_4(OTf)^{2+}$	976.5(< 5)	988.7(40)	993.7(20)	994.1(< 5)	
$ML_2(OTf)_2^+$	1269.0(< 5)	1294.1(10)	1304.2(5)	1305.0(< 5)	1175.0(10)

[a] The m/z values quoted are for the most intense peak observed in the isotope profile. The values are within 0.5 Da of the theoretical m/z.

[b] $La(OTf)_3L_4$.

[c] The ligand itself shows the following peaks in the +20 V spectrum in methanol: $[L+H]^+$ 417.2(100), $[L_2+Na+NH_4]^{2+}$ 436.5(5), $[L+Na]^+$ 439.1(10), $[L_3+Na+NH_4]^{2+}$ 644.4(10), $[L_2+H]^+$ 833.3(10), $[L_2+NH_4]^+$ 850.3(10), $[L_2+Na]^+$ 855.2(10).

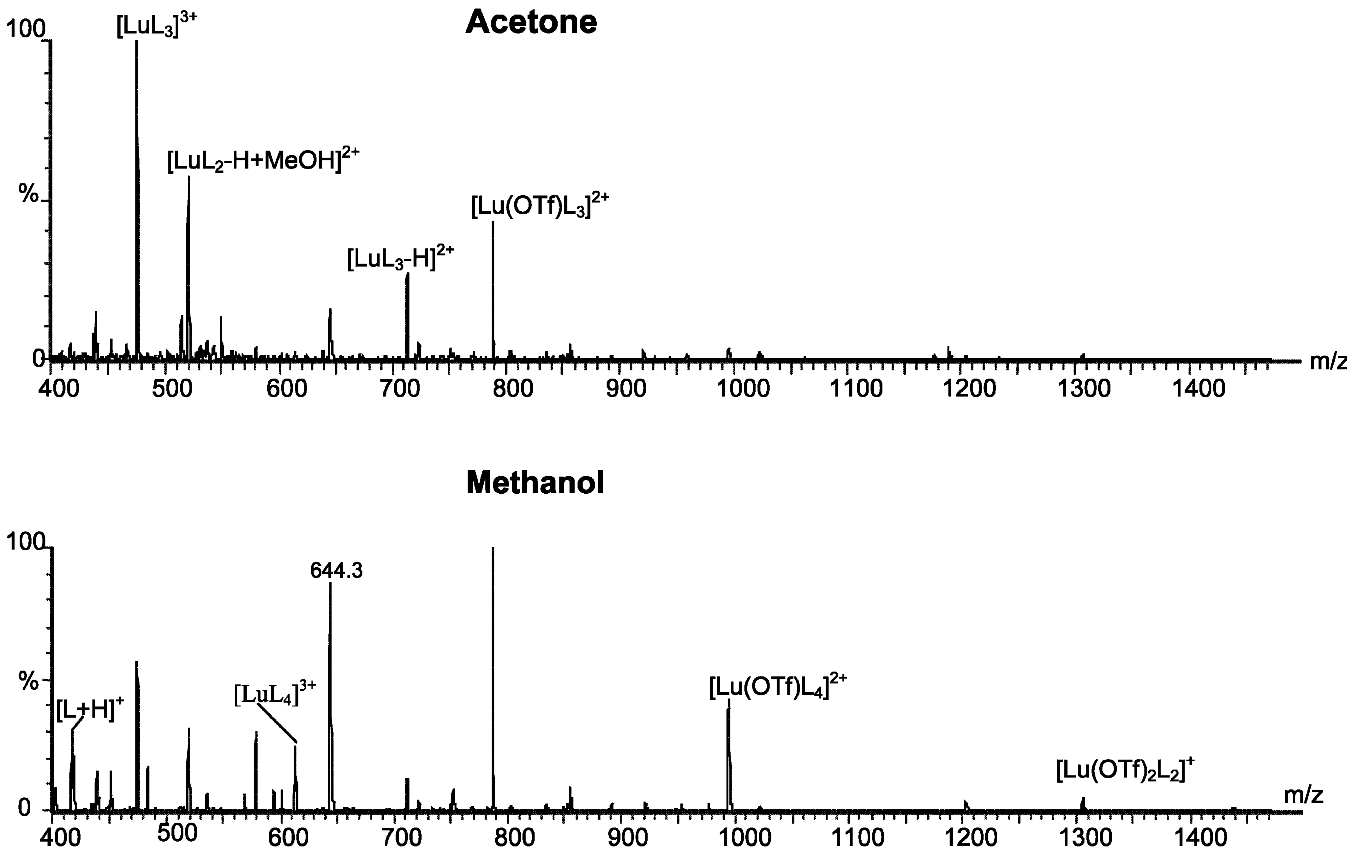
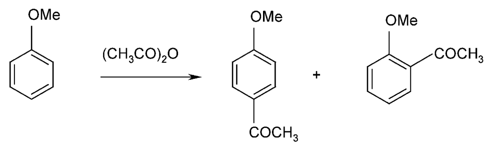


Fig. 5. Positive ion electrospray mass spectra of $M(OTf)_3L_3$ in acetone and methanol.

and sodium ions in the mobile phase (see Table 3) and the fact that at higher cone voltages this peak becomes less intense and a similar broad peak at $m/z = 436.5$ due to $[L_2+Na+NH_4]^{2+}$ appears, this declustering being common at higher cone voltages. The presence of methanol containing cations in the spectra run in acetone is due to the use of methanol as the mobile phase.

A preliminary examination of the behaviour of the scandium and ytterbium complexes as catalysts for the Friedel Crafts acylation of anisole with acetic anhydride was undertaken. The ytterbium complex showed some catalytic activity whilst $Sc(OTf)_3L_3$ displayed no activity at all. This difference is almost certainly due to the lack of access to the primary coordination sphere in the ScL_3^{3+} cation. Dissociation of the coordinated water or the phosphine oxide would both increase access to the metal. The ^{31}P NMR spectra show that the later certainly occurs. On addition of free ligand to nitromethane solutions of $Sc(OTf)_3L_3$ two resonances were seen at δ 41.2(complex) and δ 23.1(free ligand). An identical experiment carried out with $Lu(OTf)_3L_3H_2O$ (δ 37.0 ppm in nitromethane) produced a single broadened signal at δ 32.1 ($\Delta\nu_{1/2} = 60$ Hz) indicative of rapid exchange between free and bound ligand. It thus is apparent that $[LnL_3H_2O]^{3+}$ is sufficiently labile in solution to allow access of substrates to the metal as part of the catalytic process. In the scandium complex the higher charge density of Sc^{3+} is presumably responsible for its stronger binding to the ligand. Comparative experiments of the catalytic activity and selectivity of scandium triflate its triphenylphosphine oxide complex and $Yb(OTf)_3L_3H_2O$ were carried out. The results are summarised in Table 4. On refluxing overnight in nitromethane only a low conversion of 4% was attained but with some enhanced selectivity over scandium triflate and its triphenylphosphine oxide

Table 4
Catalytic acylation of anisole



Catalyst	Mol%	Conversion (%)	4:2 Isomer ratio
$Sc(OTf)_3 \cdot 9H_2O$ [a]	0.6	40	20:1
$Sc(OTf)_3(Ph_3PO)_4$ [a]	0.6	15	30:1
$Yb(OTf)_3L_3H_2O$	0.1	4	50:1

[a] Data from Ref. [14].

complex under the same conditions. The lower mol% of the ytterbium complex was necessitated due to its low solubility.

3. Experimental

3.1. Mass spectrometry

Electrospray mass spectra were obtained by the EPSRC National Mass Spectrometry Service Centre at the University of Wales, Swansea as described previously [15]. Assignments are based on the *m/z* ratio and the comparison between observed and calculated isotope distribution patterns which are particularly characteristic for some of the lanthanides.

3.2. X-ray crystallography

All data were collected on a Bruker APEX 2000 CCD diffractometer. Full spheres of diffraction data were collected using graphite monochromated Mo Kα radiation ($\lambda = 0.7107$ Å). Data were corrected for Lorentz and polarisation effects. Empirical absorption corrections were applied to all data. The structures were solved by Patterson methods and refined by full-matrix least squares on F^2 using SHELXTL [20]. All carbon bonded hydrogen atoms were included in calculated positions (C–H = 0.96Å) with isotropic displacement parameters set to 1.2 U_{eq}(C). The hydrogen atoms of water molecules were not included in the refinements. All non-hydrogen atoms were refined with anisotropic displacement parameters. For the Lu structure two phenyl rings were refined as disordered and for the both the La and Lu structures triflate ions were modelled with disorder.

3.3. Molecular modeling studies

The ab initio calculations were carried out using the SPARTAN V (version 4.1) package on a dedicated Silicon Graphics work station using an STO basis set.

3.4. Preparative work

The complexes were prepared by mixing methanol solutions of the metal salts with solutions of the ligand. The resulting precipitates were collected washed with a little methanol and dried. Yields are between 80 and 90% giving complexes with the characteristic colour of the lanthanide ions. Some representative examples are given below.

3.4.1. $Sc(OTf)_3L_3$

Scandium triflate (0.30 g 0.5 mmol) in 5 ml methanol was added to a solution of the ligand (0.42 g 1.01 mmol) in 5 ml methanol. The resulting precipitate was filtered washed with methanol and dried to give 0.55 g (94%) white solid. Analysis expected (found) C53.80 (53.35); H 3.83 (4.04)%.

3.4.2. $La(OTf)_3L_4$

As above, lanthanum triflate (0.35 g 0.50 mmol) and the ligand (0.42 g 1.01 mmol) gave 0.53 g (94%) of the complex. Analysis expected (found) C54.94 (55.17); H 3.94 (4.10)%.

3.4.3. $Lu(OTf)_3L_3 \cdot H_2O \cdot 3.5H_2O$

As above, lutetium triflate (0.36 g 0.49 mmol) and the ligand (0.42 g 1.01 mmol) gave 0.55g (85%) of the complex. Analysis expected (found) C 47.98 (48.21); H 3.87 (3.90)%.

Crystal suitable for single crystal X-ray work formed spontaneously during the precipitation of the complexes with the exception of the lanthanum complex which was recrystallised from ethanol.

Catalytic reactions were carried out on 5 ml of approximately 2 M solutions of the reagents in nitromethane. The reactions were heated to reflux and monitored by GC MS using a Carlo Erba MFC 500 gc linked to a Trio 1000 mass spectrometer.

4. Supplementary material

Crystallographic data for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 193678, 193679 and 193680 for the Sc, Lu and La complexes, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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