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Synthesis and molecular structures of lanthanocene amide complexes and their catalytic activity for addition of amines to nitriles

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ABSTRACT

Reaction of $(CH_3C_5H_4)_2LnCl(THF)$ with NaNHAr in a 1:1 molar ratio in THF afforded the amide complexes $(CH_3C_5H_4)_2LnNHAr(THF)$ [(Ar = 2,6-$Me_2C_6H_3$, Ln = Yb (**I**), Y (**III**); Ar = 2,6-$^iPr_2C_6H_3$, Ln = Yb (**II**)]. X-ray crystal structure determination revealed that complexes **I–III** are isostructural. The central metal in each complex coordinated to two methylcyclopentadienyl groups, one amide group and one oxygen atom from THF to form a distorted tetrahedron. Complexes **I–III** and a known complex $(CH_3C_5H_4)_2YbN^iPr_2(THF)$ **IV** all can serve as the catalysts for addition of amines to nitriles to monosubstituted *N*-arylamidines. The activity depended on the central metals and amide groups, and the active sequence follows the trend **IV** ≈ **III** < **I** < **II**.

1. Introduction

Lanthanide amide complexes have found wide applications in homogeneous catalyses. For example, lanthanide amides are active catalysts for a variety of olefin and alkyne transformations, including hydroamination/cyclization [1,2], hydroalkoxylation/cyclization [3], ethylene polymerization [4,5], hydrosilylation [6], the addition of terminal alkynes to nitriles [7], the monocoupling reaction of isocyanides with terminal alkynes [8]. They are also found to be the catalysts for the polymerization of polar monomers, such as methylmethacrylate [9–11], phenylisocyanate [12], ε-caprolactone and lactide [13,14], as well as for the Tishchenko reaction [15,16], guanylation of amines with carbodiimides [14], amidation of aldehydes with amines [17], and Mannich-type reaction [18]. etc.

Amidines are of interest as the structural units with wide utility in drug design and as synthons for the synthesis of heterocyclic compounds. Addition of amines to nitriles is a highly atomic economic approach for the formation of monosubstituted amidines. However, no efficient catalyst for this transformation has been found till 2008. Very recently, we have reported the ytterbium amide complex supported by a bridged bis(amidinate) ligand can serve as an efficient catalyst for addition of amines to nitriles to monosubstituted *N*-arylamidines [19]. To address the effect of ancillary ligand on the activity of lanthanide amide complexes, cyclopentadienyl anions were chosen as the candidate as they are the versatile ligands in organometallic chemistry of lanthanide metals. Although a variety of cyclopentadienyl-stabilized lanthanide amides have been reported [20–24], no example concerning the catalytic activity for addition of amines to nitriles has been found. Thus, a series of new biscyclopentadienyl lanthanide amides $(CH_3C_5H_4)_2LnNHAr(THF)$ [(Ar = 2,6-$Me_2C_6H_3$, Ln = Yb (**I**), Y (**III**); Ar = 2,6-$^iPr_2C_6H_3$, Ln = Yb (**II**)] were synthesized and their catalytic activity for addition of amines to nitriles were tested. The catalytic activity of a known amide complex, $(CH_3C_5H_4)_2YbN^iPr_2(THF)$ **IV** was also examined to assess the influence of amide groups.

It was found that all these complexes examined can serve as the catalysts for addition of amines to nitriles to monosubstituted amidines and the activity depended on the ancillary ligands, the amide groups, and the central metals. The active sequence follows as $-N^iPr_2$ < –NH(2,6-$Me_2C_6H_3$) < –NH(2,6-$^iPr_2C_6H_3$) for the amide groups; Y < Yb for the central metals and methylcyclopentadienyl ligand < bridged bisamidinate ligand for the ancillary ligands. Here we report the results.

2. Results and discussion

2.1. Syntheses and molecular structures of complexes *I–III*

The reaction of $(CH_3C_5H_4)_2YbCl(THF)$, which was prepared in situ from the reaction of anhydrous $YbCl_3$ with 2 equiv of $Na(CH_3C_5H_4)$ [10], with one equiv of NaNH(2,6-$Me_2C_6H_3$) was conducted in THF. The reaction went smoothly and the chlorine can be

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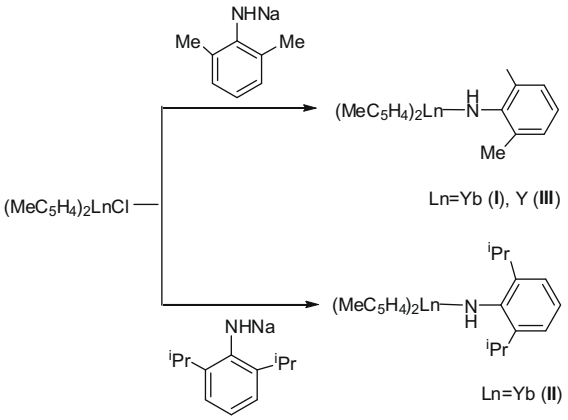


Scheme 1.

simply replaced by the amide group to give the corresponding amide complex **I** as green crystals upon crystallization from a mixture of THF and diethyl ether in good yield (Scheme 1). Complex **I** was confirmed by an X-ray diffraction study.

The same reaction with NaNH(2,6-$^{i}Pr_2C_6H_3$) afforded the amide complex **II** in good yield (Scheme 1). To address the influence of central metals on the activity of amide complexes, the preparation of yttrium complex with –NH(2,6-$Me_2C_6H_3$) group was also tried by the methathesis reaction of $(CH_3C_5H_4)_2YCl(THF)$ with NaNH(2,6-$Me_2C_6H_3$) in a 1:1 molar ratio. After workup, the expected complex **III** was isolated as colorless crystals in 42% yield (Scheme 1).

Complexes **I–III** are soluble in THF, DME, and diethyl ether. In their IR spectra, the strong absorptions of N–H stretch at about 3470 cm^{-1}, phenyl stretch at approximate 1600 cm^{-1} and methylcyclopentadienyl stretch at about 1450 cm^{-1} were observed. The acceptable NMR spectra for complexes **I** and **II** are difficult to be obtained because of the paramagnetic of ytterbium. ^{1}H NMR spectra of complex **III** shows all the chemical shifts assigned to the methylcyclopentadienyl group, amide group and solvated THF molecule. The molecular structures of **I–III** were further determined by single-crystal X-ray diffraction.

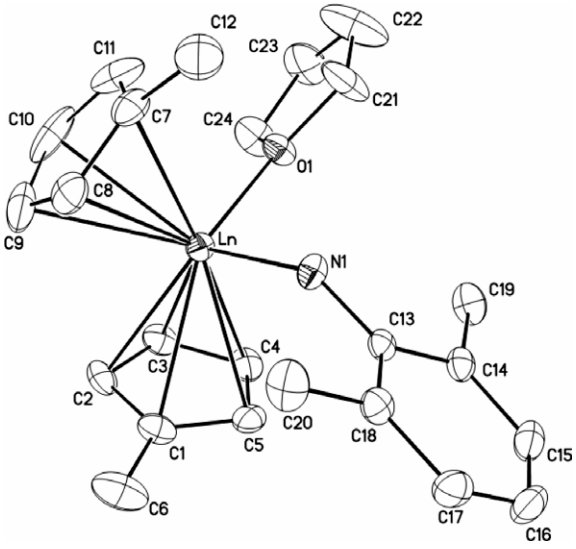


Fig. 1. ORTEP diagram of complexes [Ln = Yb (**I**), Y (**III**)] showing atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level. Hydrogen atoms are omitted for clarity.

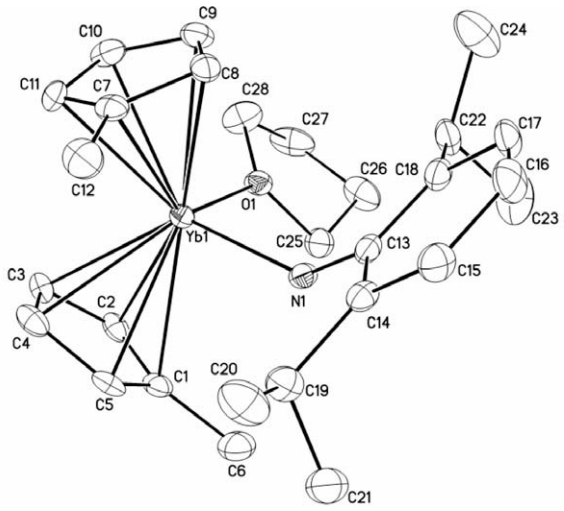


Fig. 2. ORTEP diagram of complex **II** showing atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level. Hydrogen atoms are omitted for clarity.

Crystals of **I–III** suitable for X-ray diffraction study were obtained by cooling a diethyl ether solution. The molecular structures of **I** and **III** and **II** are shown in Figs. 1 and 2, respectively, the selected bond lengths and angles are listed in Table 1. Complexes **I–III** are isostructural and each has a THF-solvated monomeric structure. Two methylcyclopentadienyl ligands, one oxygen atom from a THF molecule and one nitrogen atom of an amide group complete a distorted tetrahedral geometry around the central metal, if each of cyclopentadienyl rings is considered to occupy one position. The two methyl groups on methylcyclopentadienyl groups locate at opposite sides, as the steric demand. The coordination number of central metal for each complex is eight. The solid-state structures of **I–III** are quite similar to those for lanthanocene amides reported previously [10,12].

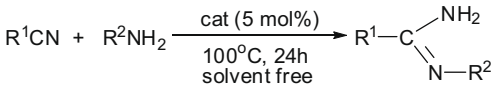
The average bond lengths of Ln–Cent (center of cyclopentadienyl ring) are 2.336 Å for **I**, 2.327 Å for **II** and 2.378 Å for **III**, which are comparable to each other when the difference in ion radii between Yb and Y was considered. The Ln–N bond lengths of 2.206(9) Å for **I**, 2.188(3) Å for **II** and 2.241(4) Å for **III** fall into the range of Yb–amide and Y–amide bond lengths, respectively

Table 1
Selected bond lengths (Å) and angles (°) for **I**, **II** and **III**.

	I	II	III
Ln–Cent(1)	2.322	2.346	2.366
Ln–Cent(2)	2.349	2.307	2.390
Ln–N(1)	2.206(9)	2.188(3)	2.241(4)
Ln–O(1)	2.295(5)	2.299(3)	2.335(2)
Ln–C(1)	2.613(9)	2.675(4)	2.653(4)
Ln–C(2)	2.620(10)	2.635(4)	2.653(5)
Ln–C(3)	2.613(8)	2.603(5)	2.639(4)
Ln–C(4)	2.607(9)	2.607(6)	2.650(4)
Ln–C(5)	2.607(8)	2.634(5)	2.650(4)
Ln–C(7)	2.698(11)	2.595(5)	2.737(5)
Ln–C(8)	2.641(8)	2.599(5)	2.665(5)
Ln–C(9)	2.612(10)	2.610(5)	2.620(6)
Ln–C(10)	2.598(11)	2.599(5)	2.617(6)
Ln–C(11)	2.657(9)	2.583(6)	2.682(5)
Cent(1)–Ln–N(1)	108.900(241)	105.605(105)	108.482(84)
Cent(2)–Ln–N(1)	104.314(271)	110.242(102)	104.129(101)
N(1)–Ln–O(1)	100.0(3)	97.10(11)	101.58(13)
Cent(1)–Ln–Cent(2)	130.252(27)	129.019(12)	129.770(17)

Cent(1) is the centroid of the C(1)–C(5) ring and Cent(2) is the centroid of the C(7)–C(11) ring.

Table 2
Addition of amines to nitriles catalyzed by complexes **I**–**IV**.[a]



1 2 3

Entry	Cat	Nitrile R^1		Amine R^2		Product	Yield (%)[b]
1	**I**	C_6H_5	**1a**	4-FC_6H_4	**2a**	**3aa**	91
2	**I**	C_6H_5	**1a**	2,6-^{i}Pr$_2$C$_6$H$_3$	**2b**	**3ab**	62
3	**II**	C_6H_5	**1a**	4-FC_6H_4	**2a**	**3aa**	96
4	**II**	C_6H_5	**1a**	2,6-iPr$_2$C$_6$H$_3$	**2b**	**3ab**	74
5	**III**	C_6H_5	**1a**	4-FC_6H_4	**2a**	**3aa**	65
6	**III**	C_6H_5	**1a**	2,6-^{i}Pr$_2$C$_6$H$_3$	**2b**	**3ab**	35
7	**IV**	C_6H_5	**1a**	4-FC_6H_4	**2a**	**3aa**	57
8	**IV**	C_6H_5	**1a**	2,6-^{i}Pr$_2$C$_6$H$_3$	**2b**	**3ab**	39

[a] Reaction run using 1 equiv of nitrile and 2 equiv of amine.
[b] Isolated yields.

[25–30]. The Ln–O bond lengths of 2.295(5) Å for **I**, 2.299(3) Å for **II** and 2.335(2) Å for **III** are normal. The bond angles around the central metal for complexes **I**–**III** are well comparable.

To further assess the influence of amide group on the reactivity, a known aliphatic amide complex $(CH_3C_5H_4)_2YbN^iPr_2(THF)$ **IV** was also prepared by the published method [12].

2.2. Catalytic activity of complexes *I*–*IV* for addition of amines to nitriles

The mode reactions of $NH_2(4\text{-}FC_6H_4)$, and $NH_2(2,6\text{-}^iPr_2C_6H_3)$ with C_6H_5CN were examined using complexes **I**–**IV**, respectively. As shown in Table 2, all the complexes can serve as the catalysts for these two reactions to the corresponding monosubstituted *N*-arylamidines under solvent free condition, however, the influence of both central metals and amide groups on the activity was observed. The active sequence follows as Y < Yb for the metals, and $-N^iPr_2 < -NH(2,6\text{-}Me_2C_6H_3) < -NH(2,6\text{-}^iPr_2C_6H_3)$ for the amide groups. Thus, complex **II** is the most active among the four complexes.

Since complex **II** performed well for this transformation, a range of other substrates were surveyed using complex **II** as the catalyst. These results are listed in Table 3. For comparison the data published with the amide ytterbium complex supported by a bridged bisamidinate, $[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3)]Yb(NH[2,6\text{-}^iPr_2C_6H_3])(THF)$ **V**, [19] were also listed. It can be seen that the catalytic behavior for the two systems with complexes **II** and **V**, respectively, is quite similar. For both systems the reactions of aromatic nitriles, except *o*-methoxybenzonitrile (entry 7) with primary aromatic amines proceed smoothly to give the corresponding monosubstituted *N*-arylamidines in good to excellent yields (entries 1, 3, 5 and 9), while the reactions with aliphatic nitriles, and aliphatic amines went sluggishly (entries 4, 10 and 11). However, the difference in activity between the two catalysts was observed: the amide complex with a bridged bis(amidinate) ligand, **V**, is more efficient than the amide complex supported by a cyclopentadienyl ligand, **II**, in addition of amines to nitriles.

3. Conclusion

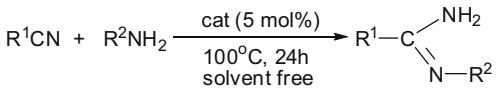
We have synthesized and structurally characterized a series of lanthanide amide complexes supported by methylcyclopentadienyl ligand. These complexes have been found to be efficient catalysts for addition of amines to nitriles to give monosubstituted *N*-arylamidines. The catalytic activity depends on the central metal and amide group. Further research on structure-reactivity relationships of lanthanide amides in homogeneous catalysis is ongoing in our laboratory.

4. Experimental

4.1. General procedures

All manipulations and reactions were performed under an atmosphere of purified argon using standard Schlenk techniques. Toluene, THF, hexane and diethyl ether (analytical grade) were dried and freed from oxygen by refluxing over Na or sodium benzophenone ketyl and distilled prior to use. All other reagents were purchased from Acros and used as received without further purification. Complexes $(CH_3C_5H_4)_2LnCl(THF)$ (Ln = Y and Yb) were prepared according to the literature procedure [10]. Complex $(CH_3C_5H_4)_2YbN^iPr_2(THF)$ was prepared by the published method

Table 3
Addition of amines to nitriles catalyzed by complex **II**: substrate scope.[a]



1 2 3

Entry	Nitrile R^1		Amine R^2		Product	Yield (%)[b]	Yield (%)[c]
1	C_6H_5	**1a**	C_6H_5	**2c**	**3ac**	90	94
2	C_6H_5	**1a**	2,6-$Me_2C_6H_3$	**2d**	**3ad**	76	91
3	C_6H_5	**1a**	4-$CH_3C_6H_4$	**2e**	**3ae**	86	91
4	C_6H_5	**1a**	$CH_3(CH_2)_5$	**2f**	–	Trace	Trace
5	4-$CH_3OC_6H_4$	**1b**	4-FC_6H_4	**2a**	**3ba**	92	98[d]
6	4-$CH_3OC_6H_4$	**1b**	2,6-^{i}Pr$_2$C$_6$H$_3$	**2b**	**3bb**	75	92
7	2-$CH_3OC_6H_4$	**1c**	4-FC_6H_4	**2a**	–	Trace	Trace
8	4-ClC_6H_4	**1d**	2,6-^{i}Pr$_2$C$_6$H$_3$	**2b**	**3db**	58	84
9	3-$CH_3C_6H_4$	**1e**	4-FC_6H_4	**2a**	**3ea**	80	95[d]
10	$PhCH_2$	**1f**	4-FC_6H_4	**2a**	**3fa**	55	62
11	CH_3	**1g**	4-FC_6H_4	**2a**	**3ga**	25	45

[a] Reaction run using 1 equiv of nitrile and 2 equiv of amine.
[b] Isolated yields.
[c] Ref. [19].
[d] 12 h.

[12]. Infrared spectra were recorded on a Magna-IR 550 spectrometer as KBr pellet. ^{1}H and ^{13}C NMR spectra were recorded on a Unity Inova-400. Melting points were measured in a sealed Ar-filled capillary tube, and uncorrected. Lanthanide analyses were carried out by a complexometric titration. Carbon, hydrogen and nitrogen analyses were preformed by direct combustion on a Carlo–Erba EA 1110 instrument.

4.2. Synthesis of complex *I*

A THF solution (5 mL) of NaNH(2,6-$Me_2C_6H_3$) (0.46 g, 3.20 mmol) was added to a stirring toluene solution (20 mL) of $(CH_3C_5H_4)_2$YbCl(THF) (1.39 g, 3.20 mmol). The reaction mixture was stirred for 12 h at room temperature. The volatiles were removed under reduced pressure and the residue was extracted with diethyl ether and the extracts were concentrated to about 10 mL. Cooling the solution at 0 °C afforded the green crystals. Yield: 0.98 g (59%). Mp: 123–125 °C. Anal. Calc. for $C_{24}H_{32}$NOYb: C, 55.01; H, 7.12; N, 2.67; Yb, 32.14. Found: C, 54.74; H, 6.87; N, 2.82; Yb, 31.86%. IR (KBr, cm^{-1}): 3467 (s), 2966 (w), 2922 (w), 2854 (w), 1623 (m), 1476 (s), 1378 (w), 1229 (vs), 1154 (vs), 1090 (w), 759 (m), 639 (m), 555 (m), 502 (s).

4.3. Synthesis of complex *II*

This complex was prepared as green crystals from the reaction of $(CH_3C_5H_4)_2$YbCl(THF) (1.96 g, 4.51 mmol) with NaNH(2,6-^{i}Pr$_2$C$_6$H$_3$) (0.90 g, 4.51 mmol) in THF (20 mL) under being stirred for 12 h at room temperature and recrystallization from a diethyl ether solution by a procedure similar to that used for the synthesis of complex **I**. Yield: 1.21 g (46%). Mp: 160–163 °C. Anal. Calc. for $C_{28}H_{40}$NOYb: C, 57.96; H, 6.90; N, 2.42; Yb, 29.96. Found: C, 57.49; H, 7.12; N, 2.30; Yb, 29.40%. IR (KBr, cm^{-1}): 3482 (s), 3068 (w), 2962 (vs), 2870 (w), 2773 (w), 1620 (s), 1438 (s), 1383 (m), 1213 (s), 1155 (vs), 1085 (w), 745 (s).

4.4. Synthesis of complex *III*

This complex was prepared as colorless crystals from the reaction of $(CH_3C_5H_4)_2$YCl(THF) (1.47 g, 4.20 mmol) with NaNH(2,6-$Me_2C_6H_3$) (0.60 g, 4.20 mmol) in THF (20 mL) under being stirred for 12 h at room temperature and recrystallization from a diethyl ether solution by a procedure similar to that used for the synthesis of complex **I**. Yield: 0.77 g (42%). Mp: 164–167 °C. Anal. Calc. for $C_{24}H_{32}$NOY: C, 65.60; H, 7.28; N, 3.18; Y, 20.25. Found: C, 64.94; H, 7.01; N, 3.05; Y, 19.83%. IR (KBr, cm^{-1}): 3467 (s), 2966 (w), 2922 (w), 2854 (w), 1623 (m), 1476 (s), 1378 (w), 1229 (vs), 1154 (vs), 1090 (w), 759 (m), 639 (m), 555 (m). 502 (s). ^{1}H NMR(C_6D_6, 400 MHz, 25 °C): δ 6.82–7.12 (m, 3H, Ph), 5.97–6.20 (m, 8H, MeC_5H_4), 4.46 (s, 1H, NH), 3.34 (s, 4H, THF), 2.24 (s, 6H, MeC_5H_4), 2.04 (s, 3H, CH_3), 1.84 (s, 3H, CH_3), 1.11 (s, 4H, THF).

4.5. X-ray crystallography of complexes *I*–*III*

Crystals of complexes **I**–**III** suitable for X-ray diffraction study were sealed in a thin-walled glass capillary filled under argon. Diffraction data were collected on a Rigaku Mercury CCD area detector in ω scan mode using Mo Kα radiation (λ = 0.71070 Å). The diffracted intensities were corrected for Lorentz polarization effects and empirical absorption corrections. Details of the intensity data collection and crystal data are given in Table 4.

The structures were solved by direct methods and refined by full-matrix least-squares procedures based on $|F|^2$. All the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were all generated geometrically, assigned appropriate isotropic thermal parameters, and allowed to ride on their parent carbon atoms. All the H atoms were held stationary and included in the structure factor calculation in the final stage of full-matrix least-squares refinement. The structures were solved and refined using SHELXL-97 program.

Table 4
Details of the crystallographic data and refinements for complexes **I**, **II** and **III**.

	I	II	III
Empirical formula	$C_{24}H_{32}$NOYb	$C_{28}H_{40}$NOYb	$C_{24}H_{32}$NOY
Formula weight	523.55	579.65	439.42
T (K)	223(2)	223(2)	293(2)
Wavelength (Å)	0.71075	0.71075	0.71070
Crystal system	Monoclinic	Orthorhombic	Monoclinic
Space group	$P2_1$	$P2_12_12_1$	$P2_1$
a (Å)	8.272(6)	20.652(3)	8.321(3)
b (Å)	14.583(9)	12.177(2)	14.686(4)
c (Å)	9.196(6)	10.4921(18)	9.308(3)
α (°)	90.00	90.00	90.00
β (°)	97.645(15)	90.00	98.262(6)
γ (°)	90.00	90.00	90.00
V (Å^3)	1099.3(13)	2638.5(8)	1125.6(6)
Z	2	4	2
D_{calc} (g/cm^{-3})	1.582	1.459	1.296
μ (mm^{-1})	4.266	3.562	2.603
F(0 0 0)	522	1172	460
Crystal size (mm)	0.22 × 0.16 × 0.08	0.70 × 0.30 × 0.25	0.80 × 0.52 × 0.50
θ Range	3.11–25.50	3.23–27.48	3.07–25.35
Reflections collected	5394	9909	11 003
Independent reflections	3462	5853	4077
Parameters refined	211	292	253
Good-of-fit (GOF)	0.973	0.980	0.933
R	0.0331	0.0323	0.0337
wR	0.0605	0.0504	0.0571

4.6. General procedure of addition of amines to nitriles

Amine (2 mmol) was added to a mixture of nitrile (1 mmol) and catalyst (5 mol%). The reaction mixture was stirred at 100 °C for 24 h. The product was isolated by distilling the reaction mixture under vacuum to remove unreacted starting materials. The residue was recrystallized from toluene/hexane to afford the desired products. All products were identified by comparison with those of authentic samples.

Acknowledgements

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Appendix A. Supplementary material

CCDC 721341, 721342 and 721343 contain the supplementary crystallographic data for complexes **I**, **II** and **III**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.05.011.

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