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Ruthenium(III) complexes of dipicolinic acid with $PPh_3/AsPh_3$ as co-ligand: Synthesis and structural characterization

Duraiswamy Sukanya, Rathinasabapathi Prabhakaran, Karuppannan Natarajan *

Department of Chemistry, Bharathiar University, Maruthamalai Road, Coimbatore 641 046, Tamilnadu, India

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Abstract

New hexa-coordinated Ru(III) complexes of the type $[Ru(dipic)(EPh_3)_2X]$ have been synthesized by reacting 2,6-pyridine dicarboxylic acid (dipicolinic acid, H_2dipic) with the appropriate starting complexes $[RuX_3(EPh_3)_3]$ (where X = Cl, Br; E = P, As). The ligand behaves as tridentate dibasic chelate. Dipicolinic acid which was expected to form a bridge between two metal centers, formed only mononuclear complexes, irrespective of the metal to ligand ratio. All the complexes have been characterized by analytical and spectroscopic (IR, electronic and EPR) data. Single-crystal X-ray analysis of the complex $[Ru(dipic)(PPh_3)_2Cl]$ revealed that the coordination environment around ruthenium metal consists of an NO_2P_2Cl octahedron with (dipic) occupying equatorial plane. Electrochemical behavior of the complexes was studied using cyclic voltammetry. The new complexes were found to catalyze the oxidation of alcohols to aldehydes using *N*-methyl morpholine-*N*-oxide as co-oxidant.

Keywords: Ruthenium(III) complexes; Dipicolinic acid; Crystal structure; EPR; Electrochemistry; Catalytic activity

1. Introduction

Ruthenium chemistry of triphenylphosphine and triphenylarsine has been enriched with various ligand types [1–4] and their role as potential catalysts for many organic syntheses is well documented [5,6]. Work on dicarboxylic acids as ligands for ruthenium is meagre [7–10]. Among the dicarboxylic acids, dipicolinic acid is known for its various ligating modes [11–15] and application in analytical chemistry [16,17], corrosion inhibition [18], decontamination of nuclear reactors [19] and diverse biological activity [20–22]. Moreover, dipicolinate complexes have been used as electron carriers in some model biological systems [23], as specific molecular tools in DNA cleavage [24] and as NO scavengers [9]. The paucity of ruthenium complexes containing both triphenylphosphine/arsine and dicarboxylic acid for use as catalyst precursor in organic synthesis has initiated the synthesis of such compounds. Herein, we report the synthesis, characterization (XRD), spectroscopic properties and redox behavior of Ru(III) compounds containing $PPh_3/AsPh_3$ and dipicolinic acid. The new complexes were tested for their catalytic activity in the oxidation of alcohols.

2. Experimental

2.1. Materials

$RuCl_3 \cdot 3H_2O$ and 2,6-pyridine dicarboxylic acid were purchased from Himedia and used without further purification. Solvents were distilled following the literature procedures [25]. The starting complexes $[RuCl_3(PPh_3)_3]$ [26], $[RuCl_3(AsPh_3)_3]$ [27], $[RuBr_3(AsPh_3)_3]$ [28], $[RuBr_3(PPh_3)_2(MeOH)]$ [29] were prepared as reported earlier.

2.2. Physical measurements

Infrared spectra of the ligand and the complexes have been recorded on a Nicolet Avatar model FT-IR

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* Corresponding author. Tel.: +91 422 2424655; fax: +91 422 2422387.
E-mail address: k_natraj6@yahoo.com (K. Natarajan).

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spectrophotometer using KBr discs in the range of 4000–400 cm^{-1}. Electronic spectra were recorded on a Systronics 119 UV–Vis spectrophotometer using methanol as solvent. Elemental analyses were performed with a Vario EL III Elementar analyzer. Room temperature EPR spectra were recorded using an E-112 Varian model instrument. Cyclic voltammetric experiments were carried out using BAS CV-27 electrochemical analyzer with a glassy carbon working electrode. A platinum wire and silver–silverchloride electrode were used as counter- and reference electrodes, respectively. Melting points were recorded with a Lab India apparatus.

2.3. Crystallography

Single crystals of $[Ru(dipic)(PPh_3)_2Cl]$ suitable for X-ray diffraction studies were obtained from slow evaporation of a solution of the complex in benzene/ethanol mixture. Intensity data were collected on a Nonius MACH 3 four circle diffractometer equipped with graphite monochromated Mo Kα radiation ($\lambda = 0.7107$ Å). The structure was solved by the direct method. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares, with a ridging model for the hydrogen atoms, using the SHELXTL/PC package [30].

2.4. Synthesis of $[Ru(dipic)(EPh_3)_2X]$ (X = Cl, Br; E = P, As)

All the new ruthenium(III) complexes were prepared by the following general procedure. A benzene (20 ml) solution of $[RuX_3(EPh_3)_3]$ (where E = P, As; X = Cl, Br) (0.1 mmol) was added to a refluxing solution of dipicolinic acid (0.1 mmol) in ethanol (20 ml). The mixture was heated under reflux for 6 h. The solution was filtered while hot, reduced to half of its volume and left for slow evaporation. The crystalline product that separated out was filtered off, washed with ethanol and dried under vacuum. The product was recrystallized from benzene/ethanol mixture.

$[Ru(dipic)(PPh_3)_2Cl]$ (**1**) was prepared from $[RuCl_3(PPh_3)_3]$ (0.0994 g, 0.1 mmol) and dipicolinic acid (0.018 g, 0.1 mmol) as reddish brown crystals. Yield: 75%. *Anal.* Calc. for $RuC_{43}H_{33}NO_4ClP_2$: C, 62.51; H, 4.02; N, 1.69. Found: C, 62.04; H, 4.51; N, 1.74%. IR: 1659, 1352, 1431, 1091, 697 cm^{-1}. UV: λ_{max}: 268(4236), 405(9219) nm(dm^3/mol/l); m.p. >230 °C.

$[Ru(dipic)(AsPh_3)_2Cl]$ (**2**) was prepared from $[RuCl_3(AsPh_3)_3]$ (0.112 g, 0.1 mmol) and dipicolinic acid (0.018 g, 0.1 mmol) as shining red crystals. Yield: 80%. *Anal.* Calc. for $RuC_{43}H_{33}NO_4ClAs_2$: C, 56.44; H, 3.63; N, 1.53. Found: C, 56.17; H, 3.82; N, 1.60%. IR: 1671, 1344, 1434, 1077, 692 cm^{-1}. UV: λ_{max}: 268(4767), 315(6296), 530(964) nm(dm^3/mol/l); m.p. >290 °C.

$[Ru(dipic)(AsPh_3)_2Br]$ (**3**) was prepared from $[RuBr_3(AsPh_3)_3]$ (0.125 g, 0.1 mmol) and dipicolinic acid (0.018 g, 0.1 mmol) as brown crystals. Yield: 85%. *Anal.* Calc. for $RuC_{43}H_{33}NO_4BrAs_2$: C, 59.32; H, 3.82; N, 1.60. Found: C, 59.27; H, 3.94; N, 1.58%. IR: 1673, 1350, 1434, 1077, 692 cm^{-1}. UV: λ_{max}: 268(4706), 315(5652), 416(10184), 534(590) nm(dm^3/mol/l); m.p. >270 °C.

$[Ru(dipic)(PPh_3)_2Br]$ (**4**) was prepared from $[RuBr_3(PPh_3)_2(MeOH)]$ (0.0897 g, 0.1 mmol) and dipicolinic acid (0.018 g, 0.1 mmol) as orange brown crystals. Yield: 75%. *Anal.* Calc. for $RuC_{43}H_{33}NO_4BrP_2$: C, 53.82; H, 3.46; N, 1.45. Found: C, 53.79; H, 3.63; N, 1.39%. IR: 1666, 1347, 1440, 1080, 690 cm^{-1}. UV: λ_{max}: 268(4344), 416(9180) nm(dm^3/mol/l); m.p. >240 °C.

2.5. Catalytic oxidation

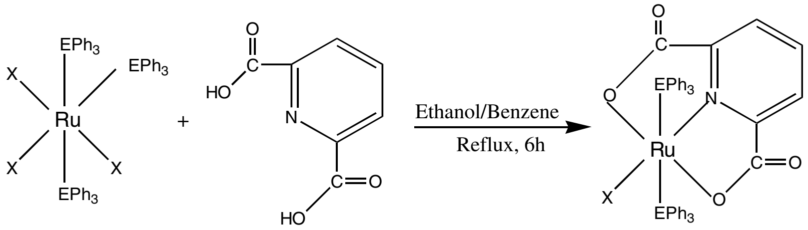
To a solution of the alcohol (1 mmol) in CH_2Cl_2 (20 ml), *N*-methyl morpholine-*N*-oxide (3 mmol) and ruthenium complex (0.01 mmol) were added. The solution was heated under reflux for 3 h. The mixture was evaporated to dryness and extracted with petroleum ether (60–80 °C). The combined petroleum ether mixture was filtered and evaporated to give the corresponding aldehyde, which was then quantified as its 2,4-dinitrophenyl hydrazone derivative [31].

3. Results and discussion

New hexa-coordinated Ru(III) complexes of the type $[Ru(dipic)(EPh_3)_2X]$ have been prepared by reacting 2,6-pyridine dicarboxylic acid (dipicolinic acid, H_2dipic) with $[RuCl_3(PPh_3)_3]$, $[RuCl_3(AsPh_3)_3]$, $[RuBr_3(AsPh_3)_3]$ and $[RuBr_3(PPh_3)_2(MeOH)]$ in a 1:1 mole ratio. The ligand replaces one EPh_3 and two X atoms, (E = As or P; X = Cl or Br), from the starting complex to yield $[Ru(dipic)(EPh_3)_2X]$ (Scheme 1). Dipicolinic acid acts as a dibasic tridentate ligand in all the complexes. The analytical data conform to the stoichiometry of the ruthenium(III) complexes as $[Ru(dipic)(EPh_3)_2X]$.

3.1. Infrared spectroscopy

The IR spectrum of the free ligand (H_2dipic) shows a strong band around 1700 cm^{-1} assignable to $\nu(C{=}O)$ of COOH moiety [7]. In all the complexes, the band due to $\nu_{as}(COO^-)$ was observed in the region 1675–1610 cm^{-1} and that due to $\nu_s(COO^-)$ in the region 1352–1344 cm^{-1}. A large difference of 327–307 cm^{-1} between ν_{as} and ν_s vibrations indicates a monodentate coordination of the carboxylic group in all the complexes [32]. A broad band observed in the region 3340–2500 cm^{-1} region due to $\nu(O–H)$ of the carboxyl group in the free ligand disappeared in all the complexes indicating the deprotonation and subsequent coordination through oxygen donor atom. The absence of any peak around 1700 cm^{-1} reveals that both COOH groups are involved in coordination. All the characteristic peaks due to PPh_3/$AsPh_3$ were observed in the usual regions [31].



E = As, P
X = Cl,Br

Scheme 1.

3.2. EPR spectroscopy

The EPR spectra of the powdered samples were recorded at room temperature at X-band frequencies. The nature of the spectra revealed the absence of any hyperfine splitting due to interaction with any other nuclei present in the complexes. Complexes **1** and **4** showed two lines with two different 'g' values ($g_x = g_y \neq g_z$) indicating magnetic anisotropy and suggestive of tetragonal distortion in octahedral geometry [33]. However, complexes **2** and **3** showed three lines with three different 'g' values ($g_x \neq g_y \neq g_z$) indicating rhombic distortion [34]. The 'g' values are in the range 2.12–2.21. The nature of spectra obtained is in good agreement with that of the previously reported Ru(III) complexes [35]. A representative case is depicted in Fig. 1.

3.3. Electrochemistry

The redox behavior of the complexes was studied using cyclic voltammetry at a glassy carbon working electrode at a scan rate of 100 mV s^{-1}. [NBu$_4$]BF$_4$ (0.1 M) was used as supporting electrolyte in acetonitrile solution of 0.001 M of complex. The cyclic voltammetric data are given in Table 1 and a representative cyclic voltammogram is displayed in Fig. 2. The $E_{1/2}$ of the oxidation process was in the range from 0.71 to 0.79 V and reduction process was in the range from −0.73 to −0.80 V. The oxidation and reduction waves

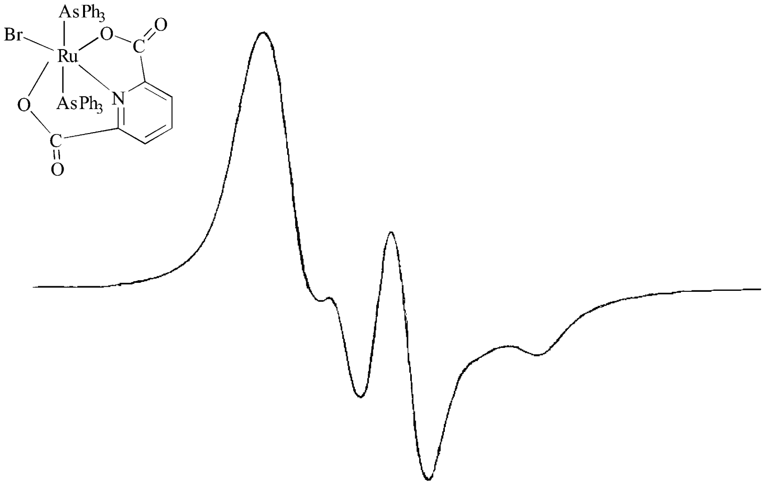


Fig. 1. EPR spectrum of **3**.

Table 1
Cyclic voltammetric data[a] of new Ru(III) complexes

Complex	Ru(III)–Ru(IV)				Ru(III)–Ru(II)			
	E_{pc} (V)	E_{pa} (V)	E_f (V)	ΔE_p (mV)	E_{pc} (V)	E_{pa} (V)	E_f (V)	ΔE_p (mV)
1	0.60	0.84	0.72	236	−0.87	−0.72	−0.79	152
2	0.65	0.77	0.71	120	−0.84	−0.62	−0.73	224
3	0.72	0.84	0.78	127	−0.87	−0.73	−0.80	141
4	0.69	0.88	0.79	192	−0.84	−0.71	−0.78	137

[a] Supporting electrolyte: [NBu$_4$]ClO$_4$ (0.05 M); complex concentration: 0.01 M; scan rate: 100 mV s^{-1}; all the potentials are referenced to Ag/AgCl.

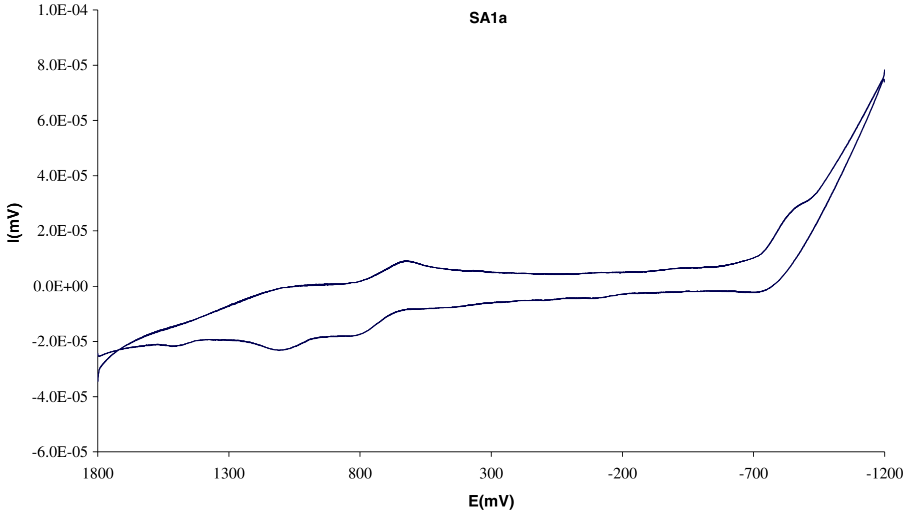


Fig. 2. Cyclic voltammogram of **1**.

are due to the metal centered Ru(III) → Ru(IV) and Ru(III) → Ru(II) processes, respectively. Any redox behavior of the ligand in the range 0.71 to −0.80 V is ruled out because dipicolinic acid showed quasi-reversible and irreversible processes only around −1.58 V in its complexes [36]. Moreover, potential difference between the two successive oxidation processes is ≈1.5 V which agrees well with the average potential difference between the redox processes of the ruthenium center ($Ru^{II/III}$–$Ru^{III/IV}$) (≈1.0–1.5 V) observed for other mononuclear complexes [37]. All the complexes exhibit quasi-reversible oxidative couples with peak to peak separations (ΔE_p) of 120–236 mV [38]. Replacement of PPh_3 by $AsPh_3$ showed no much variation in the redox potential. It has been observed from the electrochemical data that the present ligand system is ideally suitable for stabilizing the higher oxidation state of the ruthenium ion [39–41].

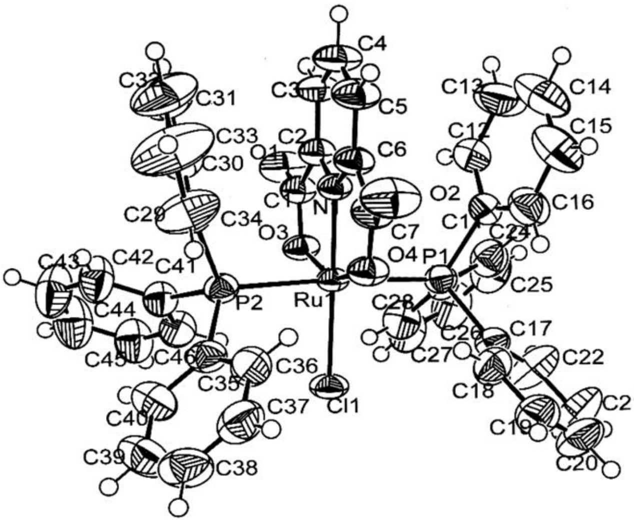


Fig. 3. ORTEP diagram [Ru(dipic)(PPh_3)$_2$Cl] (**1**).

3.4. Crystal structure of [Ru(dipic)(PPh_3)$_2$Cl]

The ORTEP diagram of [Ru(dipic)(PPh_3)$_2$Cl] is shown in Fig. 3. The crystal data and refinement parameters are summarized in Table 2. Selected bond lengths and bond

Table 2
Crystal data and structure refinement parameters for [Ru(dipic)(PPh_3)$_2$Cl]

Empirical formula	$C_{43}H_{33}ClNO_4P_2Ru$
Formula weight	826.16
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system, space group	monoclinic, *C*2/*c*
Unit cell dimensions	
a (Å)	34.882(14)
b (Å)	9.998(4)
c (Å)	29.132(12)
α (°)	90
β (°)	121.32(3)
γ (°)	90
Volume (Å^3)	8679(6)
Z, calculated density (Mg/m^3)	8, 1.265
Absorption coefficient (mm^{-1})	0.535
F(000)	3368
Crystal size (mm)	0.22 × 0.19 × 0.14
θ Range for data collection (°)	2.15–24.98
Limiting indices	$0 \leqslant h \leqslant 41$, $-1 \leqslant k \leqslant 11$, $-34 \leqslant l \leqslant 29$
Reflections collected/unique (R_{int})	8653/7584 (0.0171)
Completeness to θ = 24.98 (%)	99.4
Absorption correction	ψ-scan
Maximum and minimum transmission	0.9998 and 0.9647
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	7584/0/469
Goodness-of-fit on F^2	1.057
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0327$, $wR_2 = 0.0913$
R indices (all data)	$R_1 = 0.0517$, $wR_2 = 0.1055$
Largest difference in peak and hole (e A^{-3})	0.530 and −0.603

angles are given in Table 3. The N(1)–Ru(1)–Cl(1) bond angle is 178.95(9)° showing that Cl atom lies *trans* to ring nitrogen. The bite angles around Ru(III) are N(1)–Ru(1)–O(4) = 77.42(10)°; N(1)–Ru(1)–O(3) = 77.28(9)°; O(4)–Ru(1)–Cl(1) = 103.60(7)° and O(3)–Ru(1)–Cl(1) = 101.70(6)°, summing up the in-plane angle to be exactly 360°. This shows the high planarity of the Cl and O, N, O donor atoms of dipicolinic acid. This is further supported by the other *cis* angles mentioned in Table 3. Thus, the acid occupies the equatorial plane around the Ru(III) octahedron, along with Cl. The bond angle P(1)–Ru(1)–P(2) = 175.62(3)° shows that the two PPh_3 groups are trans to each other occupying the axial positions. The two Ru–P bonds are slightly bent away from the dipicolinic acid towards the Cl atom which is evident from the P(1)–Ru(1)–Cl(1) bond angle is 88.84° which is smaller than P(1)–Ru(1)–N(1) = 91.41° and P(2)–Ru(1)–N(1) = 92.51° [42]. The Ru–P, Ru–O, Ru–N and Ru–Cl bond lengths found in the complex agree well with that reported for similar ruthenium complexes [3,7,43,44].

3.5. Catalytic activity

The oxidation of benzyl alcohol and cinnamyl alcohol to benzaldehyde and cinnamaldehyde was carried out with the new ruthenium(III) complexes as catalysts in the presence of *N*-methyl morpholine-*N*-oxide as co-oxidant in CH_2Cl_2. After 3 h of reflux the aldehydes formed were quantified as their 2,4-dinitrophenyl hydrazone derivatives [25]. The results obtained are given in Table 4. In cinnamyl alcohol, only the alcoholic group gets oxidized selectively without affecting the double bond. The relatively higher yield of cinnamaldehyde when compared to benzaldehyde is due to the fact that α-CH unit of cinnamyl alcohol is more acidic than benzyl alcohol [45]. The replacement of PPh_3 by $AsPh_3$ group in the complexes did not result in much. On comparing the catalytic activity of these complexes with that of the other reported ruthenium(III) complexes, it is found that theses complexes exhibit comparatively lower activity [31,46]. This may be due to the stronger chelation of the ligand which may hinder the formation of catalytically active species.

Table 3
Selected bond lengths (Å) and bond angles (°) for $[Ru(dipic)(PPh_3)_2Cl]$

Bond lengths	
Ru(1)–Cl(1)	2.3340(12)
Ru(1)–O(4)	2.050(2)
Ru(1)–N(1)	1.996(3)
Ru(1)–P(1)	2.3945(12)
Ru(1)–O(3)	2.053(2)
Ru(1)–P(2)	2.4052(12)
Bond angles	
N(1)–Ru(1)–O(4)	77.42(10)
O(3)–Ru(1)–P(1)	90.81(8)
N(1)–Ru(1)–O(3)	77.28(9)
Cl(1)–Ru(1)–P(1)	88.84(3)
O(4)–Ru(1)–O(3)	154.70(8)
N(1)–Ru(1)–P(2)	92.57(8)
N(1)–Ru(1)–Cl(1)	178.95(9)
O(4)–Ru(1)–P(2)	89.36(8)
O(4)–Ru(1)–Cl(1)	103.60(7)
O(3)–Ru(1)–P(2)	91.85(8)
O(3)–Ru(1)–Cl(1)	101.70(6)
Cl(1)–Ru(1)–P(2)	87.22(3)
N(1)–Ru(1)–P(1)	91.41(8)
P(1)–Ru(1)–P(2)	175.62(3)
O(4)–Ru(1)–P(1)	89.72(8)

Table 4
Catalytic activity of new Ru(III) complexes

Complex	Substrate	Yield[a] (%)	Turnover number[b]
1	benzyl alcohol	56	57
	cinnamyl alcohol	60	59
2	benzyl alcohol	36	37
	cinnamyl alcohol	60	59
3	benzyl alcohol	30	30
	cinnamyl alcohol	67	66
4	benzyl alcohol	39	40
	cinnamyl alcohol	62	61

[a] Yield based on substrate.

[b] Moles of product per mole of catalyst.

4. Conclusion

Dipicolinic acid can coordinate to the metal in a number of fashions. In spite of this variable ligating ability, it acts as tridentate dibasic chelating ligand forming only mononuclear complex irrespective of the metal-to-ligand ratio.

Acknowledgement

UGC-CSIR, New Delhi, India, is acknowledged for their financial assistance.

Appendix A. Supplementary data

Crystallographic data for $[Ru(dipic)(PPh_3)_2Cl]$ (**1**) have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 288561. 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 http://www.ccdc.cam.ac.uk). Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2006.01.023.

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