Synthesis, characterisation of few N-substituted 1,8-naphthalimide derivatives and their copper(II) complexes

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Abstract. A few 1,8-naphthalimide derivatives with phenyl (1), benzyl (2), 3,4-dimethoxyphenyl ethyl (3), 4-pyridyl (4), 2-hydroxy ethyl (5), 4-pyridylmethyl (6) groups attached to the nitrogen atom are synthesized and characterized. Cyclic voltammograms of all these compounds show one-electron reversible redox cycle (-1.24 V to -1.18 V) due to formation of anion radicals. However, in the case of (5), quenching of this redox process occurs when polyhydroxy-aromatic compounds such as 1,3-dihydroxy benzene and 1,3,5-trihydroxybenzene are added. Copper complexes, namely *bis*-{N-(4-pyridylmethyl)1,8-naphthalimide}copper (II) perchlorate (8), *bis*-{N-(4-pyridylmethyl)1,8-naphthalimide} copper (II) perchlorate (8) and (9) show reversible redox couple of the ligand without any significant interaction with the redox active copper (II) centre.

Keywords. N-substituted 1,8-naphthalimides; Cu(II) complexes of N-substituted 1,8-naphthalimides; redox active Cu(II) centre.

1. Introduction

Imides of aromatic dicarboxylic acids are important in the construction of macromolecules^{1,2} as well as supramolecular assembly.³⁻⁷ Ease of formation of imides from the corresponding anhydrides has made them simple to synthesise.^{8,9} They are useful fluoro-probes for various studies^{8,10-14} and also serve as precursors for protection of the amine group.¹⁵⁻¹⁹ Photochemistry of phthalimide derivatives results in selective organic transformations.^{20,21} Phthalimide analogues such as naphthalimide derivatives are electroactive, and there is scope for their study in supramolecular environments.^{3–7} 1,8-Naphthalimides also show one-electron reversible redox property.^{23–25} We have demonstrated that N-2-hydroxyethyl phthalimide forms an adduct with 1,3-dihydroxy benzene and 1,3,5-trihydroxybenzene, and has an interesting supramolecular structure.²⁶ Thus, understanding of the redox behaviour of 1,8-naphthalimides having different groups anchored to the nitrogen atom is expected to throw light on their implications on supramolecular binding and this also would provide information on their suitability as electrochemical probes. This paper deals with the synthetic and spectroscopic properties of a few N-substituted 1,8naphthalimides and their co-ordination chemistry with copper (II).

2. Experimental

2.1 Synthesis of N-(4-pyridylmethyl)1,8naphthalimide (**6**)

To a solution of 1,8-naphthalic anhydride (0.99 g, 5 mmol) in tetrahydrofuran (THF) (25 ml) 4-pyridylmethyl amine (0.54 g, 5 mmol) was added drop wise at room temperature. The solution was stirred for six hours at room temperature to obtain an offwhite coloured precipitate. The precipitate was collected and washed with THF (10 ml) and dried in air. It was finally recrystallised from a mixture of ethanol and chloroform (2 : 8 mixture). Yield: 1.38 g (97%). Elemental analysis Calcd. for $C_{18}H_{10}N_2O_2$, C, 75.52, H, 3.49, N, 9.79%: found C, 75.46, H, 3.42, N, 9.65%. Spectroscopic details are given in table 1.

2.2 Synthesis of N-(4-pyridylmethyl)phthalimide (7)

Finely ground phthalic anhydride (1.48 g, 10 mmol) was dissolved in THF. To this solution, 4-pyridyl-methyl amine (1.08 g, 10 mmol) was added drop-

Dedicated to the memory of the late Professor Bhaskar G Maiya *For correspondence

Structure	NMR signals (ppm, CDCl ₃)	Characteristic IR-frequencies (KBr, cm^{-1})	$\begin{array}{c} E_{1/2}; \left(\ddot{\mathrm{A}} E \\ i p_c / i p_a \right)^{\#} \end{array}$	
	¹ H: 8.63 (d , J = 8 Hz, 2H), 8.27 (dm , J = 8 Hz, 2H), 7.78 (t , J = 8 Hz, 2H), 7.57–7.47 (m , 3H), 7.31 (dm , J = 6 Hz, 2H)	3073(<i>w</i>), 1701(<i>s</i>), 1655(<i>s</i>), 1583(<i>s</i>), 1501(<i>s</i>), 1440(<i>s</i>), 1352(<i>s</i>), 1335(<i>s</i>), 1183(<i>s</i>), 1112(<i>s</i>), 1076(<i>m</i>), 1025(<i>m</i>), 784(<i>s</i>)	-1260 mV (80 mV, 1·18)	
	¹³ C: 163·73, 151·22, 143·52, 134·96, 132·09, 128·71, 128·0, 127·35, 124·30, 122·53	1025(m), 784(s)		
	¹ H: 8.62 (d , J = 6 Hz, 2H), 8.21 (m , 2H), 7.74 (m , 2H), 7.55 (t , J = 6 Hz, 2H), 7.32–7.23 (m , J = 6 Hz, 3H), 5.39 (s , 2H)	3061(<i>w</i>), 2965(<i>w</i>), 1687(<i>s</i>), 1652(<i>s</i>), 1586(<i>s</i>), 1495(<i>w</i>), 1440(<i>s</i>), 1374(<i>s</i>), 1228(<i>s</i>), 1177(<i>m</i>), 1137(<i>m</i>), 1066(<i>s</i>), 945(<i>s</i>), 778(<i>s</i>)	-1201 mV (80 mV, 0·948)	
	¹³ C: 164·28, 137·51, 134·10, 131·66, 131·48, 129·23, 128·0, 128·61, 127·68, 127·06, 122·75, 43·93			
	¹ H: 8·6 (d , J = 6 Hz, 2H), 8·2 (d , J = 6 Hz, 2H), 7·7 (t , J = 6 Hz, 2H), 6·9 (m , 3H), 4·4 (m , 2H), 3·8 (s , 6H) 3·0 (m , 2H)	3083(<i>w</i>), 2991(<i>m</i>), 2945(<i>m</i>), 2843(<i>m</i>), 1669(<i>s</i>), 1665(<i>s</i>), 1598(<i>s</i>), 1521(<i>s</i>), 1450(<i>s</i>), 1393(<i>s</i>), 1347(<i>s</i>), 1271(<i>s</i>), 1235(<i>s</i>), 1153(<i>s</i>), 1030(<i>s</i>), 948(<i>m</i>), 851(<i>m</i>), 784(<i>s</i>)	-1195 mV (80 mV, 1·2)	
J	¹³ C: 163.61, 148.44, 148.17, 133.61, 131.19, 130.98, 130.82, 127.72, 126.6, 120.64, 111.81, 110.94, 55.72, 55.67, 41.83, 33.76			
	¹ H: 8·83 (d , J = 6·4 Hz, 2H), 8·64 (d , J = 6·4 Hz, 2H), 8·32 (d , J = 6·4 Hz, 2H), 7·81 (t , J = 6·4 Hz, 2H), 7·32 (dm , J = 6 Hz, 2H)	3068(<i>w</i>), 1711(<i>s</i>), 1670(<i>s</i>), 1588(<i>s</i>), 1352(<i>s</i>), 1240(<i>s</i>), 1189(<i>s</i>), 861(<i>s</i>), 789(<i>s</i>)	-1180 mV (80 mV, 1·25)	
	¹³ C: 163·64, 150·28, 148·49, 136·59, 133·89, 132·61, 131·18, 127·82, 126·63, 123·05, 121·97			
О ОН	¹ H: 8·6 (d , J = 6 Hz, 2H) 8·22 (d , J = 6 Hz), 7·8 (t , J = 6 Hz), 4·5 (m , 2H), 4·0 (m , 2H), 2·4 (broad s , 1H)	3483(<i>s</i>), 3073(<i>w</i>), 2966(<i>w</i>), 2879(<i>w</i>), 1706(<i>s</i>), 1660(<i>s</i>), 1593(<i>s</i>), 1445(<i>m</i>), 1388(<i>s</i>), 1327(<i>s</i>), 1235(<i>s</i>), 1081(<i>w</i>), 1025(<i>s</i>), 007(<i>w</i>), 770(<i>s</i>)	5(<i>m</i>), (85 mV,	
0 5	¹³ C: 165, 134, 131, 128, 126, 122, 61, 42	1035(<i>s</i>), 907(<i>w</i>), 779(<i>s</i>)		
	¹ H: 8·6 (d , J = 7·2 Hz, 2H), 8·5 (d , J = 6 Hz, 2H), 8·2 (d , J = 8 Hz, 2H), 7·7 (t , J = 7·2 Hz, 2H), 7·3 (d , J = 6 Hz, 2H), 5·3 (s , 2H)	3083(<i>w</i>), 3022(<i>w</i>), 2966(<i>w</i>), 17778(<i>m</i>), 1724(<i>vs</i>), 1706(<i>s</i>), 1655(<i>s</i>), 1598(<i>s</i>), 1516(<i>s</i>), 1434(<i>s</i>), 1383(<i>s</i>), 1312(<i>s</i>),	-1240 (70 mV, 1·01)	
L ₁ 6	¹³ C: 163·59, 149·53, 145·59, 134·03, 131·26, 127·75, 126·67, 122·94, 121·82, 42·78	1434(s), 1383(s), 1312(s), 1240(s), 1178(s), 948(s), 779(s)		
	¹ H: $8.5 (d, J = 5.6 \text{ Hz}, 2\text{H}), 7.8 (dd, J = 8.4, 4 \text{ Hz}, 4\text{H}), 7.29 (d, J = 6.4 \text{ Hz}, 2\text{H}), 4.9 (s, 2\text{H})$	3099(<i>w</i>), 3037(<i>w</i>), 1718(<i>w</i>), 1706(<i>vs</i>), 1609(<i>w</i>), 1419(<i>s</i>), 1393(<i>s</i>), 1317(<i>w</i>), 1112(<i>m</i>), 943(<i>s</i>), 794(<i>m</i>), 728(<i>s</i>)	_	
C C R L ₂ 7	¹³ C: 167·85, 150·31, 144·97, 134·47, 132·02, 123·77, 123·12, 40·85	120(3)		

 Table 1.
 Spectroscopic properties of naphthalimide derivatives.

[#]All electrode potentials reported are measured in acetonitrlie (1 mmol), TBAP as supporting electrolyte, platinum electrodes as working electrodes and Ag/AgCl as reference electrode (+ve scan, 100 mV/s) and are with respect to the ferrocene couple

wise. The reaction mixture became warm and an off-white precipitate was obtained. The reaction mixture was stirred for three hours for completion of the reaction. The precipitate thus formed was filtered and dried. The precipitate was recrystallised from an ethanol–chloroform mixture (1 : 1) to give N-(4-pyridylmethyl)phthalimide. Yield: 2.3 g (97%). Elemental analysis: Calcd. for C₁₄H₁₀N₂O₂, C, 70·59, H, 4·20, N, 11·76%; found C, 70·52, H, 4·19, N, 11·58%.

Compounds 1-5 were prepared by a procedure similar to that used for 6 and 7.

2.3 Synthesis of bis-{N-(4-pyridylmethyl)1,8naphthalimide}copper (II) perchlorate (8)

Copper (II) perchlorate hexahydrate (0·19 g, 0·5 mmol) was dissolved in dry acetonitrile (5 ml) and a solution of N-(4-pyridylmethyl)1,8-naphthalimide (0·12 g, 1 mmol) in dry acetonitrile (2 ml) was added dropwise with stirring at room temperature. The dark blue solution was stirred for three hours to obtain a light-blue precipitate. The precipitate was washed with diethylether and dried in air (0·18 g, 71%). UV–Vis 603 nm (759 mol⁻¹ cm²). Molar conductance: (CH₃CN, 25°C) 30·84 ohm⁻¹ mol⁻¹ cm².

2.4 Synthesis of bis-(N-(4-pyridylmethyl) 1,8naphthalimide) copper(II)nitrate dihydrate (9)

To a solution of copper (II) nitrate trihydrate (0.24 g, 1 mmol) in methanol (10 ml), a solution of N-(4-pyridylmethyl) 1,8-naphthalimide (0.68 g, 2 mmol) in acetonitrile (5 ml) was added dropwise. The reaction mixture was stirred at room temperature for two hours to obtain a light-blue coloured precipitate. The precipitate was filtered and redissolved in CH₃CN (10 ml) by warming. On cooling, a microcrystalline precipitate was obtained as the desired complex (yield 0.32 g, 37%). Spectroscopic data of the complex are given in table 2. Molar conductance (CH₃CN, 25°C): 42.2 ohm⁻¹ mol⁻¹ cm².

The complex (10) was prepared in a similar manner to (8) to obtain 58% yield. Molar conductance of (10) in acetonitrile (25°C): $34 \cdot 2 \text{ ohm}^{-1} \text{ mol}^{-1} \text{ cm}^2$.

3. Results and discussion

Spectroscopic properties of a series of N-substituted 1,8-naphthalamides synthesized are listed in table 1.

Cyclic voltammograms of all these compounds in acetonitrile with tetrabutyl ammonium perchlorate (TBAP) as supporting electrolyte show reversible redox couple, below. The redox couple is attributed to the formation of a radical anion involving a one-electron redox process as below,^{23–25}

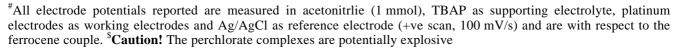
$$\bigvee_{O}^{O} \bigvee_{-e}^{+e} \bigvee_{O}^{O} \bigvee_{-e}^{O}$$
(1)

From the electrochemical data shown in table 1 it is clear that groups attached to the nitrogen atom in the ring of 1,8-naphthalimide do not significantly affect the redox property of these compounds. In the case of N-2-hydroxyethyl 1,8-naphthalimide it is observed that the redox couple is affected by the addition of poly-hydroxy aromatic compound. Thus the reversible redox couple present in the parent molecule (5) is lost when 1,3-dihydroxy benzene or 1,3,5-trihydroxybenzene is added to a solution of N-2hydroxy ethyl 1,8-naphthalimide. This is in accordance with the results on the formation of 1:1 adduct of N-2-hydroxy ethylphthalimide with 1,3-dihydroxybenzene or 1,3,5-trihydroxy benzene.²⁶ Quenching of the redox process occurs due to the formation of adduct of 1,8-naphthalimide derivative (5) with polyhydroxy aromatic compounds. It has been suggested that the binding of o-quinone derivatives to the dendrimeric hosts leads to lowering of their reduction potential and stabilization of the anionic radicals.²⁷ The ¹H NMR spectrum of N-2-hydroxyethyl-1,8-naphthalimide was recorded in deuterated chloroform solution without 1,3-dihydroxybenzene and with its addition at different concentration ratios. This study did not differentiate between chemical shifts of the original substrates and adducts, except that broadening of the OH-signal present at 3 ppm occurs when 1,3-dihydroxybenzene is added, leaving the other signals intact at the original positions. This suggests that there is a slow exchange of protons between the 1,3-dihydroxybenzene and N-2-hydroxyethyl 1,8-naphthalimide.

From the above discussion it is clear that the substituent attached to the nitrogen atom in 1,8naphthalimide does not significantly affect the redox property, but the supramolecular assembly formation can affect the redox potential. With this background, we extended our study to incorporate the 1,8naphthalimide unit in the copper complex with a

Complex	Elemental analysis (%)	IR (KBr, cm^{-1})	TG (temperature and weight loss) ^{\$}	$E_{1/2}; \\ (\ddot{A}E ip_c/ip_a)^{\#}$
8	Found: C, 51.62 H, 2.68, N, 6.27 Calcd. for ML ₂ (ClO ₄) ₂ : C, 51.52; H, 2.86; N, 6.68	2930(<i>s</i>), 2858(<i>s</i>), 1701(<i>s</i>), 1665(<i>s</i>), 1624(<i>s</i>), 1440(<i>s</i>), 1388(<i>s</i>), 1245(<i>s</i>), 1112(<i>s</i>), 1061(<i>s</i>), 963(<i>s</i>), 789(<i>s</i>)	225–270°C; 78·12% [calcd. 78·55% for loss of 2L followed by loss of HClO ₄ via hydrolytic reaction to give $Cu(OH)ClO_4$]	-1240 mV; (81 mV, 1·32)
9	Found: C, 53·6, H, 3·27, N, 7·49 Calcd. for ML ₂ (NO ₃) ₂ .2H ₂ O C, 54·03, H, 3·50, N, 7·00	3452(<i>bw</i>), 3063(<i>w</i>), 2976(<i>w</i>), 1701(<i>s</i>), 1665(<i>s</i>), 1496(<i>s</i>), 1440(<i>s</i>), 1387(<i>s</i>), 1291(<i>s</i>), 1235(<i>s</i>), 1173(<i>s</i>), 1015(<i>s</i>), 953(<i>s</i>), 779(<i>s</i>)	50–80°C; 3.9% (calcd. 4.5% for loss of water of crystallization) 240-300°C; $33.23%(calcd. 36.02\%, for lossof one ligand)240-430°C$; $31.87%(calcd. 36.02\% for lossof another ligand)450-600°C$; $17.08%[calcd. 16\%, for loss ofHNO3 via reaction withmoisture to giveCu(OH)NO3]$	-1238 mV (80 mV, 1·34)
10	Found: C, 45·33, H, 2·82, N, 7·10 Calcd. for ML ₂ (ClO ₄) ₂ , C, 44·89; H, 2·67; N, 7·48	2930(<i>s</i>), 2858(<i>s</i>), 1778(<i>w</i>), 1726(<i>s</i>), 1624(<i>m</i>), 1440(<i>s</i>), 1404(<i>s</i>), 1122(<i>s</i>), 1050(<i>s</i>), 943(<i>m</i>), 723(<i>s</i>)	220–450°C; 73.59% [calcd. 75.76% for loss of two-ligands, followed by hydrolytic cleavage to release HClO ₄ and form Cu(OH)ClO ₄]	-292 mV (270 mV, d 0.279)

 Table 2.
 Spectroscopic data for copper complexes (8–10).

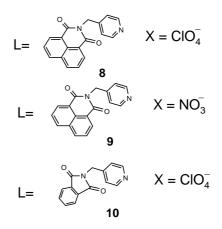


view to have a redox active metal centre that may in turn affect the electrochemistry of the 1,8-naphthalimide unit. Since the 1,8-naphthalimide is not a good enough nitrogen donor to form the complex, we attempted synthesizing copper (II) complexes with ligand (5) having a pyridine group attached to the 1,8-naphthalimide unit. This ligand did not give properly isolable copper complexes, presumably due to steric congestion. However, ligand (6) and (7) form well-characterisable copper (II) complexes with nitrate and perchlorate as anion (see (2) below). They have good solubility in methanol, acetonitrile etc. to study them in solution. In each case a ML_2X_2 type complex (where L = ligand and X = anion) is observed with or without water of crystallisation.

$$2L + CuX_2 nH_2 O \longrightarrow L_2 CuX_2$$

$$8-10$$
(2)

when



The molar conductance value of each complex has value ranging from 20-40 ohm⁻¹ cm² depending on solvent under consideration. This suggests that they are nonionic. This is attributed to the fact that the nitrate and perchlorate groups in these complexes are coordinated to the copper (II) ion in both mono-

dentate and bidentate fashion to complete five coordination geometry. Such observation is common in copper (II) coordination chemistry, as many pentacoordinated copper (II) complexes with overall N₂O₃ co-ordination around copper (II) is well documented.^{28,29} The minimised energy structure of (9) having two N-(4-pyridylmethyl)-1,8-naphthalimide ligands along with two nitrato groups attached to copper (II) ion is shown in figure 1. The elemental analysis of these complexes suggests that in the case of nitrate complex (9) there are two molecules of water of crystallization. This is supported by loss of weight from (9) observed at relatively low temperature (50-80°C) and is further supported by observation of broad and weak absorbance in IR spectra at 3452 cm⁻¹ due to OH-stretching vibrations. A freshly prepared and well-dried sample of (9) is devoid of these features; but on standing the compound under ordinary conditions rapidly picks up moisture.

Thermogravimetry shows that perchlorate complexes (8) and (10) lose the ligands at relatively low temperature; for example in the case of (8) it occurs at 225–270°C whereas in the case of (10) it occurs at 220-250°C. Final weight loss in both the compounds corresponds to formation of Cu(OH)ClO₄. Presumably this occurs, through a hydrolytic reaction of anhydrous copper (II) perchlorate formed as intermediate and liberation of HClO₄. There is a difference in the mass loss pattern between the two complexes; in the case of (10) weight loss is spread over the range 220-460°C, of which the loss at the region 220-250°C is sharp and corresponds to loss of the two neutral ligands and weight loss beyond 250°C corresponds to hydrolytic cleavage with traces of moisture in the nitrogen gas. However, the loss of weight in the case of (9) occurs in a very narrow region; there is a sharp loss of weight at 225°C, with a small increase in weight (226-235°C, 1.9%), followed by loss of weight (226-270°C, 3.9%), this presumably happens due to a loss of ligand and the

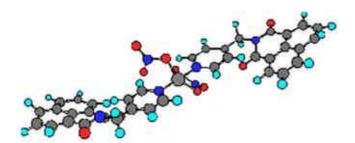


Figure 1. Energy minimized structure of (9).

hydrolytic reaction of anhydrous copper (II) perchlorate over a very narrow temperature range. It is a well established fact that anhydrous copper (II) perchlorate is generally produced in solution and is highly reactive.³⁰ This could be the reason for a hydrolytic cleavage in the reaction with traces of moisture present in nitrogen as impurity. In the case of the nitrate complex, the thermogram is relatively complicated and involves four steps. The first step corresponds to the loss of water of crystallization, second and third steps to stepwise loss of ligand whereas the fourth step involves a hydrolytic reaction of copper (II) nitrate to form Cu(OH)NO₃, thereby releasing HNO₃. In our earlier study it was observed that in bis-(N-phenyl 3,5-dimethylpyrazole) copper (II) nitrate loses N-phenyl 3,5-dimethylpyrazole ligands and N₂O₃ on heating to 450°C to finally give cupric oxide.³¹

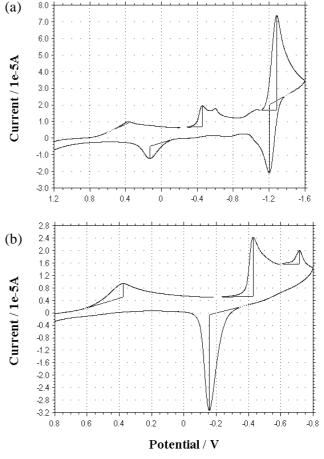


Figure 2. Cyclic voltammogram of (8) (a) and (10) (b) in acetonitrile TBAP as supporting electrolyte; platinum electrodes as working electrodes and Ag/AgCl as reference electrode (+ve scan, 100 mV/s) and potentials are with respect to the ferrocene couple.

The electrochemical study of the complex has shown no significant change in the reversible redox couple of the ligand. In the case of complex (10) the quasi-reversible Cu(I)/Cu(II) couple occurs at a different position as compared to the similar couple in compounds (8) and (9). This may occur due to the non-interacting nature of the naphthalimide and phthalimide units at a remote site with the redox active metal site. Moreover, the phthalimide derivative (7) is also redox inactive in the region of +1.0 V to -1.6 V (vs SCE). Redox peaks of complexes (8) and (9) in +1.0 V to -1.6 V region from both the metal centers and the ligand are well separated from each other and can be easily distinguished (figure 2).

In conclusion, the reversible redox couples of the 1,8-naphthalimide derivatives presented in this manuscript are not affected by substituents attached to the N-atom or the presence of a redox centre at a remote place, but are affected by supramolecular assembly. This allows scope for use of these substrates as electrochemical probes.

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