Preparation of tetraazamacrocycle ligand with several phenyl groups and structural analysis of Cu(II) complex using this ligand

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[Introduction] Macrocyclic metal complex can not only restrict coordination direction of other ligands but also expected to exhibit chromism and fluorescence properties when functional groups are introduced in macrocyclic ligand. In this study, we introduced phenyl groups into tetraazamacrocycle ligand to synthesize 5,7,12,14-tetraphenyl-1,4,8,11-tetraaza-cyclotetradecane (Ph_4-[14]-decane). And we tried to prepare a Cu(II) complex with this ligands as well as a multi-dimensional complex (compound 1) using bridging ligand.

[Experiments] The ligand precursor was derived by refluxing ether-cyclohexane (2:1 v/v) mixed solution of chalcone (1 eq.) and ethylenediamine (1 eq.) for 3 hours. After refluxing EtOH solution of this precursor (1 eq.) with ethylenediamine (1 eq.) for 3 hours, $NaBH_4$ was added to this solution and again refluxed for 3 hours to prepare Ph_4-[14]-decane. Molar ratio method was applied to confirm complexation of this ligand with several metal ions. The powder X-ray diffraction (PXRD) pattern of compound 1 was investigated by using expo2014 program.

[Results] In Fig.1, ^{1}H NMR spectrum of prepared ligand was shown. From the analysis of this spectrum, we could confirm that Ph_4-[14]-decane was successfully synthesized.

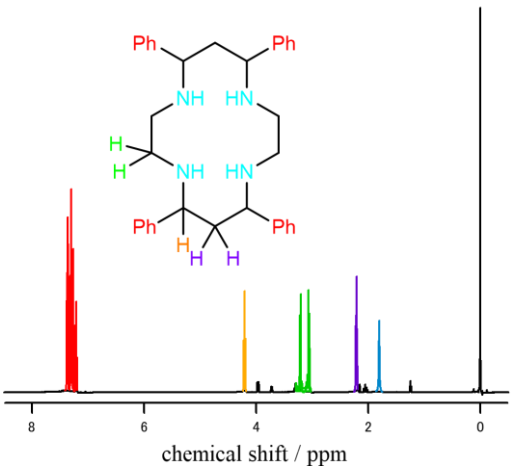

Fig. 1 ^{1}H NMR spectrum of Ph_4-[14]-decane

In ^{1}H NMR spectrum measurements with increasing metal ion concentrations (Molar ratio method), the peak shifts and broadenings were observed, which clearly indicates complexation. The dissociation constants of these complexes could be determined through the examination of peak broadenings.

The analysis of PXRD pattern of the compound 1 gave the determination of space group ($P21/a$) and lattice constants ($a = 18.2$ Å, $b = 11.9$ Å, $c = 11.9$ Å, $\beta = 95.9°$).

The details of these results will be given at the meeting.