A8 15:45-16:00

Synthesis of Tetrahomodioxacalix[4]arene tetraamide and Crystal Structure of Lead Ion Complex

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1,4-Alternate tetrahomodioxacalix[4]arene tetraamide having four *p*-phenyl groups on the upper rim has been constructed. Lead ion selectivity was observed from the ^{1}H NMR, ^{13}C NMR and two-phase extraction. They formed 1:1 complexes with Pb^{2+} in chloroform. From the X-ray crystal structure, Pb^{2+} is found to bind to two-carbonyl oxygen atoms and one phenoxy oxygen atom of the homocalix. Crystal structure and ^{1}H NMR of the ligand-Pb^{2+} complex illustrate that a rotated aromatic unit through the homooxa group positioned in paralleled direction to the Pb^{2+} to have a π-metal complexation mode. The Pb^{2+} complexed with two amide groups of lower rim of the aromatic ring *A* and *B* moves into another two amide groups of the rotated two aromatic rings by passing through the center of the homocalixarene cavity.