**Abstract**

Oil-bath reaction of respective metal nitrate with an aqueous mixture of oxydiacetic acid (H2oda) and hydrazine hydrate led to the formation of crystalline compounds with formula (N2H5)3[Ln(oda)3]·2.5H2O (where Ln = La, Ce, Pr, Nd and Sm), which are stable for a week and undergo efflorescence. The resulting complexes were characterized by infrared spectral, thermal (air and nitrogen atmosphere), UV–visible and PXRD studies. From the thermal studies, both in air and nitrogen atmosphere, these compounds show endothermic dehydration below 100 °C to give anhydrous compounds. Next, the anhydrous compounds (in air) undergo endothermic decomposition between 190 and 225 °C to form Ln(Hoda)3 intermediate, which further show exothermic decomposition to yield respective metal oxide as the end residue. But, in nitrogen atmosphere, the same anhydrous compounds exhibit endo-followed by exothermic decompositions to give respective metal as end product. This is observed as a continuous single step of decomposition in TG. The structure of (N2H5)3[Nd(oda)3]·2.5H2O has been determined by single-crystal X-ray analysis. The neodymium atom is coordinated by nine oxygen atoms from three tridentate (O, O, O) oxydiacetate ions with tricapped trigonal prismatic geometry. In addition, both the parent acid and its compounds display strong fluorescent emission due to the ligand, which renders them as fluorescent materials at room temperature.