

## **Supporting informations**

### **1. Experimental Details**

#### ***Synthesis of $(C_8H_{17})_2NCOCH_2OCOCH_3$***

To a solution of glacial acetic acid ( 5.8 gm , 0.097 mol) in 1,2- dichloroethane (100 mL), triethyl amine ( 10 gm, 0.099 mol ) was added slowly with stirring. To this solution,  $(C_8H_{17})_2NCOCH_2Cl$  ( 30 gm, 0.095 mol) and tetrabutyl ammonium bromide ( 200 mg, as a catalyst) was added and refluxed for 50 h. The solution was treated with 200 mL of 5% HCl, the organic phase was separated , dried over anhydrous  $Na_2SO_4$  and filtered. This solution on slow evaporation yielded ( 28 gm, 87.5% based on ester ) pale yellow liquid. IR( nujol,  $\nu$   $cm^{-1}$ ) : 1753(CO, ester ); 1668 ( CO, amide);  $^1H$  NMR (  $CDCl_3$ , 300 MHz,  $\delta$  in ppm ) : 0.88 (m,  $CH_3$ ); 1.28 (br,  $CH_2$ ) ; 1.55(m,  $CH_2$ ); 2.1 (s,  $CH_3$ , ester) ; 3.14 ( t,  $NCH_2$  ) ; 3.3 (t,  $NCH_2$ ); 4.7 (s,  $OCH_2CO$  ). (Spectra shows that the product is a mixture of acetate ester and final hydroxo compound).

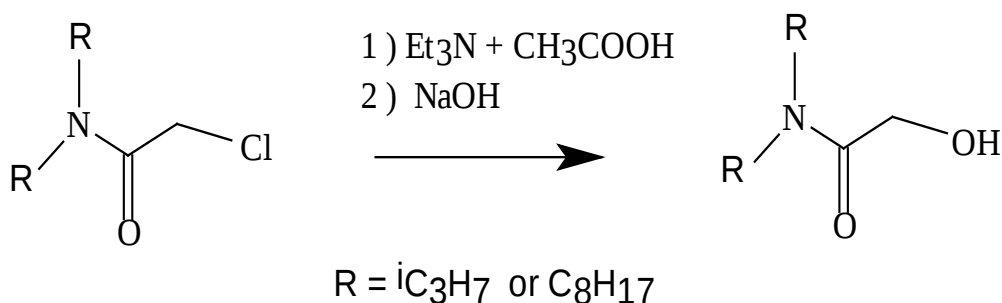
#### ***Synthesis of $(C_8H_{17})_2NCOCH_2OH$***

To a solution of NaOH ( 3.4 gm, 0.085 mol) in methanol, the above mixture ( 27 gm ) was added slowly with stirring. The solution was allowed to stir for an hour, refluxed for 30 min and treated with 200 mL of 10% HCl. The organic phase was extracted with di-isopropyl ether, dried over anhydrous  $Na_2SO_4$  and filtered. This solution on evaporation yielded (22 gm, 95% based on ester) the desired product. IR( nujol,  $\nu$   $cm^{-1}$ ) : 3411 (br, OH); 1652 (CO).  $^1H$  NMR (  $CDCl_3$ , 300 MHz,  $\delta$  in ppm ) : 0.84 (br, 6H,  $CH_3$ ); 1.25 ( br, 20H,  $CH_2$ ) ; 1.5 ( br, 4H,  $CH_2$ ); 3.01( t, 2H,  $NCH_2$ ) ; 3.32 ( t, 2H,  $NCH_2$ ) ; 3.49 ( br, 1H, OH); 4.10( s, 2H,  $CO-CH_2-O$  ). ES-MS(  $CH_2Cl_2$ , m/z ) : 300 ( $LH^+$  , 100%); 322( $L+Na$ , 22% ) ; 621 (  $2L+Na$ , 18%)

#### ***Synthesis of $(^iC_3H_7)_2NCOCH_2OH$***

This was prepared similarly to the above reported ligand by taking  $(^iC_3H_7)_2NCOCH_2Cl$ . The final product was obtained as a off white crystalline solid in 85% yield. IR( nujol,  $\nu$   $cm^{-1}$ ) : 3353

(br, OH); 1649 (CO).  $^1\text{H}$  NMR (  $\text{CDCl}_3$ , 500 MHz,  $\delta$  in ppm ) : 1.2 (d, 6H,  $\text{CH}_3$ ); 1.425 (d, 6H,  $\text{CH}_3$ ); 3.5 (m, 1H, CH); 3.6 (m, 1H, CH); 3.92 (t, 1H, OH); 4.09 (s, 2H, CO- $\text{CH}_2$ -O).



**Scheme 1** : Synthesis of N, N'- alkyl,  $\alpha$ - hydroxy acetamide ligand

***Synthesis of  $\text{La}(\text{NO}_3)_3 + (\text{iC}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$  compound***

To a solution of  $(\text{iC}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$  ( 370 mg, 2.3 mmol) in  $\text{CH}_2\text{Cl}_2$ , solid  $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  ( 200 mg, 0.46 mmol) was added and stirred ( about 10 min) till all lanthanum salts dissolve to give a clear solution. The solution on further stirring deposited white powder and the whole solution was allowed to stir for further 3hr. The resultant powder was filtered and washed with  $\text{CH}_2\text{Cl}_2$  ( about 5 ml) and dried. This powder was recrystallized from acetone/  $\text{ClCH}_2\text{CH}_2\text{Cl}$  mixture to give crystalline solid in 90% yield. IR( nujol,  $\nu \text{ cm}^{-1}$ ) : 3322 (br, OH); 1606 (CO).  $^1\text{H}$  NMR (  $\text{CD}_3\text{OD}$ , 500 MHz,  $\delta$  in ppm ) : 1.22 (d, 6H,  $\text{CH}_3$ ); 1.40 (d, 6H,  $\text{CH}_3$ ); 3.61 (m, 1H, CH); 3.72 (m, 1H, CH); 4.47 (s, 2H, CO- $\text{CH}_2$ -O).

***Synthesis of  $\text{Sm}(\text{NO}_3)_3 + (\text{iC}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$  compound***

This was prepared similar to lanthanum compound by taking  $(\text{iC}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$  (370 mg, 2.3 mmol) and  $\text{Sm}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  ( 200 mg, 0.45 mmol) in 80% yield. IR( nujol,  $\nu \text{ cm}^{-1}$ ) : 3437 - 3000 (br, OH); 1603 (CO).  $^1\text{H}$  NMR (  $\text{CD}_3\text{COCD}_3$ , 300 MHz,  $\delta$  in ppm ) : 1.34 (d, 6H,  $\text{CH}_3$ ); 1.57 (d, 6H,  $\text{CH}_3$ ); 3.81 (m, 1H, CH); 3.98 (m, 1H, CH); 4.6 (s, 2H, CO- $\text{CH}_2$ -O).

***Synthesis of  $\text{Eu}(\text{NO}_3)_3 + (\text{iC}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$  compound***

This was prepared similar to lanthanum compound by taking  $(^i\text{C}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$  (370 mg, 2.3 mmol) and  $\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  ( 200 mg, 0.45 mmol) in 85% yield. IR( nujol,  $\nu$   $\text{cm}^{-1}$ ) : 3437 - 3000 (br, OH); 1613 (CO).  $^1\text{H}$  NMR (  $\text{CD}_3\text{COCD}_3$ , 300 MHz,  $\delta$  in ppm ) : 0.245(br); 0.5( br ) ; 2.65 ( br): ( broad peaks due to paramagnetic nature).

### ***Solvent extraction experiment***

Distribution studies were performed by using a 0.2M solution of  $(\text{C}_8\text{H}_{17})_2\text{NCOCH}_2\text{OH}$  or  $(\text{C}_8\text{H}_{17})_2\text{NCOCH}_2\text{Cl}$  in dodecane with the aqueous phases spiked with  $^{233}\text{U}$ ,  $^{239}\text{Pu}$ ,  $^{241}\text{Am}$ ,  $^{152,154}\text{Eu}$ ,  $^{137}\text{Cs}$  or  $^{88,89}\text{Sr}$  tracer in a thermostated water bath for 30 min at  $25 \pm 0.1$  C. Assay of organic and aqueous phases were done in duplicate by alpha counting using dioxane based scintillator for  $^{233}\text{U}$  and  $^{239}\text{Pu}$  and gamma counting directly for  $^{241}\text{Am}$ ,  $^{152,154}\text{Eu}$ ,  $^{137}\text{Cs}$  and  $^{85,89}\text{Sr}$ . The distribution ratio (D) is defined as the ratio of the concentration of metal ion in organic phase to that of the aqueous phase.

### ***Crystallography***

Crystal data for  $\text{C}_{24}\text{H}_{52}\text{N}_6\text{O}_{16}\text{Sm}$  and  $\text{C}_{24}\text{H}_{53}\text{EuN}_6\text{O}_{16}$  were measured on a Agilent SuperNova system equipped with Titan CCD detector at 293(2) K using  $\text{CuK}_\alpha$  radiation (  $\lambda = 1.5418 \text{ \AA}$  ). The crystals were positioned at 101 mm from the CCD. 1481 and 1486 frames were measured respectively for Sm and Eu compounds with a counting time of 1 s. Data analysis were carried out with the CrysAlis program<sup>1</sup>. The structure were solved using direct methods with the Shelxs97 program<sup>2</sup>. All non- hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms bonded to carbon atoms were included in the geometric positions and given thermal parameters equivalent to 1.2 times those of the atoms to which they attached. Empirical absorption corrections were carried out using the ABSPACK program<sup>3</sup>. The structures were refined to convergence on  $F^2$  using Shelxl97<sup>b</sup>. Selected crystallographic data and important bond distances and angles are given below.

1. CrysAlis, (2006) Oxford Diffraction Ltd, Abingdon, U. K .
2. Sheldrick, G. M. Shelxs97 and Shelxl97, program for crystallographic

solution and refinement, *Acta Crystallogr.* **2008**, A64, 112 -122.

3. ABSPACK ( **2005**) Oxford Diffraction Ltd, Oxford, U. K.

**Crystal data for  $C_{24}H_{52}N_6O_{16}Sm$**

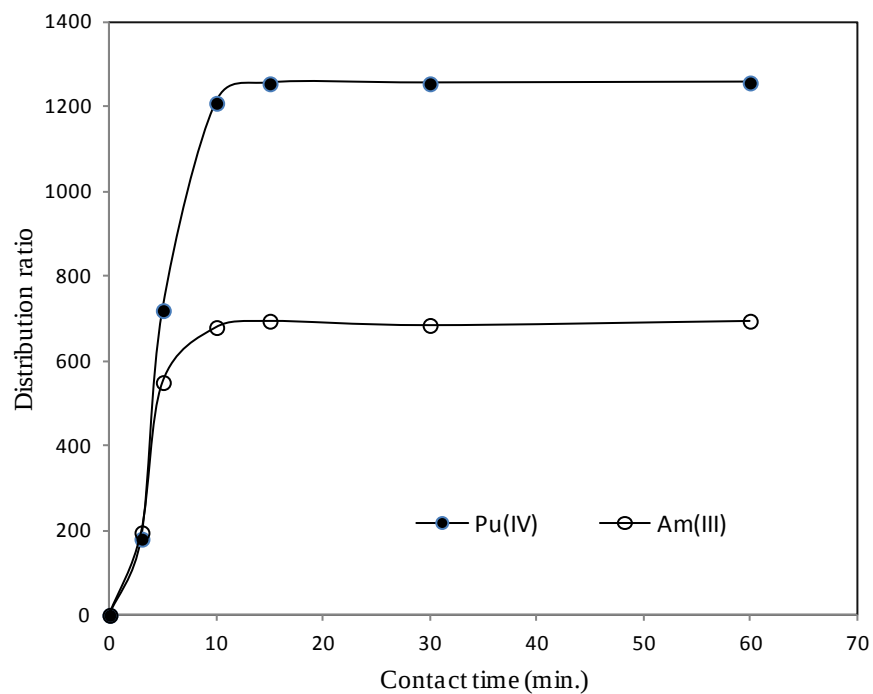
Monoclinic, space group  $P2_1/c$  (no. 14),  $a = 10.9512(4)$  Å,  $b = 27.0203(9)$  Å,  $c = 13.0160(4)$  Å,  $\beta = 101.549(3)^\circ$ ,  $V = 3773.5(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $T = 293(2)$  K,  $\mu(\text{Cu K}\alpha) = 12.306$  mm<sup>-1</sup>,  $D_{\text{calc}} = 1.463$  g/mm<sup>3</sup>, 23664 reflections measured ( $6.542 \leq 2\theta \leq 140.212$ ), 7093 unique ( $R_{\text{int}} = 0.1403$ ) which were used in all calculations. The final  $R_1$  was 0.0774( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.2243 (all data). CCDC-977001.

**Crystal Data for  $C_{24}H_{53}EuN_6O_{16}$**

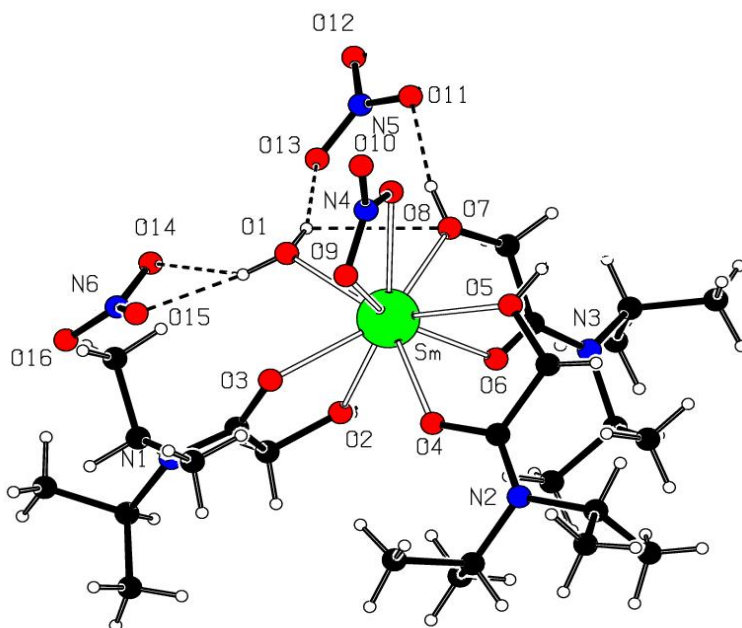
Monoclinic, space group  $P2_1/c$  (no. 14),  $a = 10.9253(2)$  Å,  $b = 26.9890(5)$  Å,  $c = 13.0207(3)$  Å,  $\beta = 101.5301(19)^\circ$ ,  $V = 3761.85(14)$  Å<sup>3</sup>,  $Z = 4$ ,  $T = 293(2)$  K,  $\mu(\text{Cu K}\alpha) = 12.557$  mm<sup>-1</sup>,  $D_{\text{calc}} = 1.472$  g/mm<sup>3</sup>, 23625 reflections measured ( $6.55 \leq 2\theta \leq 140.066$ ), 7093 unique ( $R_{\text{int}} = 0.0843$ ) which were used in all calculations. The final  $R_1$  was 0.0656 ( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.1851 (all data). CCDC-977002

**Table 1.** Important bond lengths ( Å ) and agles ( ° ) for  $C_{24}H_{52}N_6O_{16}Eu$  and  $C_{24}H_{52}N_6O_{16}Sm$

|             | Eu       | Sm        |
|-------------|----------|-----------|
| M – O1      | 2.422(5) | 2.430(8)  |
| M – O2      | 2.380(4) | 2.458(7)  |
| M – O3      | 2.451(4) | 2.401(6)  |
| M – O4      | 2.368(5) | 2.398(7)  |
| M – O5      | 2.514(5) | 2.527(7)  |
| M – O6      | 2.457(5) | 2.409(7)  |
| M – O7      | 2.400(5) | 2.464(7)  |
| M – O8      | 2.500(5) | 2.531(7)  |
| M – O9      | 2.531(5) | 2.535(8)  |
| O9 – M – O8 | 50.2(2)  | 50.41(16) |
| O2 – M – O3 | 63.2(2)  | 63.18(15) |
| O4 – M – O5 | 62.4(2)  | 63.02(14) |
| O6 – M – O7 | 62.6(2)  | 62.49(15) |



**Fig. 1: Distribution ratios of Pu and Am as a function of time**



**Fig 3 : Structure of  $[\text{SmL}_3(\text{NO}_3)(\text{H}_2\text{O})][\text{NO}_3]_2$  (  $\text{L} = (\text{C}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$  )**

## 2. Solvent Extraction data

**Table 2. Extraction of Pu(IV) at different concentration of nitric acid**

*Extraction Parameters* : Aqueous feed: Pu(IV) spiked HNO<sub>3</sub> solution at different acidity  
Conc. of Extractant: 0.20M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL,  
Contact Time: 30 min.

| Conc. of<br>HNO <sub>3</sub> in Feed<br>(M) | $\alpha$ -activity of Pu<br>in Feed<br>(cpm for 50 $\mu$ L) | $\alpha$ -activity of Pu<br>in Raffinate<br>(cpm for 50 $\mu$ L) | Extraction<br>(%) $D_{\text{Pu(IV)}}$ |      |
|---------------------------------------------|-------------------------------------------------------------|------------------------------------------------------------------|---------------------------------------|------|
| 0.50                                        | 44250                                                       | 2601                                                             | 94.12                                 | 16.0 |
| 1.00                                        | 44005                                                       | 1630                                                             | 96.30                                 | 26.0 |
| 2.00                                        | 43010                                                       | 251                                                              | 99.42                                 | 170  |
| 3.00                                        | 43901                                                       | 36                                                               | 99.92                                 | 1218 |
| 5.00                                        | 43595                                                       | 32                                                               | 99.93                                 | 1361 |
| 7.00                                        | 43270                                                       | 35                                                               | 99.92                                 | 1235 |
| 10.00                                       | 43308                                                       | 34                                                               | 99.92                                 | 1302 |

**Table 3. Extraction of Am(III) at different concentration of nitric acid**

*Extraction Parameters* : Aqueous feed: <sup>241</sup>Am spiked HNO<sub>3</sub> solution at different acidity  
Conc. of Extractant: 0.2M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL,  
Contact Time: 30 min.

| Conc. of<br>HNO <sub>3</sub> in<br>Feed<br>(M) | <sup>241</sup> Am $\gamma$ activity<br>in Feed<br>(cpm for 100 $\mu$ L) | <sup>241</sup> Am $\gamma$ activity<br>in Raffinate<br>(cpm for 100 $\mu$ L) | Extraction<br>(%) $D_{\text{Am(III)}}$ |      |
|------------------------------------------------|-------------------------------------------------------------------------|------------------------------------------------------------------------------|----------------------------------------|------|
| 0.50                                           | 17407                                                                   | 14994                                                                        | 13.86                                  | 0.16 |
| 1.00                                           | 17475                                                                   | 13734                                                                        | 21.41                                  | 0.28 |
| 2.00                                           | 17433                                                                   | 2735                                                                         | 84.31                                  | 5.37 |
| 3.00                                           | 17451                                                                   | 675                                                                          | 99.31                                  | 24.8 |
| 4.00                                           | 17450                                                                   | 90                                                                           | 99.5                                   | 193  |
| 5.00                                           | 17445                                                                   | 26                                                                           | 99.85                                  | 669. |
| 7.00                                           | 17424                                                                   | 22                                                                           | 99.87                                  | 791  |
| 10.00                                          | 17469                                                                   | 28                                                                           | 99.84                                  | 623  |

**Table 4. Extraction of Sr at different concentration of nitric acid**

*Extraction Parameters:* Aqueous feed:  $^{85+89}\text{Sr}$  spiked  $\text{HNO}_3$  solution at different acidity  
Conc. of Extractant: 0.2M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL,  
Contact Time: 30 min.

| Conc. of $\text{HNO}_3$ in Feed (M) | $^{85+89}\text{Sr}$ $\gamma$ activity in Feed (cpm per mL) | $^{85+89}\text{Sr}$ $\gamma$ activity in Raffinate (cpm per mL) | Extraction (%) $D_{\text{Sr(II)}}$ |   |
|-------------------------------------|------------------------------------------------------------|-----------------------------------------------------------------|------------------------------------|---|
| 0.50                                | 2276                                                       | 2287                                                            | Nil                                | - |
| 1.00                                | 4259                                                       | 4240                                                            | <0.5%                              | - |
| 2.00                                | 8128                                                       | 8164                                                            | Nil                                | - |
| 3.40                                | 14861                                                      | 14880                                                           | Nil                                | - |

**Table 5. Extraction of Uranium at different concentration of nitric acid**

*Extraction Parameters :* Aqueous feed:  $^{233}\text{U}$  spiked  $\text{HNO}_3$  solution at different acidity  
Conc. of Extractant: 0.2M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL,  
Contact Time: 30 min.

| Conc. of $\text{HNO}_3$ in Feed (M) | $^{233}\text{U}$ $\gamma$ activity in Feed (cpm for 20 $\mu\text{L}$ ) | $^{233}\text{U}$ $\gamma$ activity in Raffinate (cpm for 20 $\mu\text{L}$ ) | Extraction (%) $D_{\text{U(IV)}}$ |       |
|-------------------------------------|------------------------------------------------------------------------|-----------------------------------------------------------------------------|-----------------------------------|-------|
| 0.50                                | 24492                                                                  | 17590                                                                       | 28.18                             | 0.39  |
| 1.00                                | 24475                                                                  | 16230                                                                       | 33.69                             | 0.51  |
| 2.00                                | 24442                                                                  | 14690                                                                       | 39.90                             | 0.66  |
| 3.00                                | 24436                                                                  | 6306                                                                        | 74.19                             | 2.88  |
| 4.00                                | 24498                                                                  | 2184                                                                        | 91.08                             | 10.22 |
| 5.00                                | 24425                                                                  | 1010                                                                        | 95.86                             | 23.18 |
| 7.00                                | 24444                                                                  | 600                                                                         | 97.55                             | 39.74 |
| 10.00                               | 24460                                                                  | 608                                                                         | 97.51                             | 39.23 |

**Table 6. Extraction of Europium at different concentration of nitric acid**

*Extraction Parameters*

Aqueous feed:  $^{152+154}\text{Eu}$  spiked  $\text{HNO}_3$  solution at different acidity  
Conc. of Extractant: 0.2M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL,  
Contact Time: 30 min.

| Conc. of $\text{HNO}_3$ in Feed (M) | $^{152+154}\text{Eu}$ $\gamma$ activity in Feed (cpm for 50 $\mu\text{L}$ ) | $^{152+154}\text{Eu}$ $\gamma$ activity in Raffinate (cpm for 50 $\mu\text{L}$ ) | Extraction (%) $D_{\text{Eu(IV)}}$ |        |
|-------------------------------------|-----------------------------------------------------------------------------|----------------------------------------------------------------------------------|------------------------------------|--------|
| 0.50                                | 24830                                                                       | 24815                                                                            | 0.06                               | 0.0006 |
| 1.00                                | 24880                                                                       | 23120                                                                            | 7.0                                | 0.075  |
| 2.00                                | 24882                                                                       | 12453                                                                            | 49.95                              | 0.99   |
| 3.00                                | 24896                                                                       | 978                                                                              | 96.07                              | 24.4   |
| 4.00                                | 24890                                                                       | 350                                                                              | 98.6                               | 70.1   |
| 5.00                                | 24870                                                                       | 155                                                                              | 99.38                              | 159.5  |
| 7.00                                | 24844                                                                       | 128                                                                              | 99.48                              | 193.1  |
| 10.00                               | 24860                                                                       | 102                                                                              | 99.59                              | 242.7  |

**Table 7. Extraction of Am at different concentration of Extractant**

*Extraction Parameters*

Aqueous feed:  $^{241}\text{Am}$  spiked  $\text{HNO}_3$  solution at 3.0M  
Conc. of Extractant: Varying conc. 0.025-0.5M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL,  
Contact Time: 30 min.

| [Amide], M | Mean D                |
|------------|-----------------------|
| 0.025      | $7.86 \times 10^{-5}$ |
| 0.05       | 0.00374               |
| 0.1        | 0.298                 |
| 0.2        | 24.2                  |

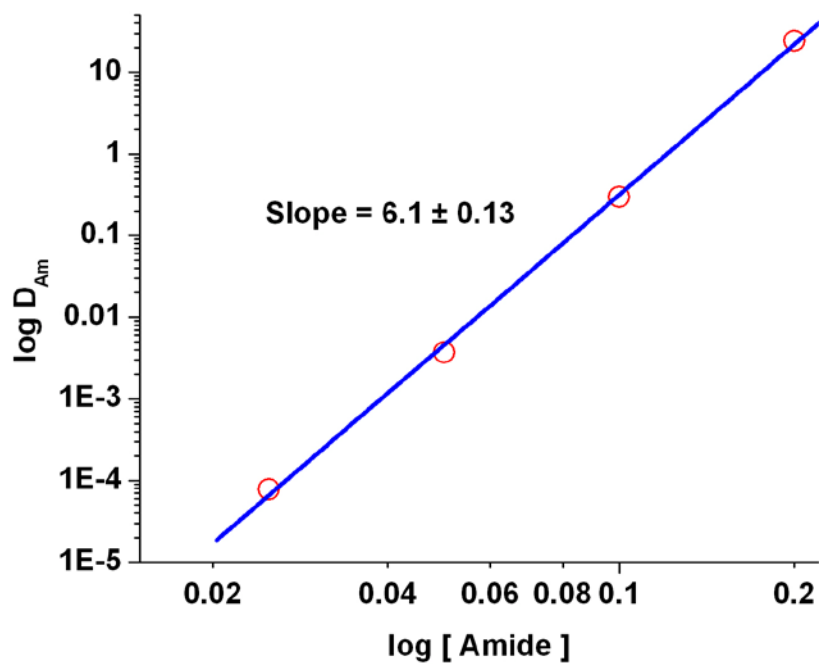


Fig 5. Log D<sub>Am</sub> Vs log [ amide]

Table 8: Variation of D Vs. [NO<sub>3</sub><sup>-</sup>] for Am(III) ; Amide = 0.2 M in dodecane

| [NO <sub>3</sub> <sup>-</sup> ], M | Mean D  |
|------------------------------------|---------|
| 0.5                                | 0.00292 |
| 1.0                                | 0.0106  |
| 2.0                                | 0.0457  |
| 3.0                                | 0.1692  |
| 5.0                                | 1.136   |

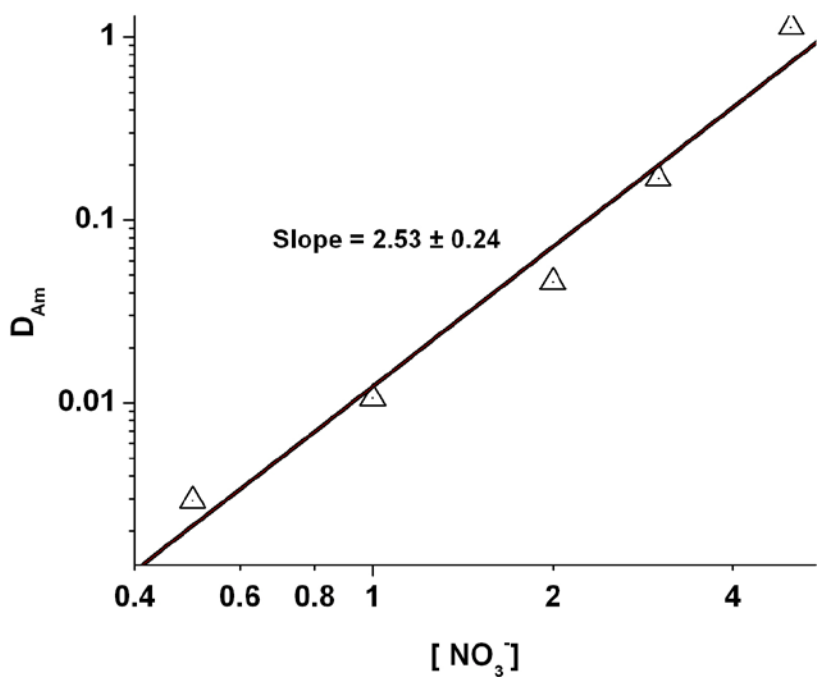


Fig 6. Log D<sub>Am</sub> Vs log [NO<sub>3</sub>]<sup>-</sup>

**Table 9. Extraction of Fission products and Alpha emitter from 100 times diluted Actual High Liquid Waste(HLW) solution using Solvent**

**Extraction Parameters:** Aqueous feed: 100 times diluted HAW-1 solution in 4.0M HNO<sub>3</sub>  
Conc. of Extractant: 0.2M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL,  
Contact Time: 30 min.

| Conc. of HNO <sub>3</sub> in Feed (M) |                       | Alpha<br>α<br>mg/L | Beta<br>β<br>mCi/L | Gamma<br>γ<br>mCi/L | <sup>144</sup> Ce<br>mCi/L | <sup>106</sup> Ru<br>mCi/L | <sup>137</sup> Cs<br>mCi/L | <sup>125</sup> Sb<br>μCi/L |
|---------------------------------------|-----------------------|--------------------|--------------------|---------------------|----------------------------|----------------------------|----------------------------|----------------------------|
| 4.00                                  | Feed                  | 98.9               | 56.81              | 20.11               | 10.21                      | 5.51                       | 8.53                       | 2.75<br>μCi/L              |
|                                       | Raffinate-1 (Aqueous) | <0.2               | 20.91              | 10.54               | 22.00<br>μCi/L             | 5.46                       | 8.43                       | -                          |
|                                       | Raffinate-2 (Aqueous) | <0.2               | 19.88              | 10.32               | 31.68<br>μCi/L             | 5.23                       | 8.18                       | -                          |

|  |                          |      |       |       |                           |      |      |   |
|--|--------------------------|------|-------|-------|---------------------------|------|------|---|
|  | Raffinate-3<br>(Aqueous) | <0.2 | 19.78 | 10.33 | 63.37<br>$\mu\text{Ci/L}$ | 5.09 | 8.22 | - |
|--|--------------------------|------|-------|-------|---------------------------|------|------|---|

**Table 10. Extraction of Fission products and Alpha emitter from 100 times diluted Actual High Active Waste-1(HAW-1) solution using Solvent**

**Extraction Parameters :** Aqueous feed: 100 times diluted HAW-1 solution in 4.0M  $\text{HNO}_3$

Conc. of Extractant: 0.2M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 4 mL,  
Contact Time: 30 min.

| Conc.<br>of<br>$\text{HNO}_3$<br>in Feed<br>(M) |                          | Alpha<br>$\alpha$<br>mg/L | Beta<br>$\beta$<br>mCi/L | Gamma<br>$\gamma$<br>mCi/L | $^{144}\text{Ce}$<br>mCi/L | $^{106}\text{Ru}$<br>mCi/L | $^{137}\text{Cs}$<br>mCi/L | $^{125}\text{Sb}$<br>$\mu\text{Ci/L}$ | $^{95}\text{Nb}$<br>$\mu\text{Ci/L}$ |
|-------------------------------------------------|--------------------------|---------------------------|--------------------------|----------------------------|----------------------------|----------------------------|----------------------------|---------------------------------------|--------------------------------------|
| 4.00                                            | Feed                     | 98.95                     | 57.71                    | 20.15                      | 10.21                      | 5.66                       | 8.43                       | 2.45<br>$\mu\text{Ci/L}$              | 13.78<br>$\mu\text{Ci/L}$            |
|                                                 | Raffinate-1<br>(Aqueous) | <0.2                      | 20.83                    | 10.43                      | 22.00<br>$\mu\text{Ci/L}$  | 5.58                       | 8.40                       | -                                     | -                                    |
|                                                 | Loaded<br>Organic        | 98.7                      | 36.84                    | 9.57                       | 10.2                       | 214<br>$\mu\text{Ci/L}$    | -                          | 1.94<br>$\mu\text{Ci/L}$              | 11.4<br>$\mu\text{Ci/L}$             |

**Table 11. Stripping of Fission products and Alpha emitter from Loaded Organic Solvent using 0.5M Nitric acid**

**Extraction Parameters**

Feed: Loaded Organic ; Strippant: 0.5M  $\text{HNO}_3$   
Conc. of Extractant: 0.2M amide in n-dodecane  
Organic to aqueous phase ratio: 1:1, Volume of each phase: 1 mL,  
Contact Time: 30 min.

|                                   | Alpha<br>$\alpha$<br>mg/L | Beta<br>$\beta$<br>mCi/L | Gamma<br>$\gamma$<br>mCi/L | $^{144}\text{Ce}$<br>mCi/L | $^{106}\text{Ru}$<br>mCi/L | $^{137}\text{Cs}$<br>mCi/L | $^{125}\text{Sb}$<br>$\mu\text{Ci/L}$ | $^{95}\text{Nb}$<br>$\mu\text{Ci/L}$ |
|-----------------------------------|---------------------------|--------------------------|----------------------------|----------------------------|----------------------------|----------------------------|---------------------------------------|--------------------------------------|
| Feed<br>(Loaded<br>Organic)       | 98.7                      | 36.84                    | 9.57                       | 10.39                      | 214<br>$\mu\text{Ci/L}$    | -                          | 1.94<br>$\mu\text{Ci/L}$              | 11.4<br>$\mu\text{Ci/L}$             |
| Raffinate-1<br>(Aqueous<br>Phase) | 98.5                      | -                        | 9.35                       | 9.63                       | 14.9<br>$\mu\text{Ci/L}$   | -                          | 1.92<br>$\mu\text{Ci/L}$              | 6.28<br>$\mu\text{Ci/L}$             |

|               |      |   |      |               |              |   |               |              |
|---------------|------|---|------|---------------|--------------|---|---------------|--------------|
| Organic Phase | <0.2 | - | 0.12 | 89.0<br>μCi/L | 189<br>μCi/L | - | 0.02<br>μCi/L | 5.1<br>μCi/L |
|---------------|------|---|------|---------------|--------------|---|---------------|--------------|

**Table 12. Stripping of Fission products and Alpha emitter from Loaded Organic Solvent using 0.2M Oxalic acid in 0.5M Nitric acid**

***Extraction Parameters***

Feed: Loaded Organic ; Strippant: 0.2M Oxalic acid in 0.5M Nitric acid

Conc. of Extractant: 0.2M amide in n-dodecane

Organic to aqueous phase ratio: 1:1, Volume of each phase: 1mL,

Contact Time: 30 min.

|                                | Alpha<br>α<br>mg/L | Beta<br>β<br>mCi/L | Gamma<br>γ<br>mCi/L | <sup>144</sup> Ce<br>mCi/L | <sup>106</sup> Ru<br>μCi/L | <sup>137</sup> Cs<br>mCi/L | <sup>125</sup> Sb<br>μCi/L | <sup>95</sup> Nb<br>μCi/L |
|--------------------------------|--------------------|--------------------|---------------------|----------------------------|----------------------------|----------------------------|----------------------------|---------------------------|
| Feed<br>(Loaded Organic)       | 92.33              | 28.84              | 9.57                | 10.39                      | 214<br>μCi/L               | -                          | 1.94<br>μCi/L              | 11.4<br>μCi/L             |
| Raffinate-1<br>(Aqueous Phase) | 92.1               | -                  | 8.96                | 9.90                       | 17.8<br>μCi/L              | -                          | 1.94<br>μCi/L              | 10.0<br>μCi/L             |
| Organic Phase                  | <0.2               | -                  | 86.07<br>μCi/L      | 94.0<br>μCi/L              | 196<br>μCi/L               | -                          | -                          | 1.43<br>μCi/L             |

**Extraction behaviour of <sup>95</sup>Zr and <sup>95</sup>Nb using Amide**

Since the direct estimation of small activity of <sup>95</sup>Zr and <sup>95</sup>Nb by gamma spectrometry is difficult in diluted HLLW sample due to the presence of large activity of <sup>137</sup>Cs. Therefore, to study the extraction behavior of <sup>95</sup>Zr and <sup>95</sup>Nb, an experiment was carried out in which 100 times diluted actual HLLW solution was used as feed after depleting <sup>137</sup>Cs by granulated ammonium molybdo phosphate (AMP). Diluted HLLW was subjected to batch contact wherein about 5 mL of the 100 times diluted HLLW solution was contacted with ~200 mg of granulated AMP for about 4h. After contact, AMP was separated and the Cs depleted solution was used for <sup>95</sup>Zr and <sup>95</sup>Nb uptake studies. Analysis of the solution showed <sup>137</sup>Cs removal above 98%. 2 mL of the <sup>137</sup>Cs depleted solution was contacted with 2 mL of 0.2M amide in n-dodecane for ~30 min. Phases were separated and analysed for <sup>95</sup>Zr and <sup>95</sup>Nb by gamma spectrometry. Percentage extraction was calculated using the activity values in feed and organic.

**Table 13: Extraction behaviour of <sup>95</sup>Zr and <sup>95</sup>Nb**

***Extraction Parameters***

Aqueous feed: Cs depleted 100 times diluted HLLW solution using granulated AMP in ~4.0M HNO<sub>3</sub>

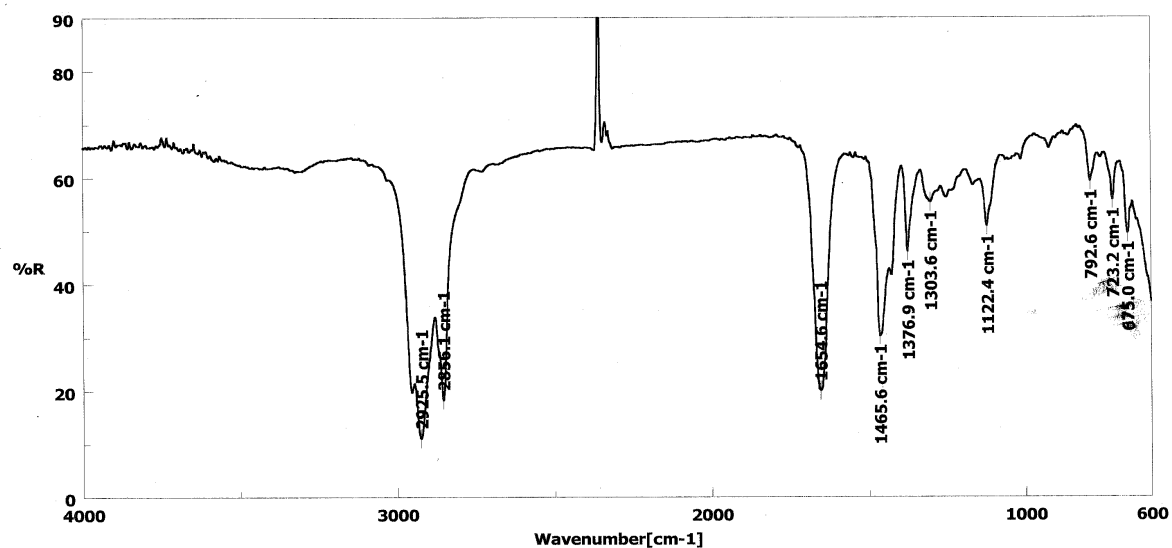
Conc. of Extractant: 0.2M amide in n-dodecane

Organic to aqueous phase ratio: 1:1, Volume of each phase: 2 mL, Contact Time: 30 min.

| Conc. of HNO <sub>3</sub> in Feed | Phases           | Uptake behavior  |                |                  |                |
|-----------------------------------|------------------|------------------|----------------|------------------|----------------|
|                                   |                  | <sup>95</sup> Zr |                | <sup>95</sup> Nb |                |
|                                   |                  | Activity         | Extraction (%) | Activity         | Extraction (%) |
| 3.96M                             | Feed, mCi/L      | 0.400            | 87.50          | 0.042            | 47.62          |
|                                   | Raffinate, mCi/L | 0.059            |                | 0.021            |                |
|                                   | Organic, mCi/L   | 0.350            |                | 0.020            |                |

Above experiment show that solvent extracts ~88% Zr and 48% Nb in single batch contact. Organic phase from extraction experiment was subjected to stripping studies using 0.2M oxalic acid containing 0.5M HNO<sub>3</sub>. Lean organic phase showed <0.01 mCi/L and 0.001 mCi/L of Zr and Nb activity. This indicate that extracted Zr and Nb activity can be stripped using mixture of oxalic acid and nitric acid.

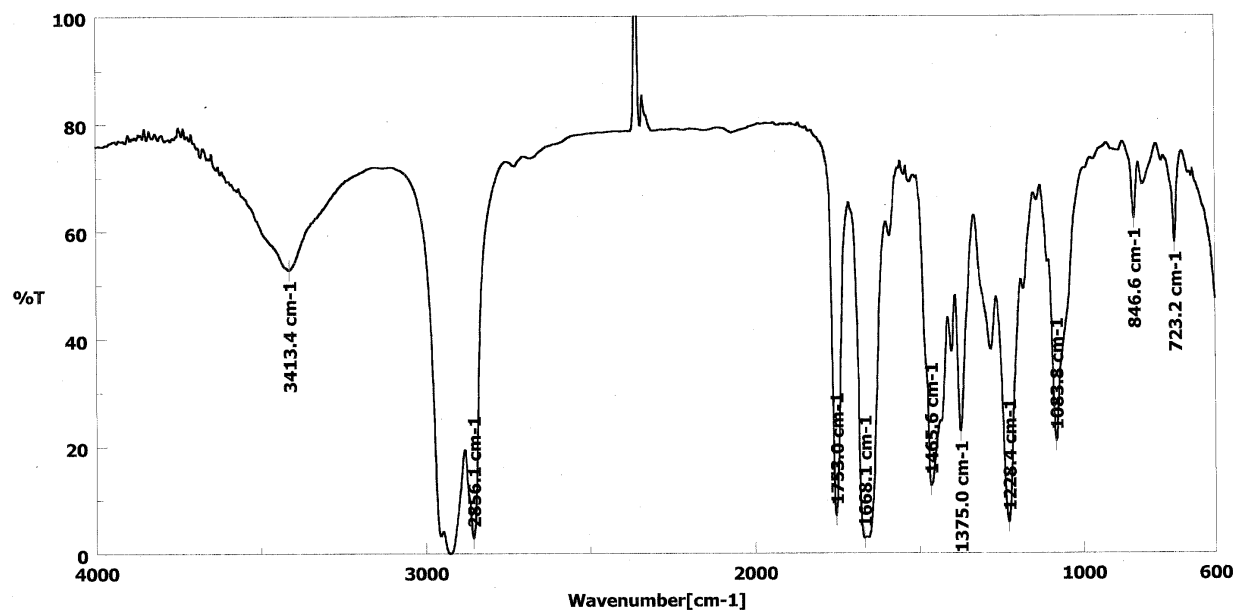
### 3. Spectral Details



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Resolution 4 cm-1  
Zero Filling OFF  
Apodization Cosine  
Gain 16  
Aperture 7.1 mm  
Scanning Speed 2 mm/sec  
Date/Time 1/1/00 0:10AM  
Update 1/1/00 0:10AM  
Operator Dr. Nandkumar Dahale  
File Name (C8H17)2NCOCH2Cl  
Sample Name TiO2 -1  
Comment Nujol

(C8H17)2NCOCH2Cl

S1 : IR spectrum of  $(C_8H_{17})_2NCOCH_2Cl$

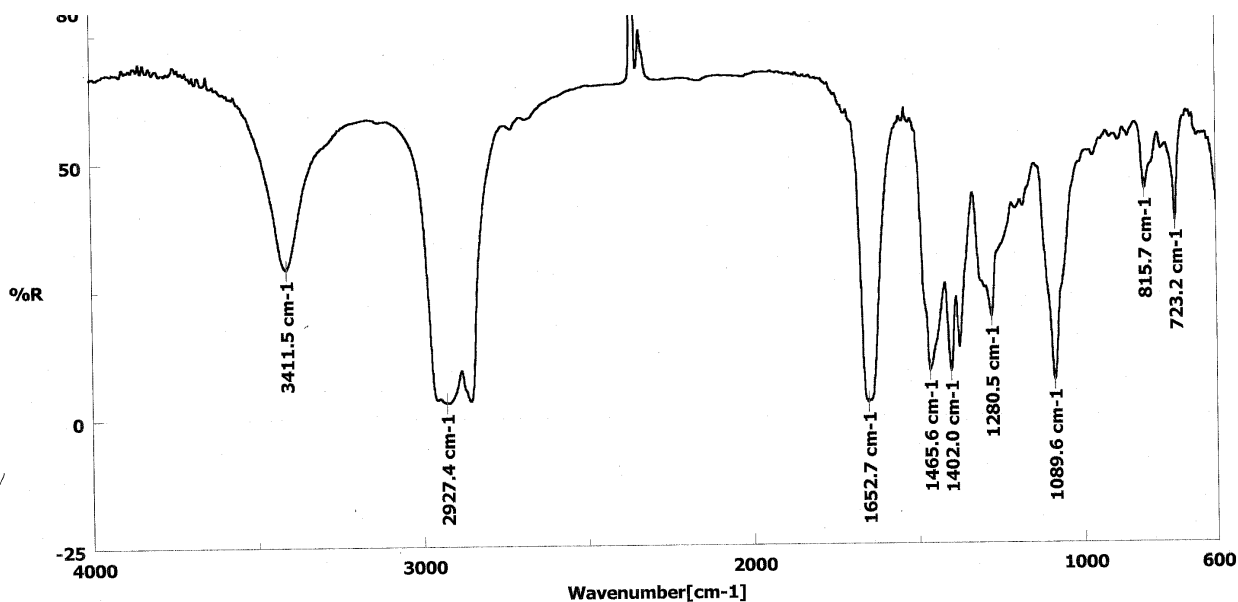


Accumulation 16  
Resolution 4 cm<sup>-1</sup>  
Zero Filling OFF  
Apodization Cosine  
Gain 4  
Aperture 7.1 mm  
Scanning Speed 2 mm/sec  
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Update 1/5/00 6:28PM  
Operator Dr. Nandkumar Dahale  
File Name (C8H17)<sub>2</sub>NCOCH<sub>2</sub>OCOCH<sub>3</sub>  
Sample Name LiOH  
Comment Nujol

(C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>NCOCH<sub>2</sub>OCOCH<sub>3</sub>

S2 : IR spectrum of (C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>NCOCH<sub>2</sub>OCOCH<sub>3</sub>

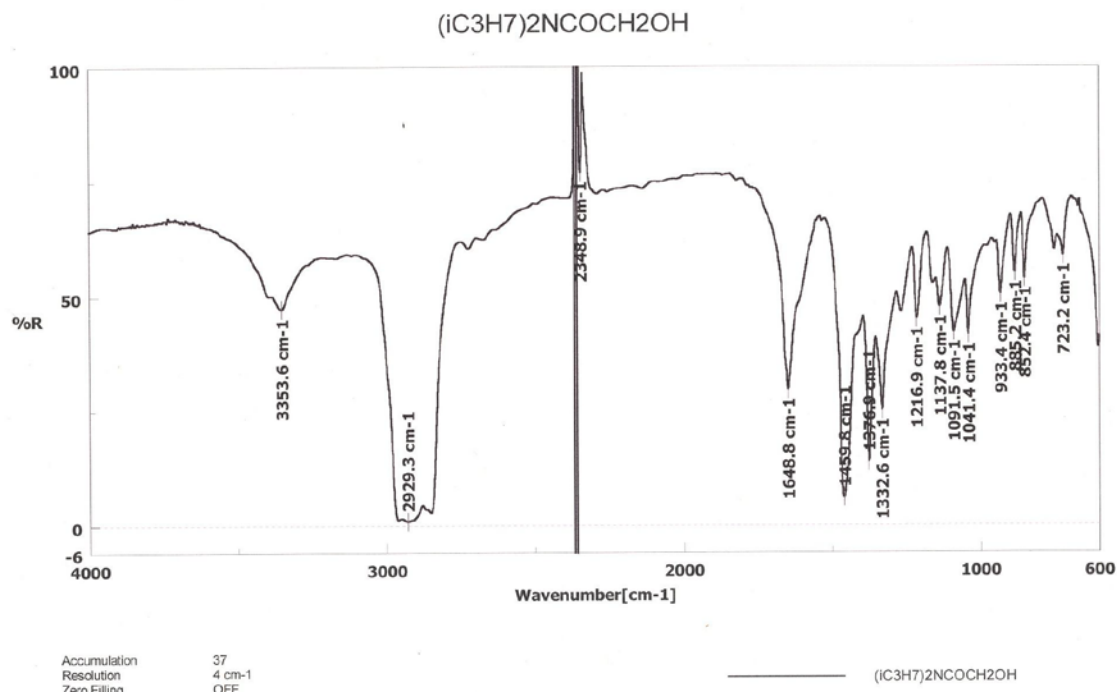
( It is a mixture of intermediate (C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>NCOCH<sub>2</sub>OCOCH<sub>3</sub> and final product (C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>NCOCH<sub>2</sub>OH )



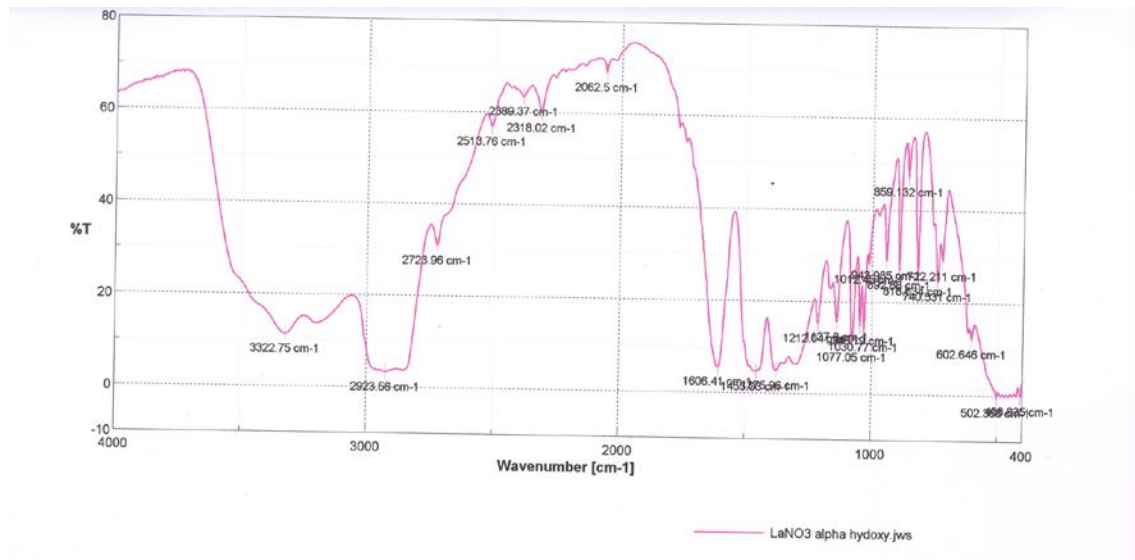
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Resolution 4 cm<sup>-1</sup>  
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Gain 8  
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Operator Dr. S. Kannan  
File Name (C8H17)2NCOCH2OH(31111)  
Sample Name Thorium Phosphate  
Comment KBr

(C8H17)2NCOCH2OH(31111)

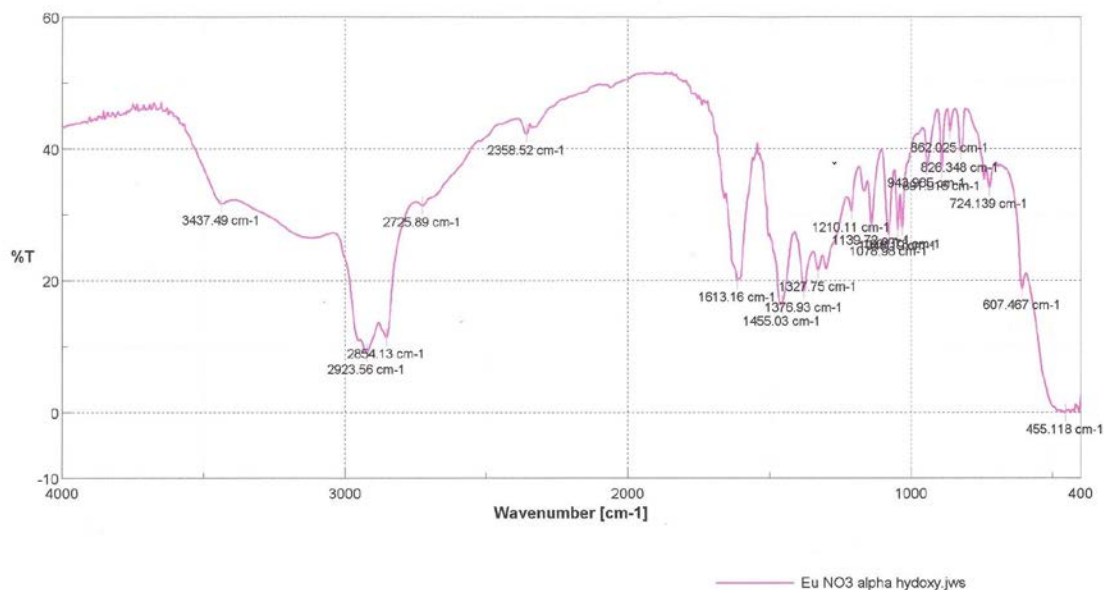
S3 : IR spectrum of (C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>NCOCH<sub>2</sub>OH



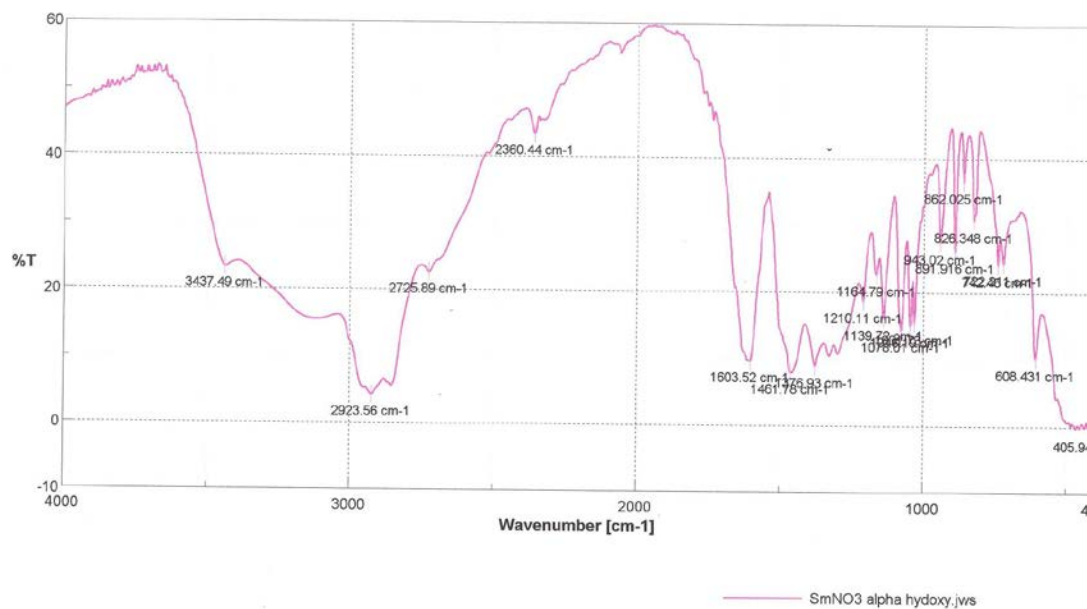
S4 : IR spectrum of (iC<sub>3</sub>H<sub>7</sub>)<sub>2</sub>NCOCH<sub>2</sub>OH



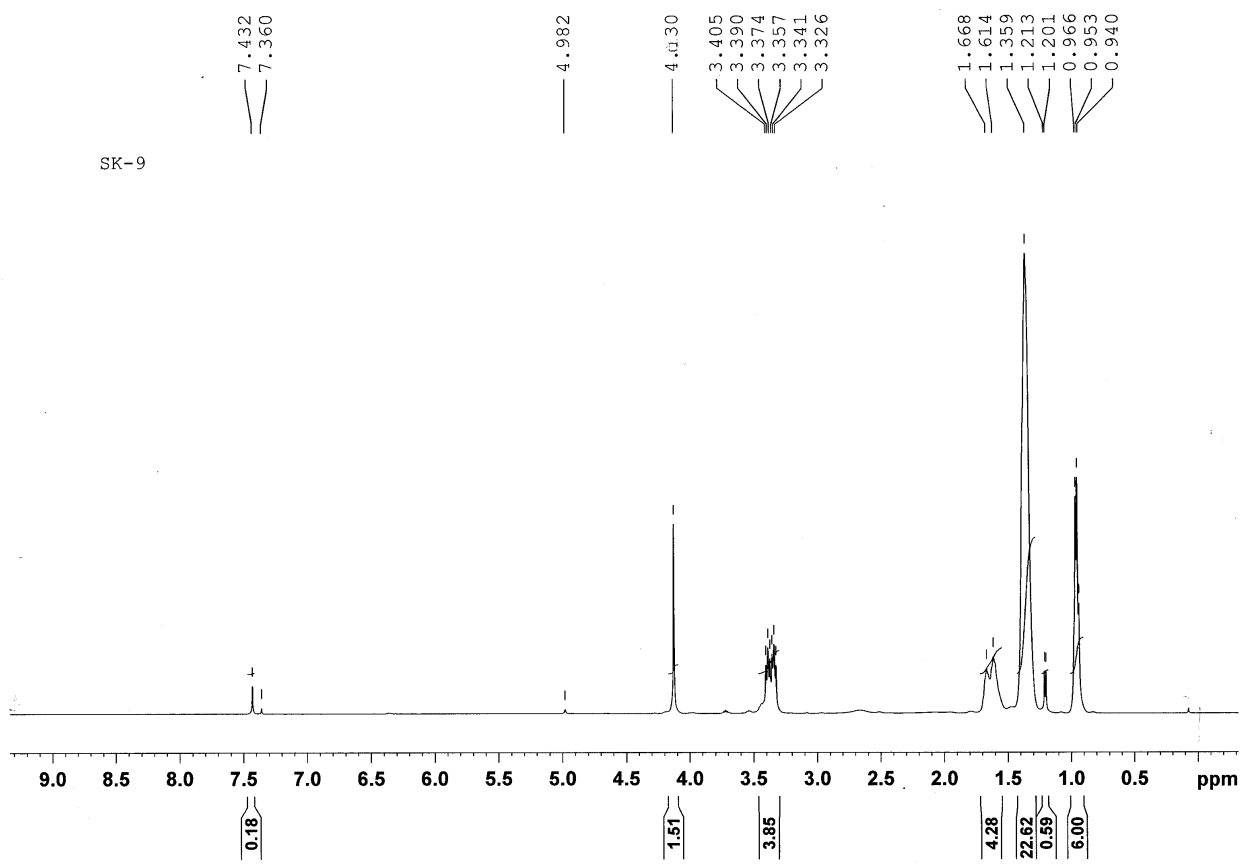
S5 : IR spectrum of La(NO<sub>3</sub>)<sub>3</sub> + (iC<sub>3</sub>H<sub>7</sub>)<sub>2</sub>NCOCH<sub>2</sub>OH compound



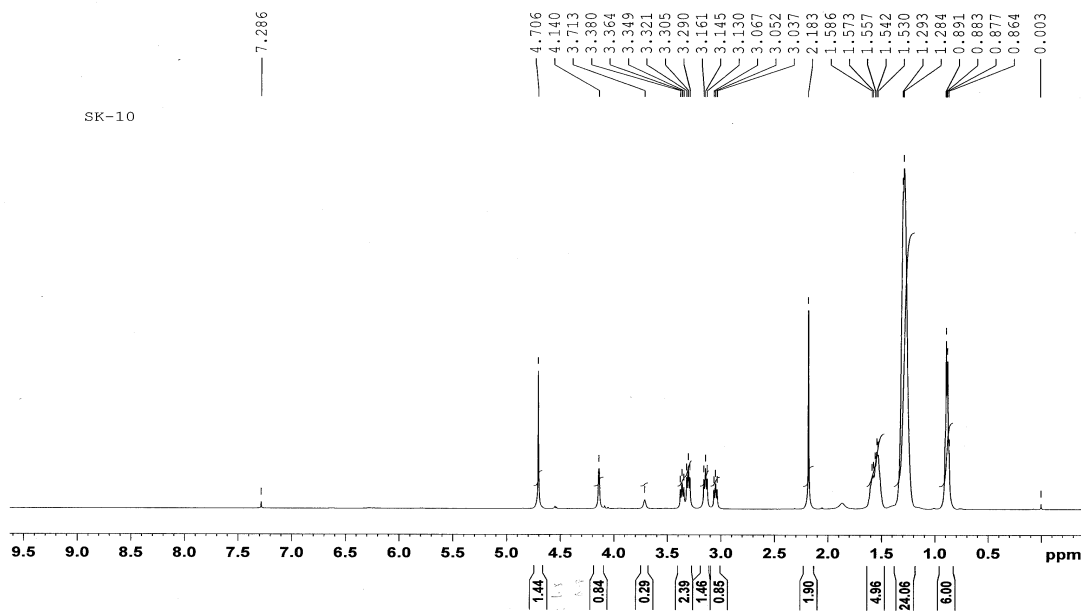
S6 : IR spectrum of Eu(NO<sub>3</sub>)<sub>3</sub> + (i-C<sub>3</sub>H<sub>7</sub>)<sub>2</sub>NCOCH<sub>2</sub>OH compound



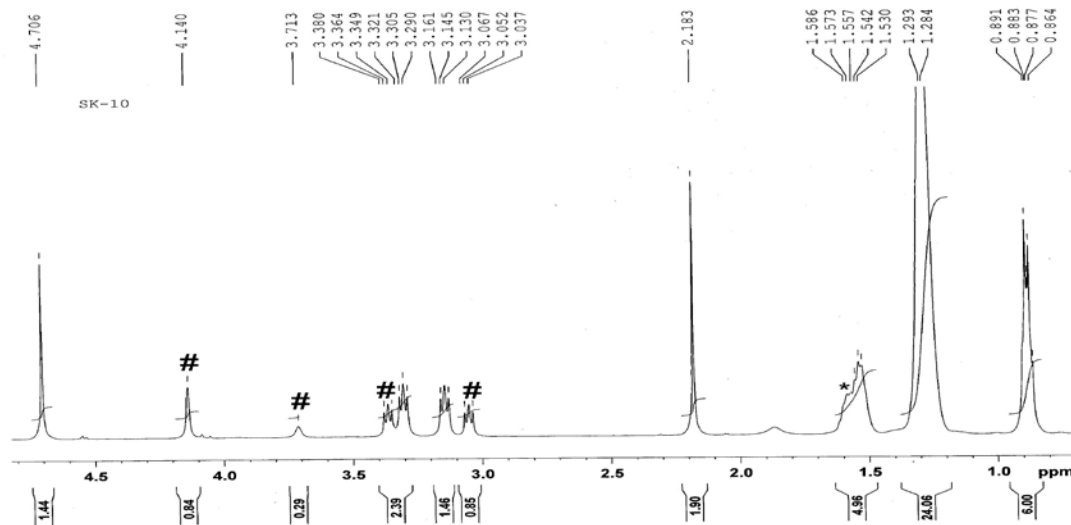
S7 : IR spectrum of Sm(NO<sub>3</sub>)<sub>3</sub> + (i-C<sub>3</sub>H<sub>7</sub>)<sub>2</sub>NCOCH<sub>2</sub>OH compound



S8 :  $^1\text{H}$  NMR spectrum of  $(\text{C}_8\text{H}_{17})_2\text{NCOCH}_2\text{Cl}$

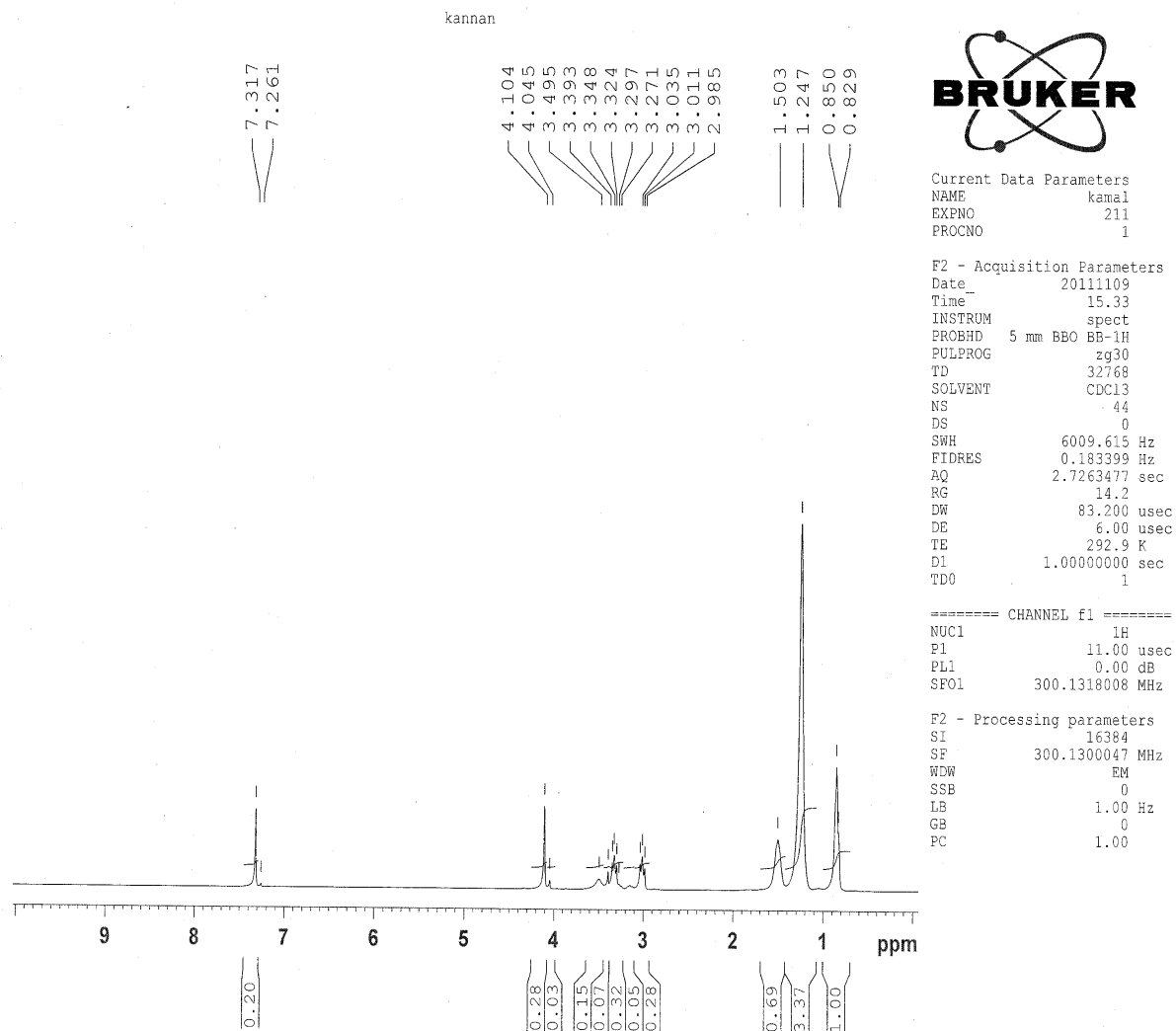


S9 :  $^1\text{H}$  NMR spectrum of intermediate compound  $(\text{C}_8\text{H}_{17})_2\text{NCOCH}_2\text{OCOCH}_3$

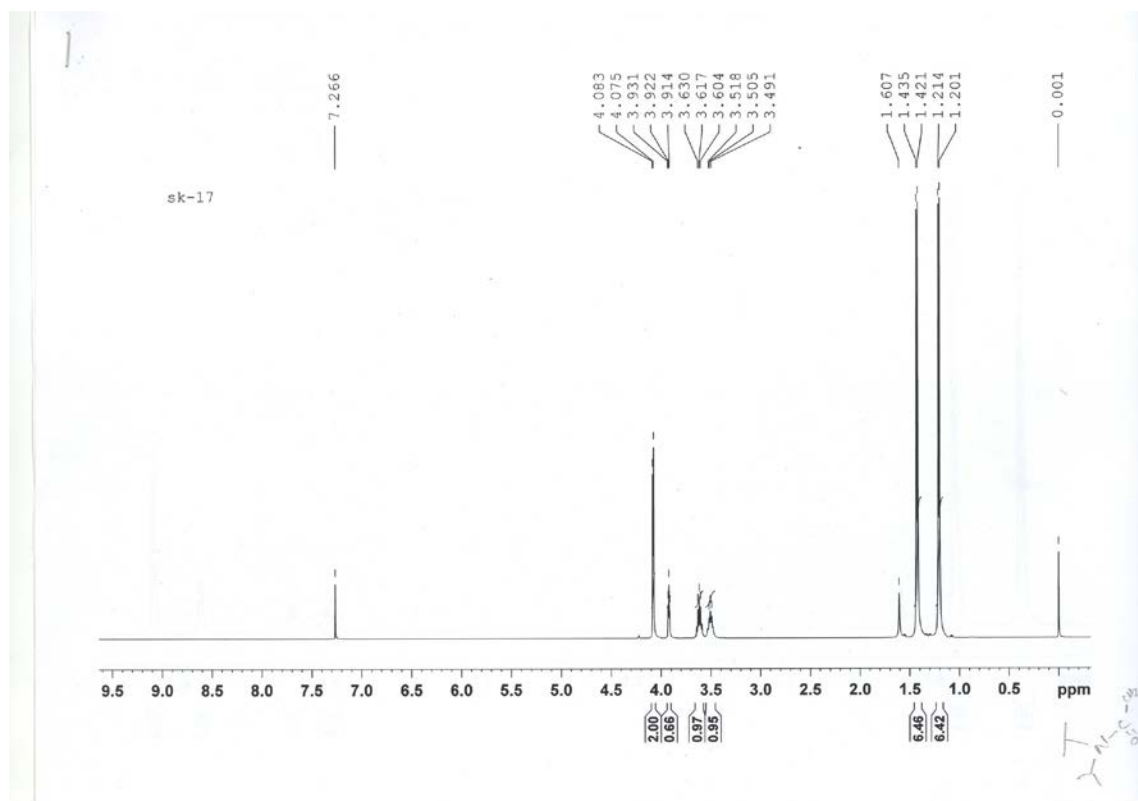


S10 :<sup>1</sup>H NMR of intermediate compound : $(C_8H_{17})_2NCOCH_2OCOCH_3$  (expanded )

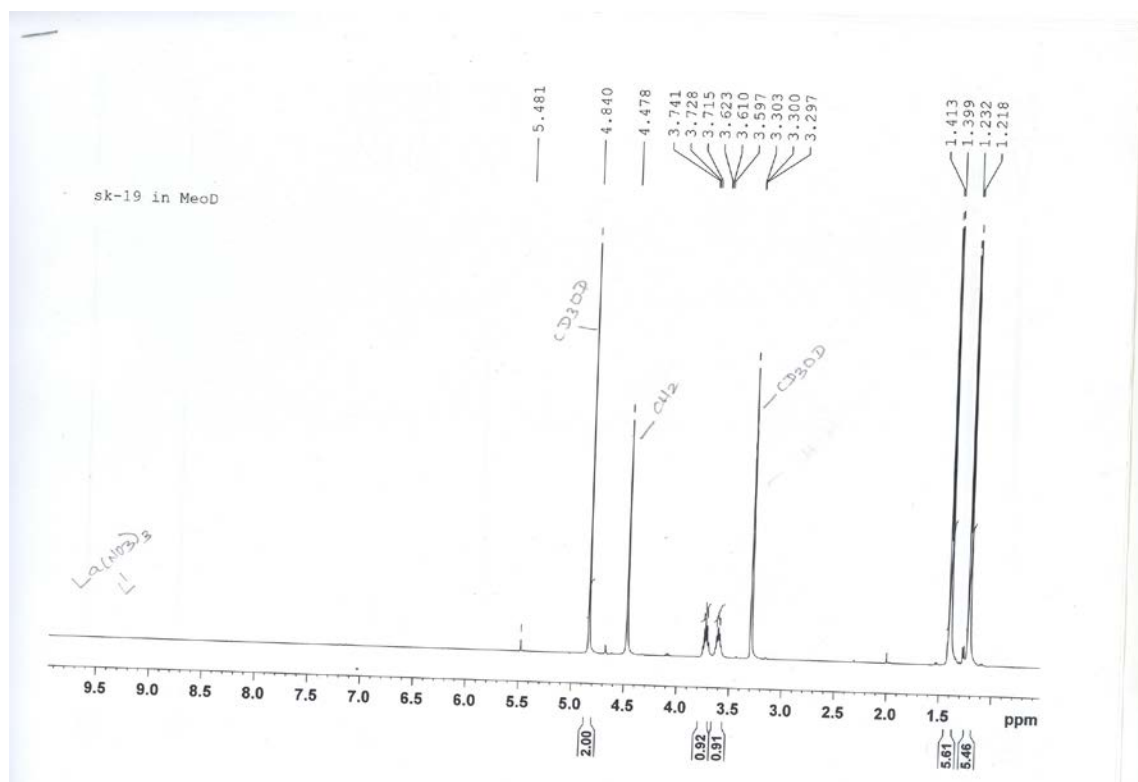
( # corresponds to the final product  $(C_8H_{17})_2NCOCH_2OH$  )



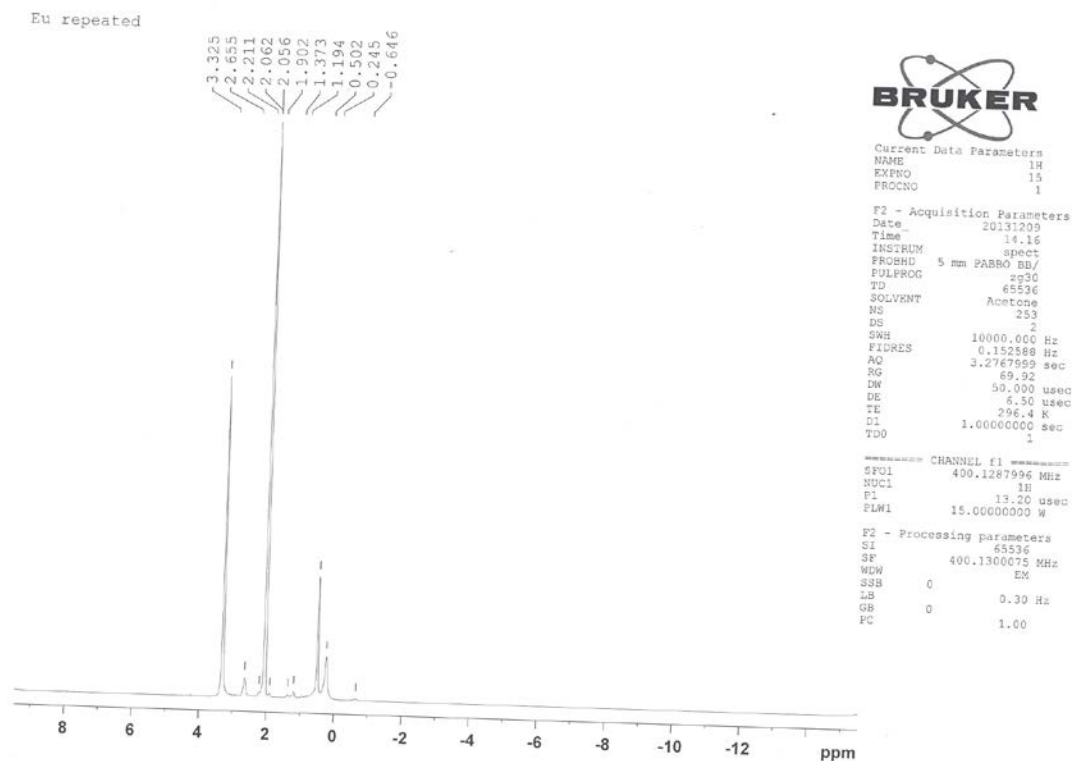
S11 :  $^1\text{H}$  NMR spectrum of :  $(\text{C}_8\text{H}_{17})_2\text{NCOCH}_2\text{OH}$



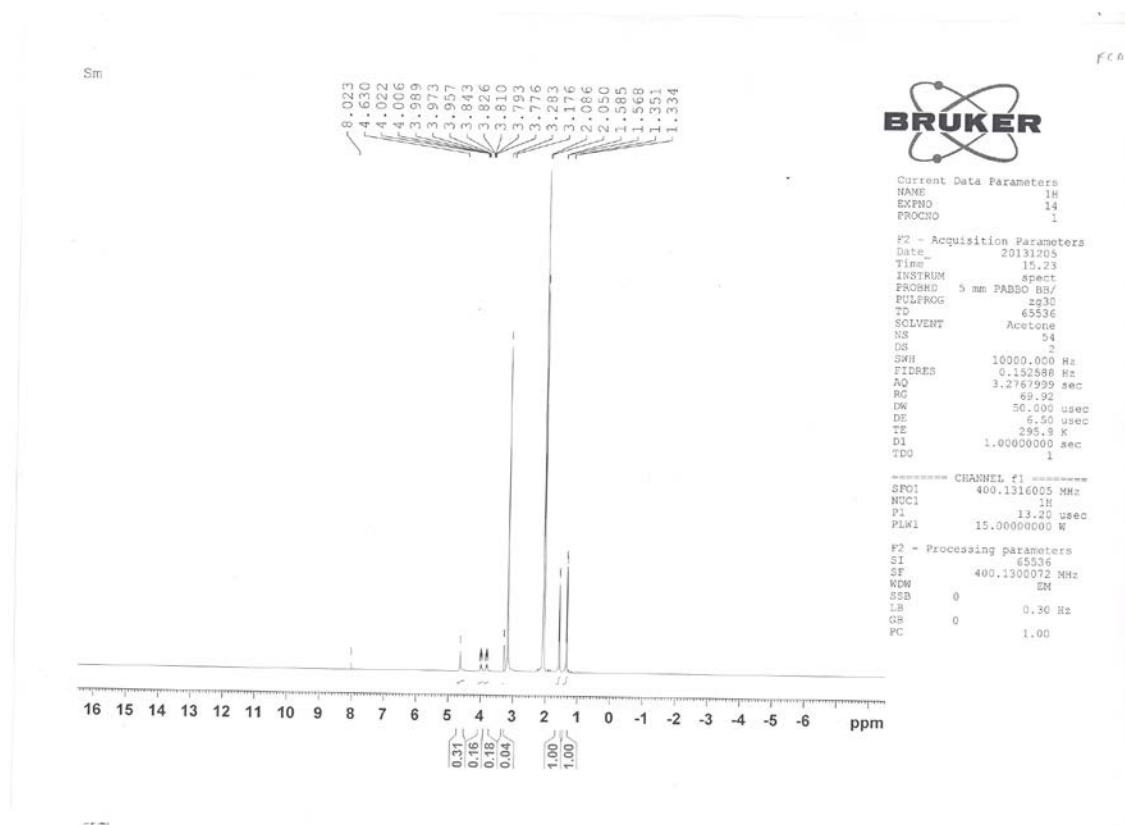
S12 :  $^1\text{H}$  NMR spectrum of :  $(\text{C}_3\text{H}_7)_2\text{NCOCH}_2\text{OH}$



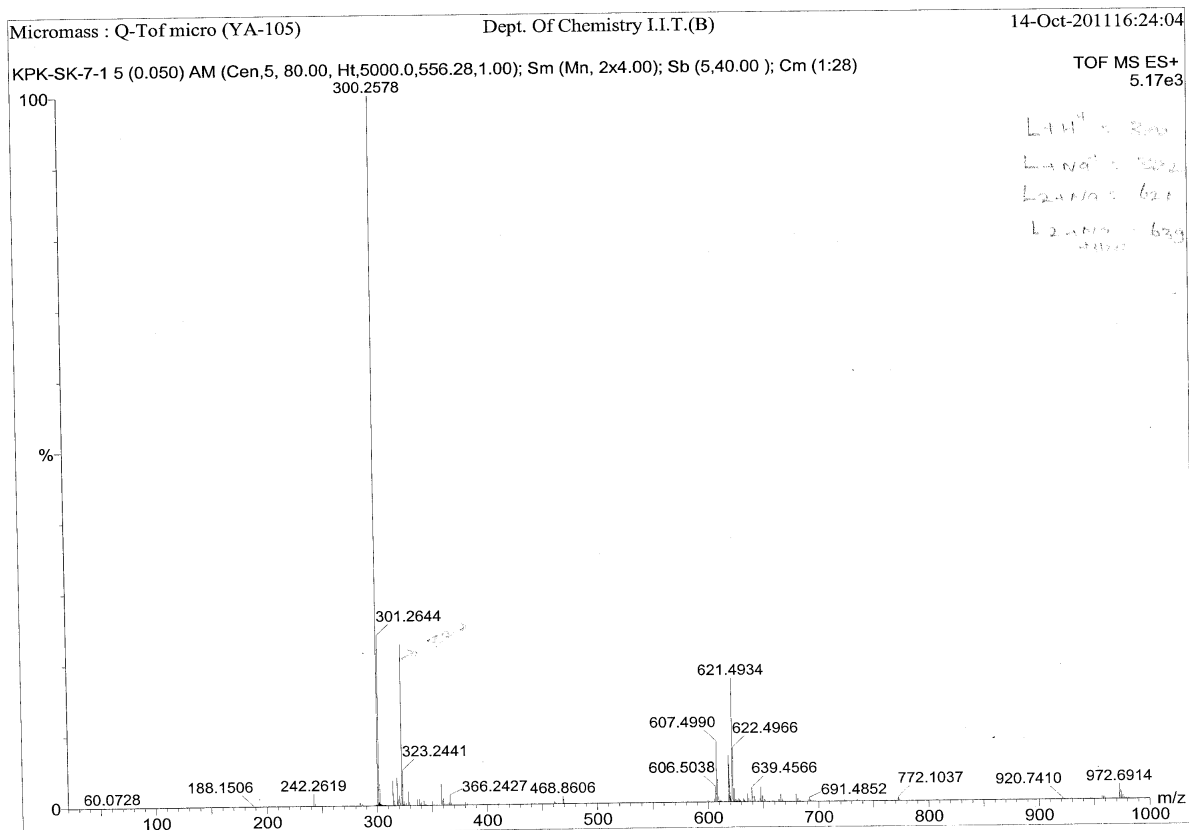
S13 : <sup>1</sup>H NMR spectrum of : La(NO<sub>3</sub>)<sub>3</sub> + (C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>NCOCH<sub>2</sub>OH compound in CD<sub>3</sub>OD



S14 :  $^1\text{H}$  NMR spectrum of :  $\text{Eu}(\text{NO}_3)_3 + (\text{C}_8\text{H}_{17})_2\text{NCOCH}_2\text{OH}$  compound in  $\text{CD}_3\text{COCD}_3$



S15 :  $^1\text{H}$  NMR spectrum of :  $\text{Sm}(\text{NO}_3)_3 + (\text{C}_8\text{H}_{17})_2\text{NCOCH}_2\text{OH}$  compound in  $\text{CD}_3\text{COCD}_3$



S8 : ES-MS spectrum of final compound :  $(C_8H_{17})_2NCOCH_2OH$  in  $CH_2Cl_2$

The starting chloro compound is un-detectable