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Synthesis and anion exchange chemistry of new intercalation hosts containing lanthanide cations, $\text{Ln}_2(\text{OH})_5(\text{NO}_3) \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y}, \text{Gd-Lu}$)

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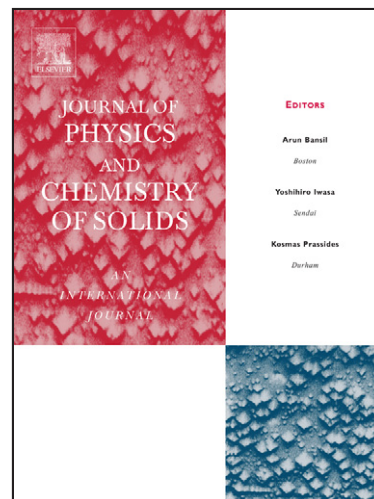
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**Synthesis and anion exchange chemistry of new intercalation hosts
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Abstract

A new class of lanthanide containing layered hydroxides, with the composition $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y, Gd - Lu}$), has been synthesised via a hydrothermal route. These materials combine the properties of the lanthanides with the flexibility of intercalation hosts and comprise cationic layers containing a single lanthanide cation with charge neutrality maintained by the nitrate anions between the layers. Powder X-ray diffraction and TEM studies indicate that they have a monoclinic structure. Additionally, powder XRD data show that $\text{Gd}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ is significantly less crystalline than the other phases and it has not yet proved possible to synthesise this phase with any of the larger lanthanides. The capacity of the $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y, Gd - Lu}$) materials for anion exchange chemistry has been demonstrated by their facile reactions with a range of inorganic and organic anions including sulfate, carbonate, aliphatic and aromatic dicarboxylates and disulfonates forming materials with the general composition $\text{Ln}_2(\text{OH})_5\text{A}_{0.5} \cdot x\text{H}_2\text{O}$.

Keywords: A. inorganic compounds, B. chemical synthesis, C. X-ray diffraction

1. Introduction

Materials capable of undergoing anion exchange reactions, in particular layered hydroxides, have attracted significant interest as hosts for intercalation reactions. These are unusual materials amongst intercalation hosts, consisting of positively charged layers and charge balancing anions in the interlayer regions which can be readily replaced by other species through an anion exchange process.[1] Research into this area is dominated by the layered double hydroxides (LDHs, $[M^{2+}_{1-x}M^{3+}_x(OH)_2]^{x+}X^{n-}_{x/n}.mH_2O$) which comprise a divalent (e.g. $M^{2+} = Mg, Zn, Co, Ni$) and a trivalent cation (e.g. $M^{3+} = Al, Cr, Fe, Ga$) within the layers.[2-4] Intercalation reactions provide routes to both modify and control the electrical, optical and magnetic properties of the material with only a minimal change in the structure and layered double hydroxides themselves have found numerous applications in fields as diverse as catalysis, medicine and molecular recognition.[5] Whilst it has proved possible to intercalate lanthanide containing guests into LDHs[6] there have been no reports of the cations being incorporated into the layers of these intercalation host lattices. The larger lanthanides are known to form layered hydroxynitrates with the general formula $Ln(OH)_2NO_3 \cdot xH_2O$ ($Ln = La, Pr, Nd, Sm$ and Gd ; $x = 0, 1$)[7-10] and anion exchange has been demonstrated for reactions between $La(OH)_2NO_3 \cdot H_2O$ and acetate, benzoate and terephthalate salts.[11]

In this paper we report the synthesis and anion exchange chemistry of a new family of hydroxynitrates with the general formula $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y}, \text{Gd} - \text{Lu}$). These materials are the first anion exchange host lattices to incorporate the smaller lanthanide cations within the layers and have the potential to combine the properties of the lanthanide elements, for example optical and catalytic, with the flexibility of lamellar intercalation hosts.

2. Experimental Details

2.1 Synthesis of $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y}, \text{Gd} - \text{Lu}$)

The $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y}, \text{Gd} - \text{Lu}$) phases are synthesised via a hydrothermal route. In a typical reaction 7.5 mL of a 0.44 M $\text{Ln}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$ aqueous solution is added to 2.5 mL of an aqueous solution containing 2.10 M NaOH and 1.44 M NaNO_3 and treated hydrothermally at 150 °C (125 °C for Y) for 48 hours. The resulting solid was filtered, washed and allowed to dry in air.

2.2 Anion exchange reactions

Anion exchange reactions were performed between the $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y}, \text{Gd} - \text{Lu}$) phases and an aqueous solution containing a threefold molar excess of a range of (di)sodium salts of organic anions at room temperature. The anions investigated were maleate, fumarate, phthalate, terephthalate, oxalate, malonate, succinate, glutarate,

suberate and decylsulfonate. All the chemicals were supplied by Aldrich and used without further purification.

3 Results and discussion

A new family of hydroxynitrates, having the composition $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y}$, $\text{Gd} - \text{Lu}$), has been synthesised via a hydrothermal route. As these materials are isostructural and display similar behaviour in anion exchange reactions they will be exemplified here by the phase with $\text{Ln} = \text{Y}$. In each case the materials were synthesised phase pure and the composition, $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ ($x \approx 1$), was confirmed by elemental analysis and thermogravimetric analysis as summarised in Table 1. The samples comprise microcrystalline powders with an approximately hexagonal plate-like morphology as shown by the SEM image in Fig. 1 for $\text{Y}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$.

The TGA data (Fig. 2) display three mass losses, typical of those seen for LDHs. For $\text{Y}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$, 6.86 % of the mass is lost below 150 °C corresponding to the loss of co-intercalated water with further mass losses of 9.79 % by 350 °C and 17.28 % by 650 °C corresponding to the decomposition of layers and the nitrate anion leaving Y_2O_3 . [12] No further mass loss is observed above 650 °C.

The phase purity of these materials was confirmed by powder X-ray diffraction which showed strong (00 l) reflections characteristic of a layered material. The powder XRD pattern for $\text{Y}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ is shown in Figure 3. In addition to the strong basal

reflections a large number of non-basal reflections are observed indicating that there is significant order within the layers in contrast to many of the LDHs. The data can be fully indexed on a monoclinic unit cell which refined to $a = 7.0808(4) \text{ \AA}$, $b = 12.7004(5) \text{ \AA}$, $c = 18.6348(4) \text{ \AA}$ and $\beta = 92.509(5)^\circ$ in a Le Bail extraction. TEM has confirmed the a and b parameters and revealed that there are no systematic absences in the ab plane (see inset to Fig. 3). Unfortunately, due to the plate-like morphology of the crystals (see Fig. 1), and despite extensive screening, it was not possible observe diffraction from along the c -axis. Whilst attempts to fully determine the crystal structure of the $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ materials are ongoing, the lattice parameters suggest that they have a layer structure similar the recently reported orthorhombic $[\text{Ln}_4(\text{OH})_{10}(\text{H}_2\text{O})_4]\text{A}$ ($\text{Ln} = \text{Y}, \text{Dy}, \text{Ho}, \text{Yb}$; $\text{A} = 2,6\text{-naphthalenedisulfonate}, 2,6\text{-anthraquinonedisulfonate}$) phases which were prepared directly via a hydrothermal route.[13] Single crystal studies revealed them to consist of cationic layers in which the lanthanide cations are 8 and 9 coordinate to hydroxide and additionally coordinated to water with charge neutrality maintained by the organic anions in the interlayer space. In the current study, however, despite extensive optimisation of the reaction conditions through control of the temperature, reagent concentration and time, it has not proved possible to obtain single crystals of $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ suitable for X-ray diffraction or to index the powder X-ray diffraction data on orthorhombic unit

cells related to those reported by Gándara et al. Powder XRD data show that $\text{Gd}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ is significantly less crystalline than the other materials and it has not proved possible to synthesise this phase with any of the larger lanthanides.

The data discussed above are consistent with the formation of a new family of layered lanthanide hydroxynitrates in which the positively charged layers contain a single lanthanide cation with charge neutrality maintained by the nitrate anion. One characteristic property of layered hydroxide materials, such as LDHs, is their ability to undergo facile anion exchange reactions with a wide variety of inorganic and organic anions. Experiments with the new $\text{Ln}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$ materials have demonstrated that the nitrate ions can be completely replaced by reaction with a range of inorganic ions and organic carboxylates and sulfonates at room temperature as evidenced by the absence of reflections characteristic of the host phase in the powder X-ray diffraction patterns and of nitrogen in the elemental analysis. Successful anion exchange reactions, forming $\text{Ln}_2(\text{OH})_5\text{A}_{0.5} \cdot x\text{H}_2\text{O}$ ($x \approx 1.5$), have been observed with, for example, sulfate, carbonate and the disodium salts of aliphatic dicarboxylic acids, fumaric, maleic, phthalic and terephthalic acids and the sodium salt of decylsulfonic acid and all the host materials giving rise to interlayer separations of up to 26 Å clearly demonstrating the flexibility of these materials for intercalation chemistry. Typical diffraction patterns of the anion

exchange products are shown in Fig. 4 and characterising data given in the ESI. TGA traces of the organic intercalation compounds are comparable to those, for example, of the LDHs showing the loss of co-intercalated water below 150 °C followed by the dehydroxylation of the layers and decomposition of the guest anions. From an analysis of the interlayer separations of the aliphatic dicarboxylate intercalates it is possible to deduce an approximate orientation of the anions between the lanthanide hydroxide layers. The plot of interlayer separation against the number of CH₂ groups in the chain is shown in Figure 5 from which a gradient of 1.17 Å/C atom is obtained. By comparing this with the intrachain C-C distance in hydrocarbons of approximately 1.27 Å it is possible to calculate a guest orientation of approximately 67 ° with respect to the layers which is comparable to that seen in similar LDH systems.[14, 15]

4. Conclusions

We report in this paper the first synthesis of a new class of layered hydroxides with layers containing the smaller lanthanide cations with the general formula, Ln₂(OH)₅NO₃.xH₂O (Ln = Y, Gd - Lu). Thorough characterisation has confirmed the composition and the layered nature of these materials has been shown by the complete replacement of the nitrate anions between the layers by organic anions in facile exchange reactions. Full details of all the Ln₂(OH)₅NO₃.xH₂O (Ln = Y, Gd - Lu) materials, their structure and

anion exchange chemistry will be reported elsewhere along with details of solid solution formation between the nitrate hosts and doping of the larger lanthanides into these materials.

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Figure captions

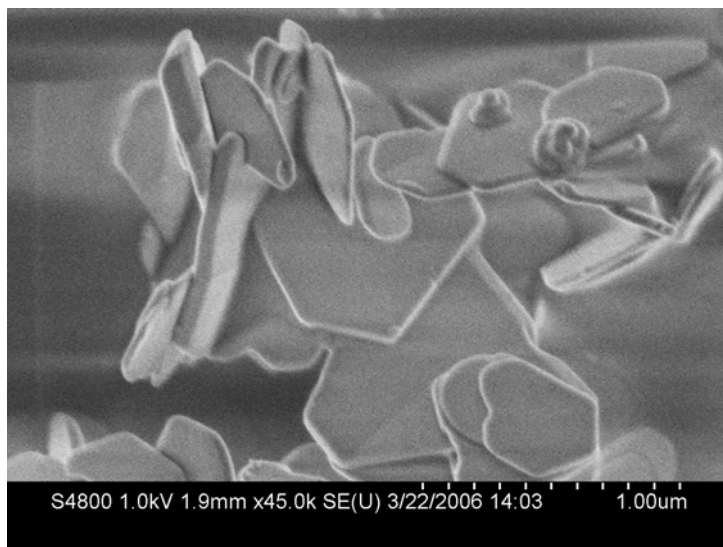
Figure 1. SEM image of $Y_2(OH)_5NO_3 \cdot xH_2O$.

Figure 2. TGA trace for $Y_2(OH)_5NO_3 \cdot xH_2O$ showing mass losses of 6.86 % below 150 °C and further mass losses of 9.79 % by 350 °C and 17.28 % by 600 °C.

Figure 3. Powder X-ray diffraction pattern of $Y_2(OH)_5NO_3 \cdot xH_2O$. Inset: SAED pattern for $Y_2(OH)_5NO_3 \cdot xH_2O$.

Figure 4. Powder XRD diffraction patterns of (a) $Y_2(OH)_5NO_3 \cdot xH_2O$ and the anion exchange intercalation compounds (b) $Y_2(OH)_5(C_2O_4)_{0.5} \cdot xH_2O$ (c) $Y_2(OH)_5(o-C_6H_4(CO_2)_2)_{0.5} \cdot xH_2O$ and (d) $Y_2(OH)_5(C_8H_{12}O_4)_{0.5} \cdot xH_2O$.

Figure 5. Plot of interlayer separation against number of $-CH_2-$ groups in the aliphatic chain of some dicarboxylate intercalates of $Y_2(OH)_5NO_3 \cdot xH_2O$.

Figure 1

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Figure 2

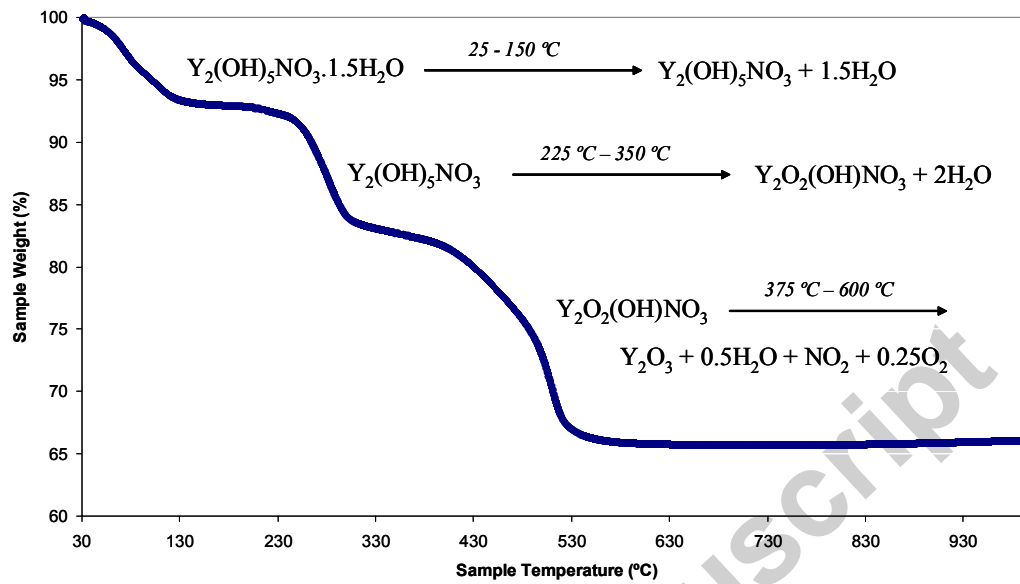


Figure 3

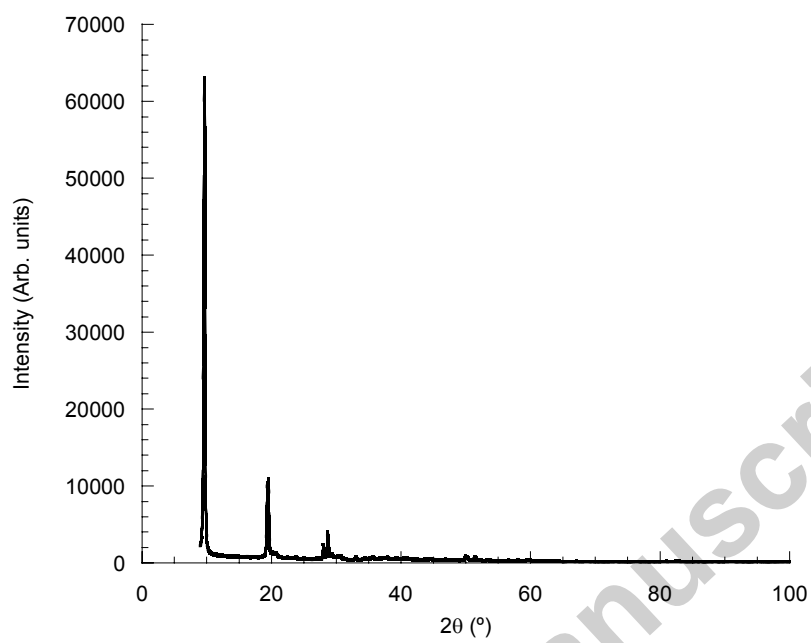


Figure 3 INSET

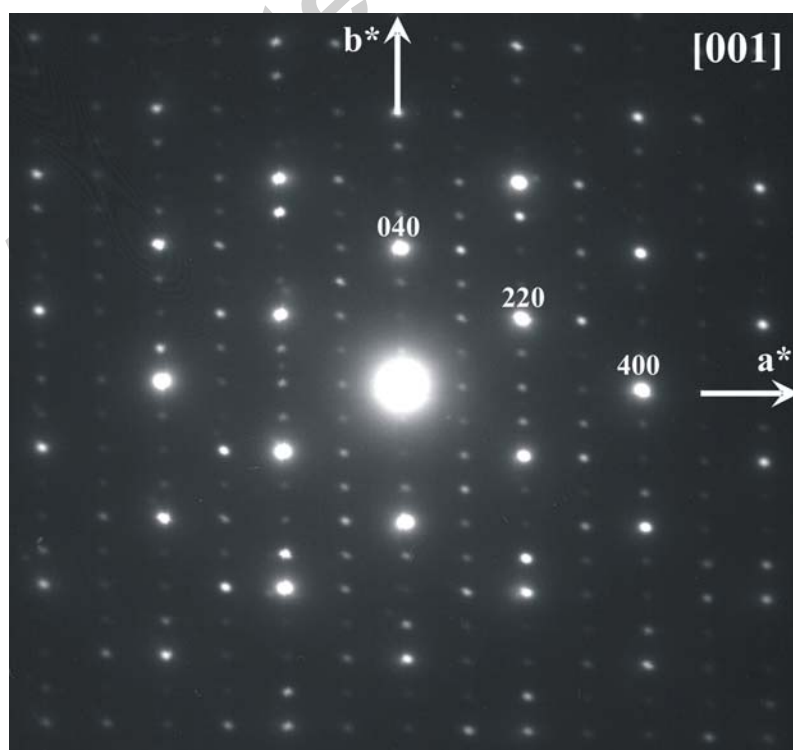


Figure 4

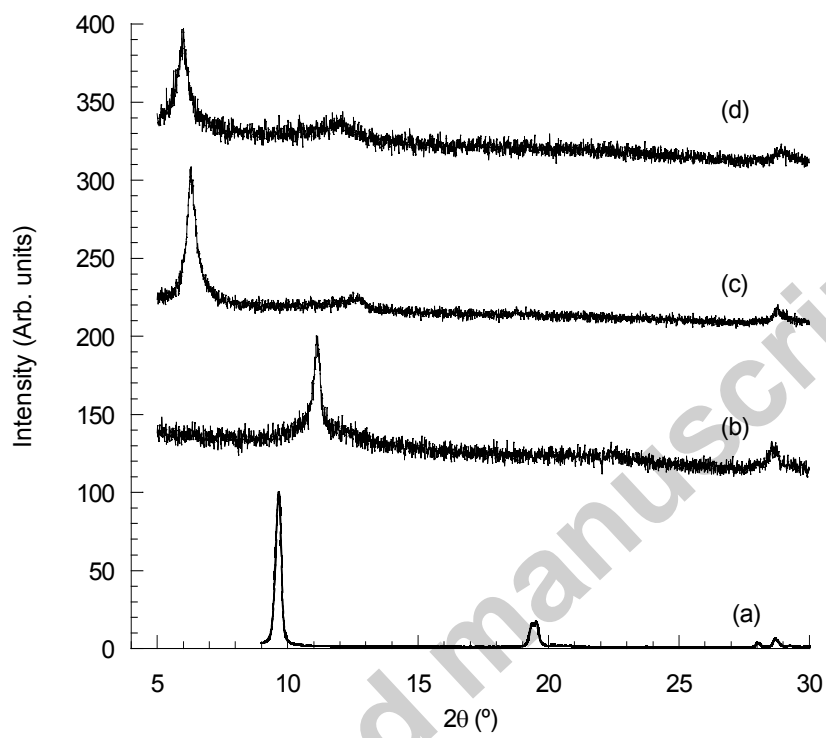
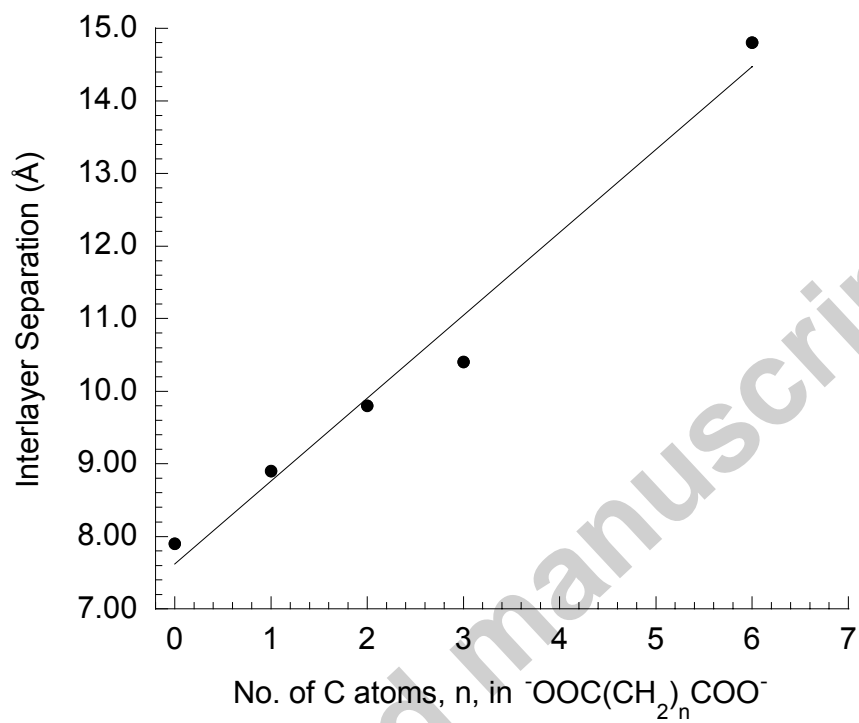


Figure 5



Tables

Table 1. Characterising data for the new layered hydroxide, $Y_2(OH)_5NO_3 \cdot xH_2O$ and the anion exchange derivatives $Y_2(OH)_5A_{0.5} \cdot xH_2O$.

A	Composition	Interlayer Separation (Å)	Elemental Analysis	
			Observed (%)	Calculated (%)
Nitrate	$Y_2(OH)_5NO_3 \cdot H_2O$	9.18	Y (49.89), N (3.91), H (2.25)	Y (50.53), N (4.09), H (2.06)
Maleate	$Y_2(OH)_5(cis-C_4H_2O_4)_{0.5} \cdot 1.5H_2O$	10.2	Y (51.00) C (6.84) H (2.57)	Y (51.25) C (6.92) H (2.62)
Fumarate	$Y_2(OH)_5(trans-C_4H_2O_4)_{0.5} \cdot 1.5H_2O$	9.6	Y (50.88) C (6.69) H (2.59)	Y (51.26) C (6.92) H (2.62)
Phthalate	$Y_2(OH)_5(o-C_8H_4O_4)_{0.5} \cdot 1.5H_2O$	14.0	Y (47.31) C (12.94) H (2.92)	Y (47.68)C (12.88) H (2.97)
Terephthalate	$Y_2(OH)_5(p-C_8H_4O_4)_{0.5} \cdot 1.5H_2O$	12.8	Y (47.29) C (12.78) H (2.93)	Y (47.68)C (12.88) H (2.97)
Oxalate	$Y_2(OH)_5(C_2O_4)_{0.5} \cdot H_2O$	7.9	Y (50.66) C (3.93) H (2.14)	Y (53.26)C (3.690) H (2.17)
Succinate	$Y_2(OH)_5(C_4H_4O_4)_{0.5} \cdot 1.5H_2O$	9.8	C (6.91) H (2.89)	C (6.90) H (2.88)
Glutarate	$Y_2(OH)_5(C_5H_6O_4)_{0.5} \cdot 1.5H_2O$	10.4	C (8.05) H (2.88)	C (8.46) H (3.12)
Suberate	$Y_2(OH)_5(C_8H_{12}O_4)_{0.5} \cdot H_2O$	14.8	C (13.96) H (3.48)	C (13.09) H (3.57)
Decylsulfonate	$Y_2(OH)_5(C_{10}H_{21}SO_4) \cdot H_2O$	23.8	C (21.70) H (5.39)	C (22.78) H (5.54)

Electronic Supplementary Information for ‘Synthesis and anion exchange chemistry of new intercalation hosts containing lanthanide cations, $\text{Ln}_2(\text{OH})_5(\text{NO}_3) \cdot x\text{H}_2\text{O}$ ($\text{Ln} = \text{Y, Gd-Lu}$)’ Laura J. McIntyre, Lauren K. Jackson and Andrew M. Fogg

$\text{Y}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$

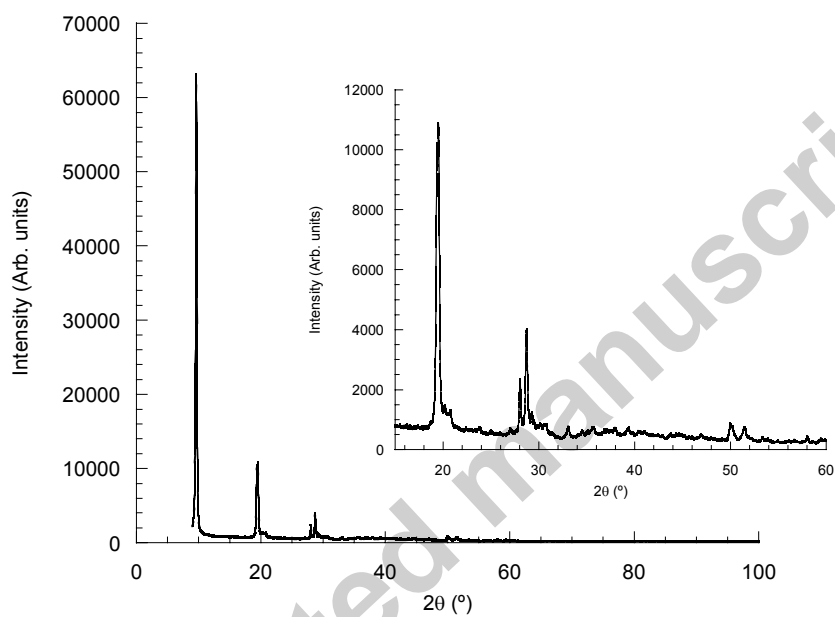


Fig. S1 Powder X-ray diffraction pattern of $\text{Y}_2(\text{OH})_5\text{NO}_3 \cdot x\text{H}_2\text{O}$.

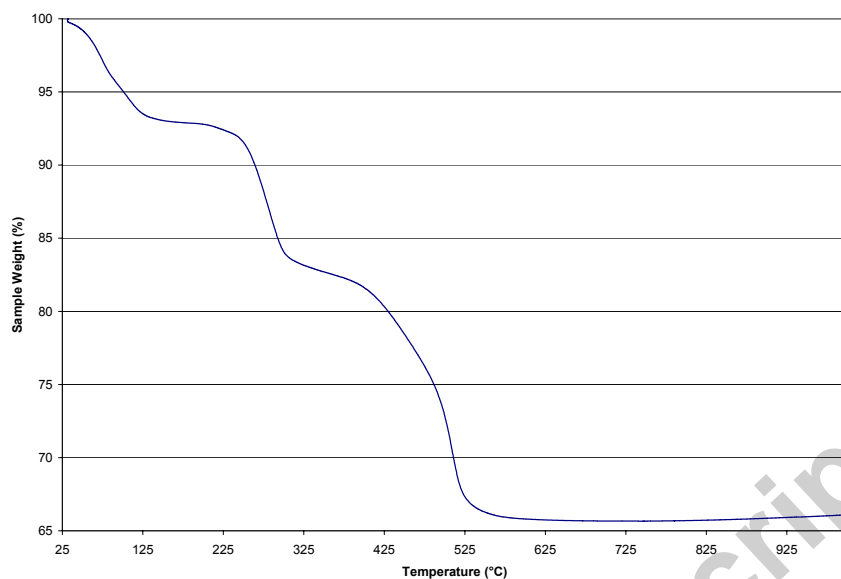


Fig. S2 TGA trace for $Y_2(OH)_5NO_3 \cdot xH_2O$ showing mass losses of 6.86 % below 150 °C and further mass losses of 9.79 % by 350 °C and 17.28 % by 600 °C.

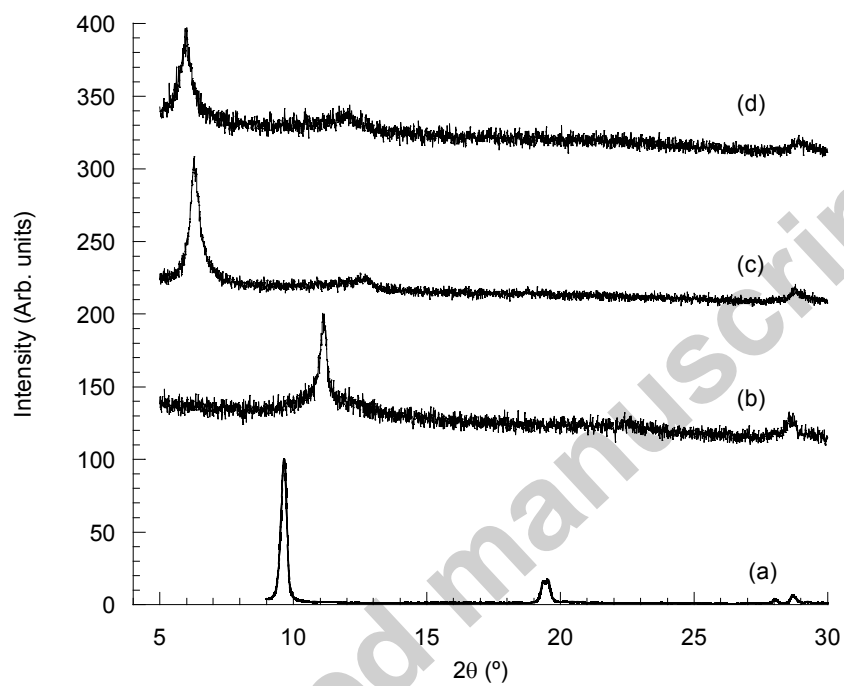
Anion Exchange Derivatives of $Y_2(OH)_5NO_3 \cdot xH_2O$ 

Fig. S3 Powder X-ray diffraction patterns of (a) $Y_2(OH)_5NO_3 \cdot xH_2O$ and the anion exchange derivatives with (b) oxalate, (c) phthalate and (d) suberate.

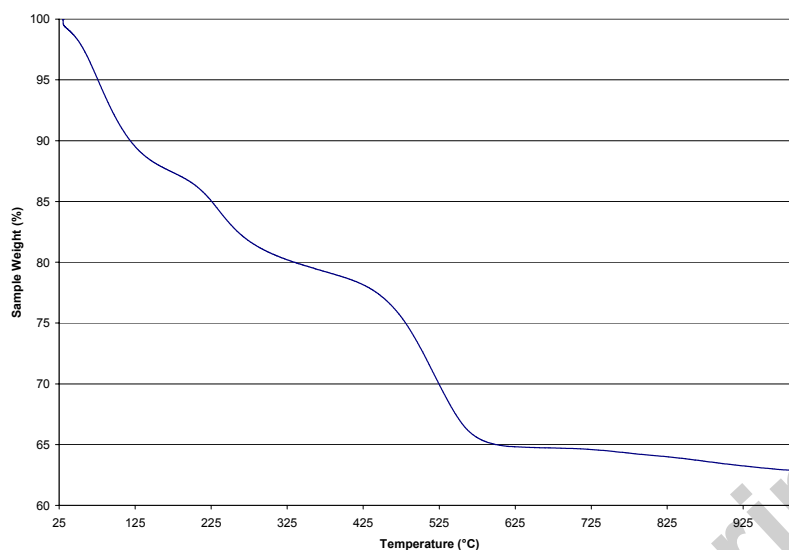


Fig. S4 TGA trace for $Y_2(OH)_5(o-C_8H_4O_4)_{0.5}.xH_2O$ showing a total mass loss of 37.14 % by 1000 °C.

Table S1 Characterising data for the organic anion exchange derivatives of $Y_2(OH)_5NO_3.xH_2O$.

A	Composition	Interlayer Separation (Å)	Elemental Analysis	
			Observed (%)	Calculated (%)
Maleate	$Y_2(OH)_5(cis-C_4H_2O_4)_{0.5}.1.5H_2O$	10.2	Y (51.00)	Y (51.25)
			C (6.84)	C (6.92)
			H (2.57)	H (2.62)
Fumarate	$Y_2(OH)_5(trans-C_4H_2O_4)_{0.5}.1.5H_2O$	9.6	Y (50.88)	Y (51.26)
			C (6.69)	C (6.92)
			H (2.59)	H (2.62)
Phthalate	$Y_2(OH)_5(o-C_8H_4O_4)_{0.5}.1.5H_2O$	14.0	Y (47.31)	Y (47.68)
			C (12.94)	C (12.88)

			H (2.92)	H (2.97)
Terephthalate	$Y_2(OH)_5(p-C_8H_4O_4)_{0.5} \cdot 1.5H_2O$	12.8	Y (47.29)	Y (47.68)
			C (12.78)	C (12.88)
			H (2.93)	H (2.97)
Oxalate	$Y_2(OH)_5(C_2O_4)_{0.5} \cdot H_2O$	7.9	Y (50.66)	Y (53.26)
			C (3.93)	C (3.690)
			H (2.14)	H (2.17)
Succinate	$Y_2(OH)_5(C_4H_4O_4)_{0.5} \cdot 1.5H_2O$	9.8	C (6.91)	C (6.90)
			H (2.89)	H (2.88)
Glutarate	$Y_2(OH)_5(C_5H_6O_4)_{0.5} \cdot 1.5H_2O$	10.4	C (8.05)	C (8.46)
			H (2.88)	H (3.12)
Suberate	$Y_2(OH)_5(C_8H_{12}O_4)_{0.5} \cdot H_2O$	14.8	C (13.96)	C (13.09)
			H (3.48)	H (3.57)
Decylsulfonate	$Y_2(OH)_5(C_{10}H_{21}SO_4) \cdot H_2O$	23.8	C (21.70)	C (22.78)
			H (5.39)	H (5.54)
