Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Solid-State NMR Characterization of Structure and Thermal Stability of Hybrid Organic-Inorganic Compounds based on $HLaNb_2O_7$ Dion-Jacobson Layered Perovskite

Supporting Information

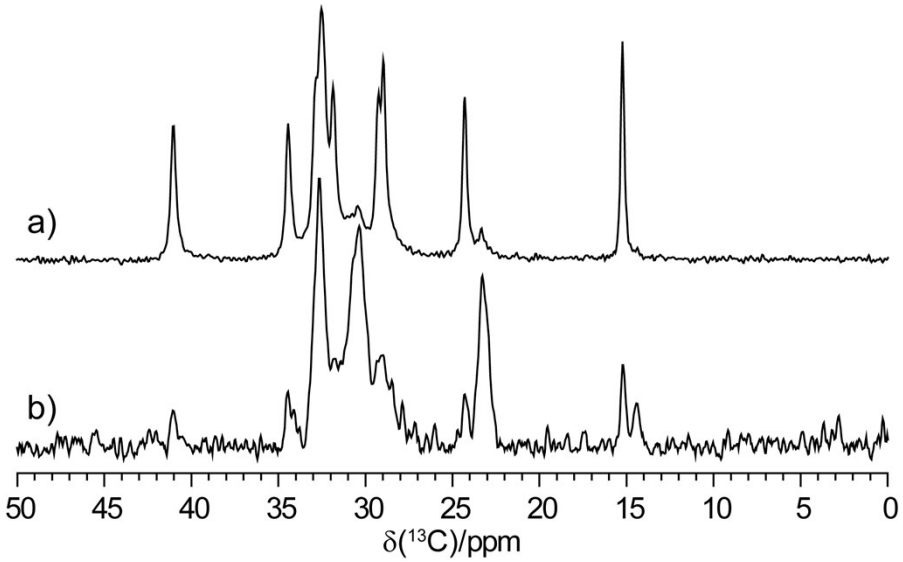

Fig. S1 Comparison between the $^{13}C(^{1}H)$ CP-MAS spectrum recorded with a contact time of 2.5 ms for the C10A sample (a), and the ^{13}C calculated projection obtained from the $^{13}C(^{1}H)$ HETCOR experiment (b), which was recorded with the same contact time and is reported in Figure 5.

	H^+ 10.2 ppm	H^+ (water) 8.7 ppm	H^+ (NH_3^+) 8.4 ppm	H^+ (defect) 7.0 ppm
HLN	72%	26%	-	2%
HLN_TT	98%	-	-	2%
0.05-C8A	22%	55%	17%	6%
0.05-C8A_TT110°C	79%	-	16%	5%
0.05-C8A_TT130°C	79%	-	15%	6%
0.05-C8A_TT150°C	71%		15%	14%
0.05-C8A_TT170°C	79%	-	12%	9%
0.05-C8A_TT175°C	81%		3%	16%
0.05-C8A_TT190°C	80%	-	-	20%
0.05-C8A_TT220°C	88%	-	-	12%
0.1-C8A	33%	31%	28%	8%
0.1-C8A_TT110°C	65%	-	28%	7%
0.1-C8A_TT130°C	69%	-	22%	9%
0.1-C8A_TT150°C	67%	-	18%	15%
0.1-C8A_TT170°C	74%	-	6%	20%
1-C8A	35%	-	59%	6%
1-C8A_TT110°C	52%	-	41%	7%
1-C8A_TT130°C	54%	-	30%	16%
1-C8A_TT150°C	44%	-	5%	51%
C8A	34%	-	41%	25%
C8A_TT110°C	43%	-	28%	29%
C8A_TT130°C	42%	-	16%	42%
C8A_TT150°C	42%	-	6%	52%
C8A_TT170°C	40%	-	5%	55%
C10A	9%	-	60%	31%
C10A_TT110°C	26%	-	27%	47%
C10A_TT130°C	18%	-	20%	62%
C10A_TT150°C	22%	-	-	78%
C3	25%	46%	-	29%
C3_TT110°C	51%	22%	-	27%
C3_TT150°C	54%	28%	-	18%
C3_TT170°C	48%	31%	-	19%
C3_TT190°C	49%	34%	.	17%

Table S1 Quantification of i) the amount of the single species of un-exchanged proton ions of the pristine HLN structure (±2%), and ii) the molar ratio H^+ /NH_3^+. Data were obtained through simulation of quantitative ^{1}H MAS NMR spectra.

as prepared	T_1/ms			
	0.05-C8A	**0.01-C8A**	**1-C8A**	**C8A**
^{1}H site				
CH_3-	352±8	331±8	342±7	354±6
-$(CH_2)_5$-	323±7	338±8	322±5	358±6
$CH_2CH_2NH^{3+}$	190±7	213±8	300±5	342±6
CH_2NH^{3+}	234±8	250±6	304±5	390±30
NH^{3+}	71±6	80±7	220±10	280±10
H_2O	88±6	131±8	200±10	240±10
H^+ HLN 7 ppm	72±6	101±7	204±8	240±9
H^+ HLN 8.2 ppm	122±8	148±8	249±9	299±8
H^+ HLN 8.7 ppm	54±5	62±6	-	-
H^+ HLN 10 ppm	170±10	201±8	235±7	280±20

110°C	T_1/ms			
	0.05-C8A	**0.01-C8A**	**1-C8A**	**C8A**
^{1}H site				
CH_3-	319±3	307±7	383±6	378±5
-$(CH_2)_5$-	338±5	312±6	384±5	376±4
$CH_2CH_2NH^{3+}$	320±10	259±6	294±3	296±6
CH_2NH^{3+}	318±4	279±2	346±4	353±4
NH^{3+}	189±6	140±10	218±4	206±6
H^+ HLN 7 ppm	228±8	218±4	334±3	393±4
H^+ HLN 8.2 ppm	248±3	218±1	306±3	328±8
H^+ HLN 10 ppm	558±2	469±7	429±10	370±11

Table S2 Quantification of ^{1}H T_1 relaxation times for the several proton species of the C8A samples (as prepared and after the thermal treatment at 110°C).

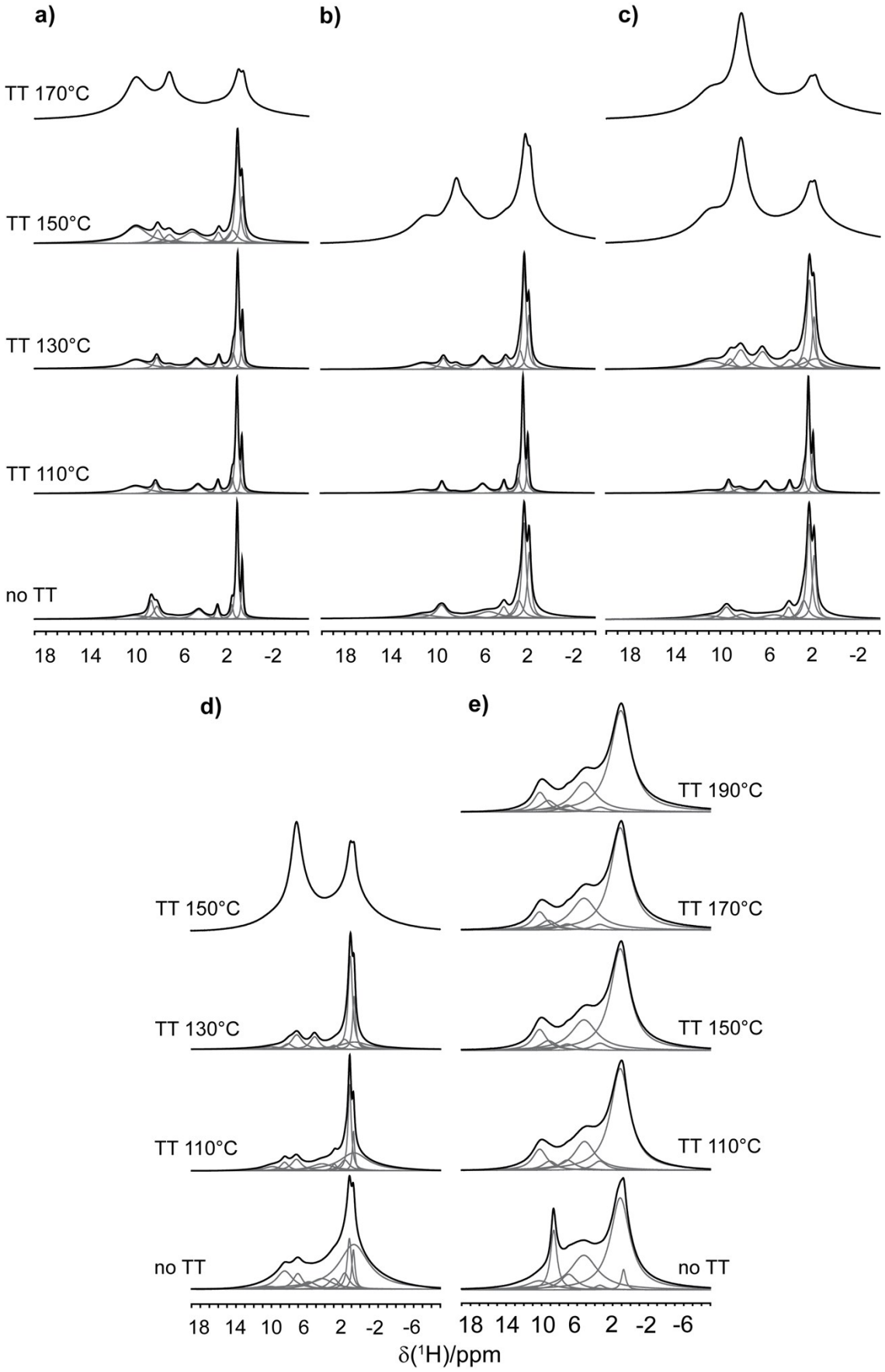

Fig. S2 ^{1}H MAS NMR spectra recorded after the different thermal treatments for 0.1-C8A (a), 1-C8A (b), C8A (c), C10A (d) and C3 (e). The gray lines are guides to follow the evolution of the different proton ions species.

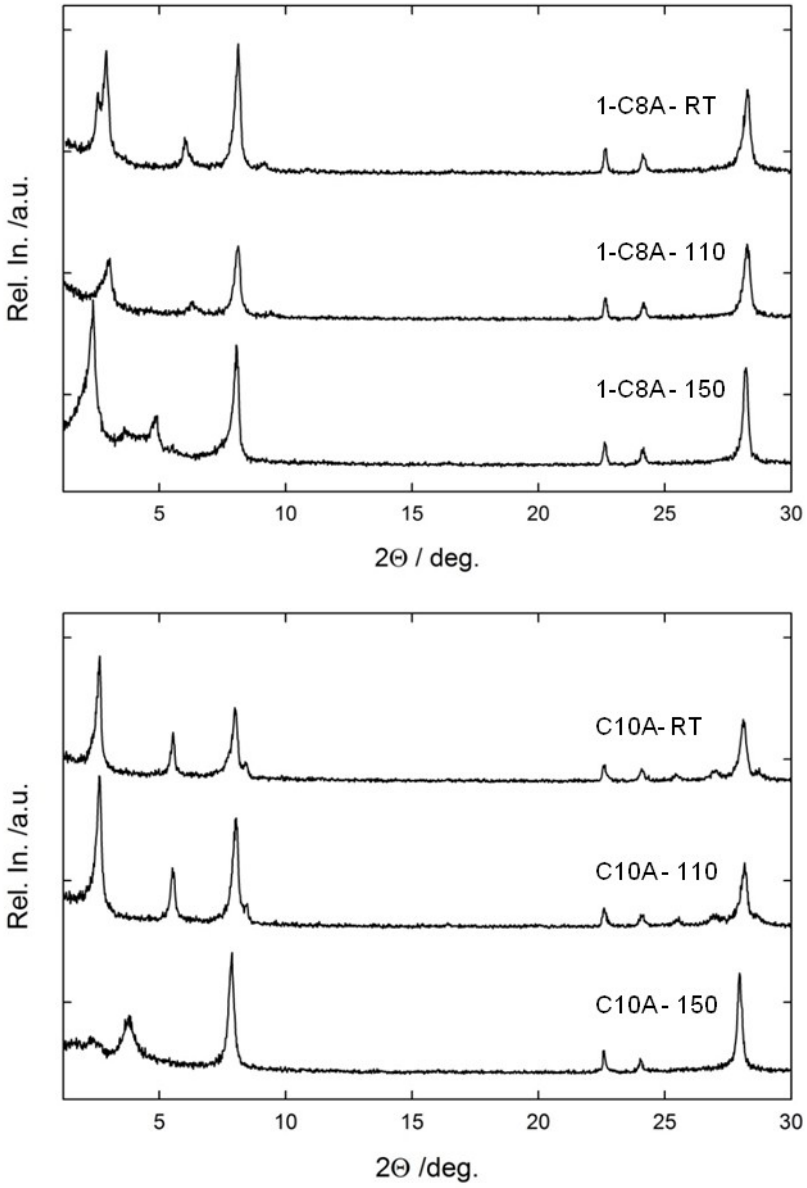

Fig. S3 X-ray diffraction patterns acquired for the octylammonium-HLN samples 1-C8A and C10A as-received, after the 110°C step and after the 150°C step.

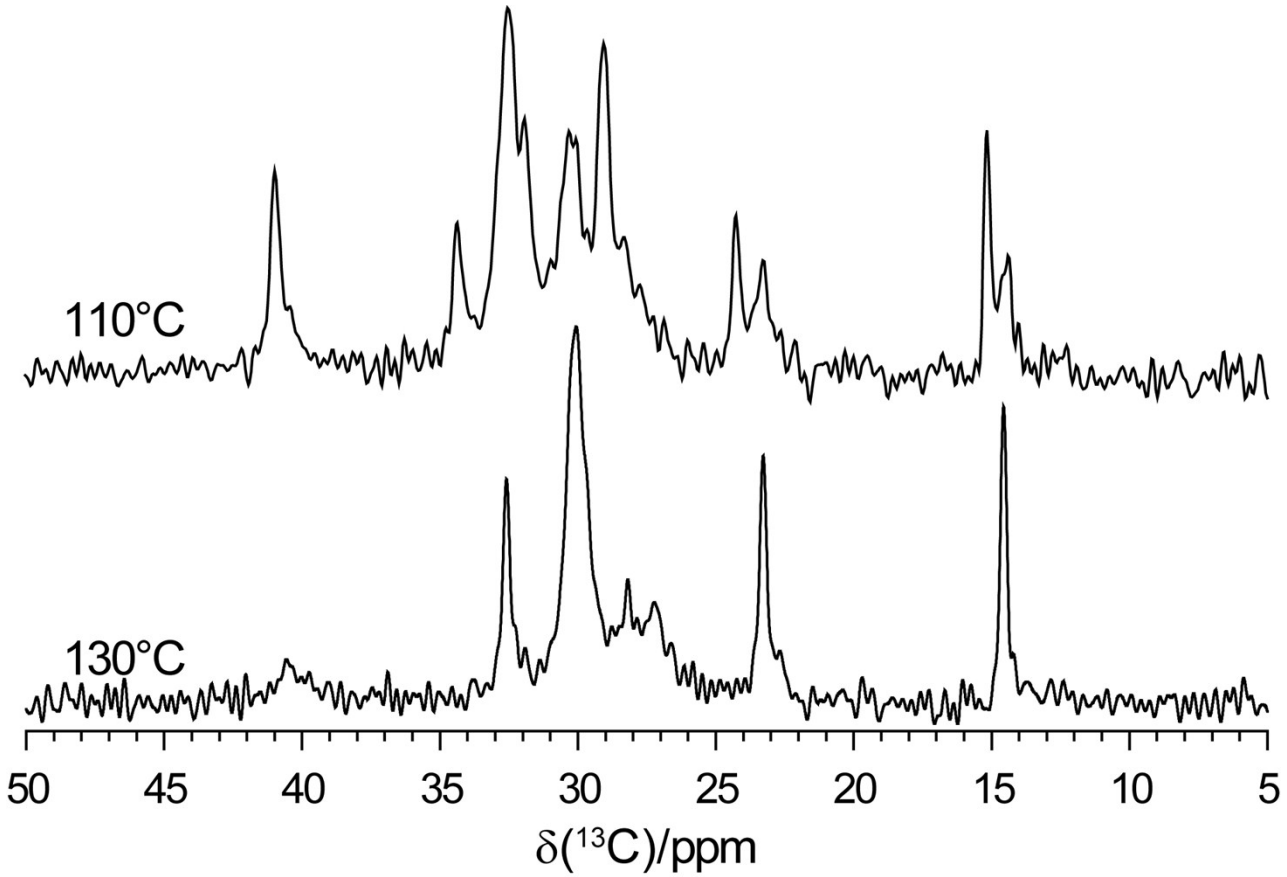

Fig. S4 $^{13}C(^{1}H)$ DD one pulse MAS spectra recorded with a recycle delay of 4 s for C10A treated at 110°C and at 130°C.