

**Supporting information**

**The ethylene glycol-mediated sol-gel synthesis of nano**

**AlF<sub>3</sub>: structural and acidic properties after different post-treatments**

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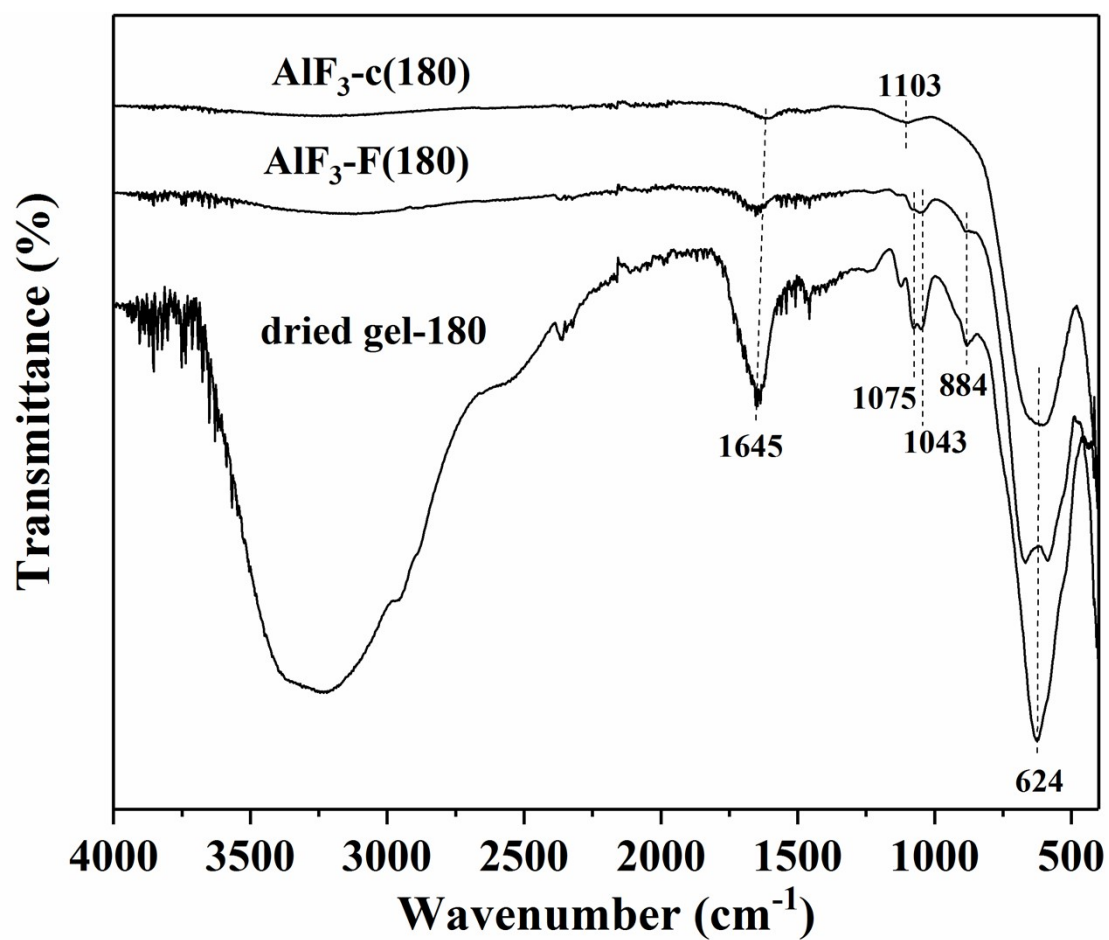


Figure S1 FT-IR spectra of the precursor (dried gel-180) and the resulting nano AlF<sub>3</sub> samples (AlF<sub>3</sub>-F(180) and AlF<sub>3</sub>-c(180)).

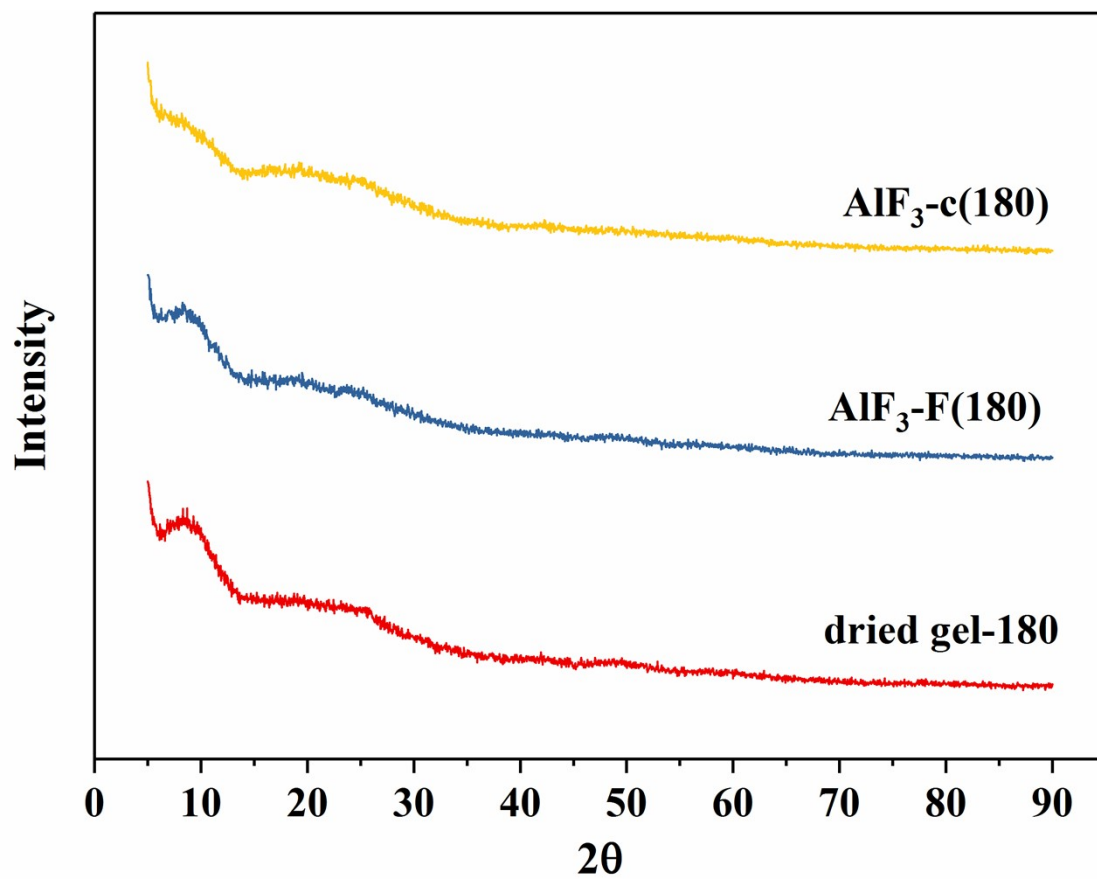


Figure S2 XRD patterns of the precursor (dried gel-180) and the resulting nano AlF<sub>3</sub> samples (AlF<sub>3</sub>-F(180) and AlF<sub>3</sub>-c(180)).

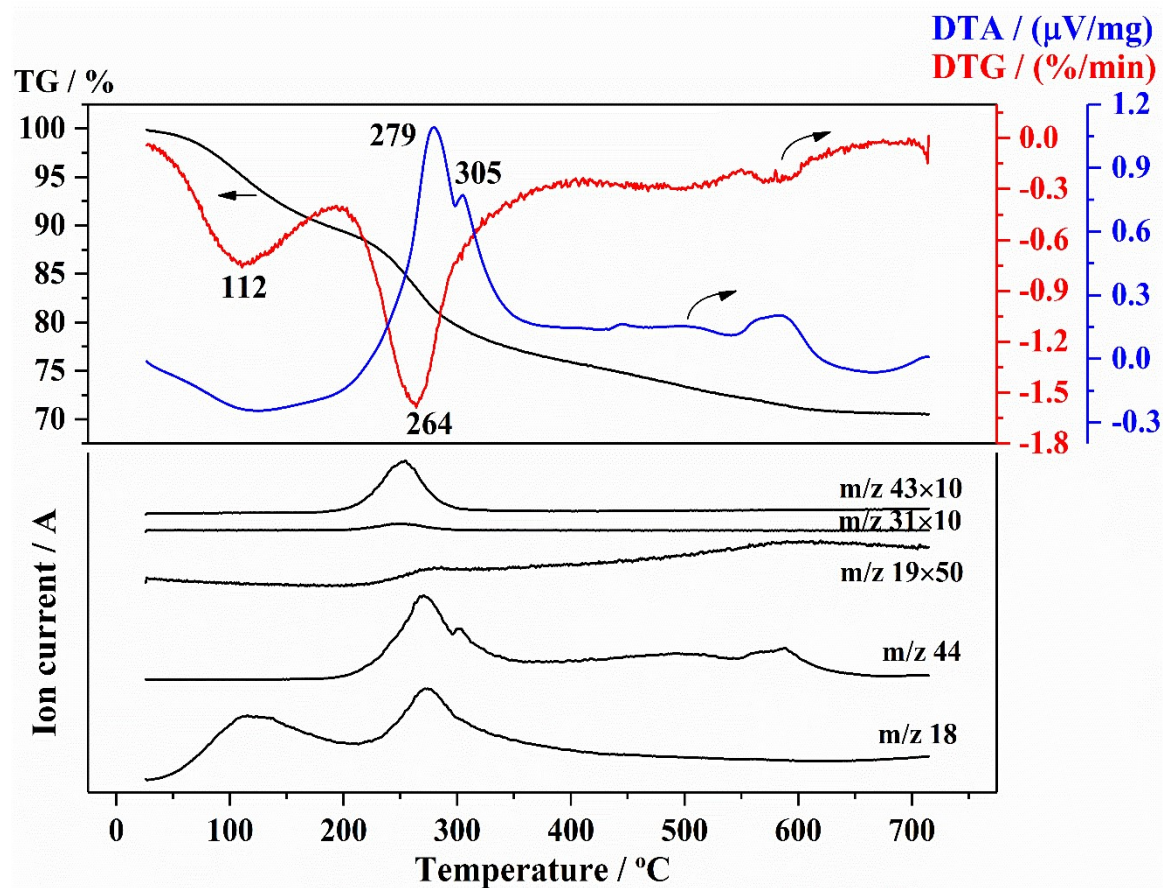


Figure S3 TG-DTA-MS of dried gel-180 under air. (a) TG-DTA curve and (b) MS curves of  $m/z$  18 ( $\text{H}_2\text{O}^+$ ),  $m/z$  19 ( $\text{F}^+$ ),  $m/z$  31 (EG fragment),  $m/z$  43 (isopropanol fragment) and  $m/z$  44 ( $\text{CO}_2^+$ ).

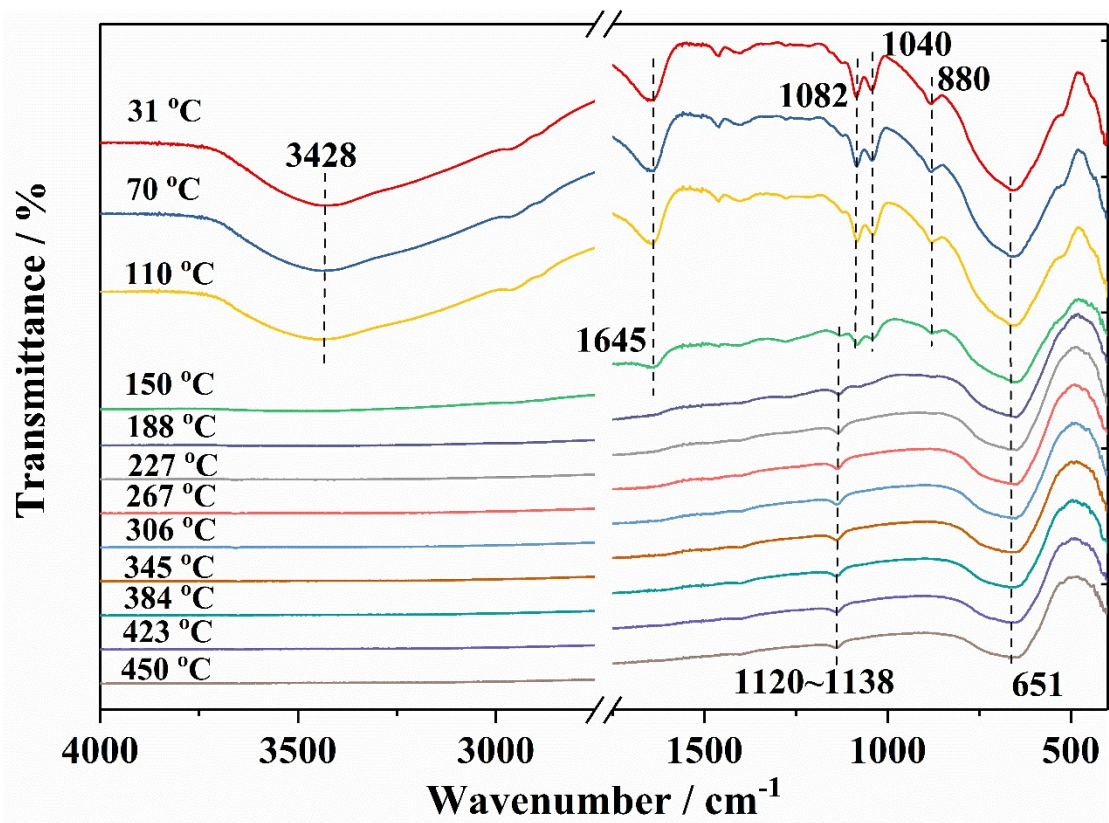


Figure S4 in situ FT-IR spectra of dried gel-120 calcined in the temperature range of room temperature~450 °C.

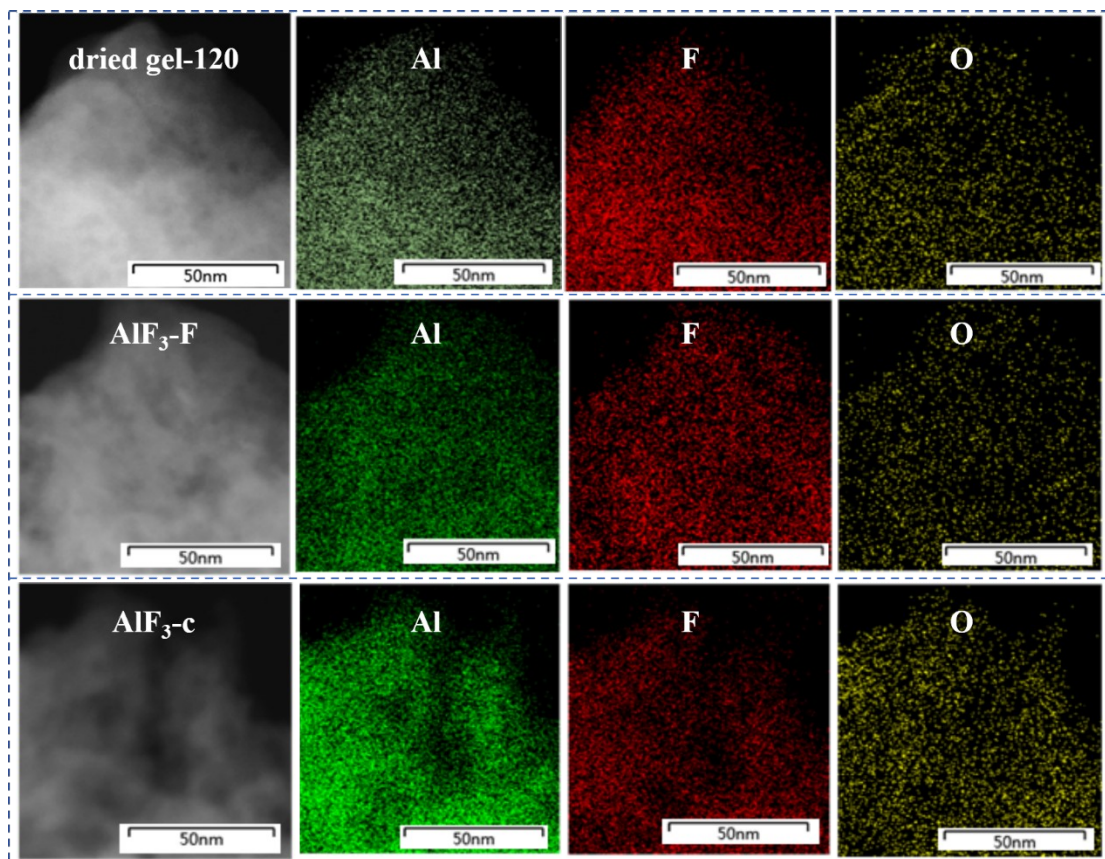


Figure S5 TEM element mapping of the precursor (dried gel-120) and the resulting nano AlF<sub>3</sub> samples (AlF<sub>3</sub>-F and AlF<sub>3</sub>-c).

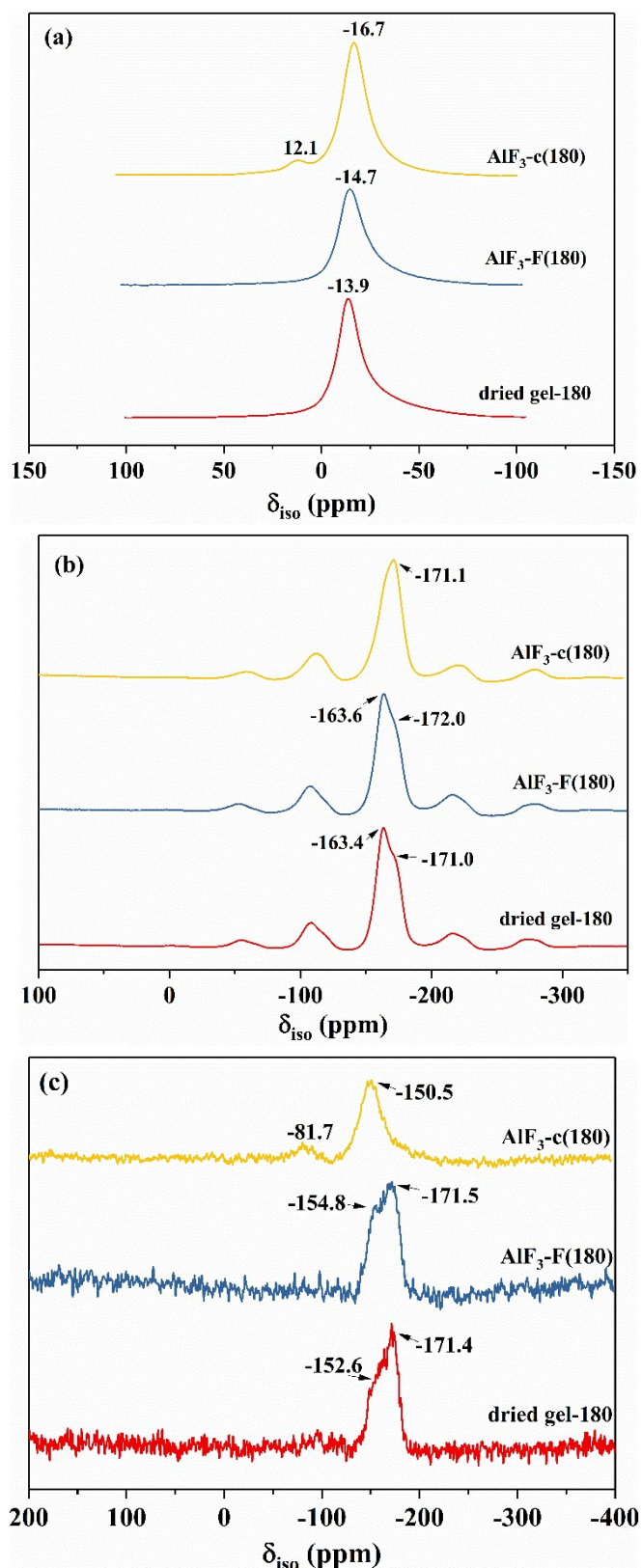


Figure S6 Solid-state MAS NMR spectra of the precursor (dried gel-180) and the resulting nano  $\text{AlF}_3$  after different post treatments. (a) central transition of  $^{27}\text{Al}$  NMR spectra, (b)  $^{19}\text{F}$  MAS NMR spectra and (c) rotor synchronized  $^{19}\text{F}$  spin-echo MAS NMR spectra.

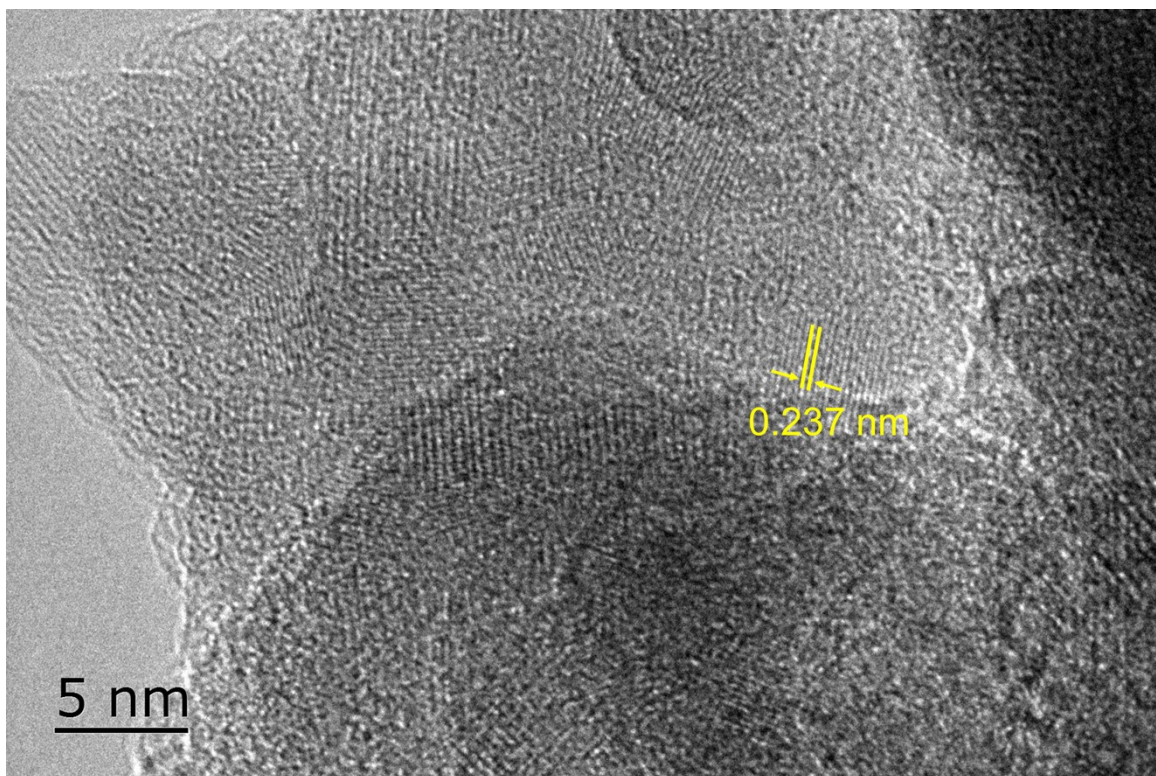


Figure S7 HRTEM of AlF<sub>3</sub>-c.



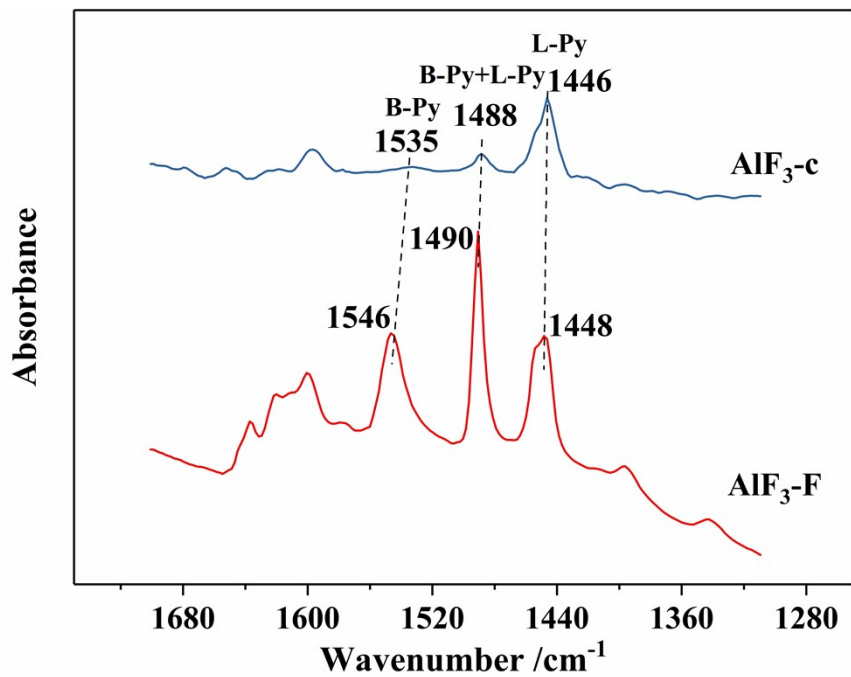


Figure S8 IR spectra of pyridine adsorbed on AlF<sub>3</sub>-F and AlF<sub>3</sub>-c.