

## Supporting Information

# Modifying the Reactivity of a Solid Lewis Acid: Niobium and Antimony Doped Nanoscopic Aluminum Fluoride

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### BET Surface Area

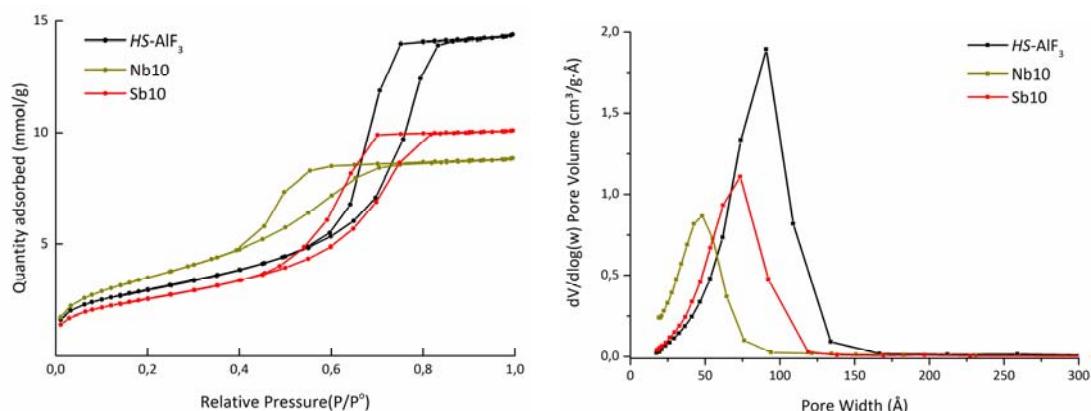


Figure S1: Nitrogen Adsorption Isotherms at 77K for Nb10, Sb10 and the undoped HS-AlF<sub>3</sub> (left) and pore size distribution (right)

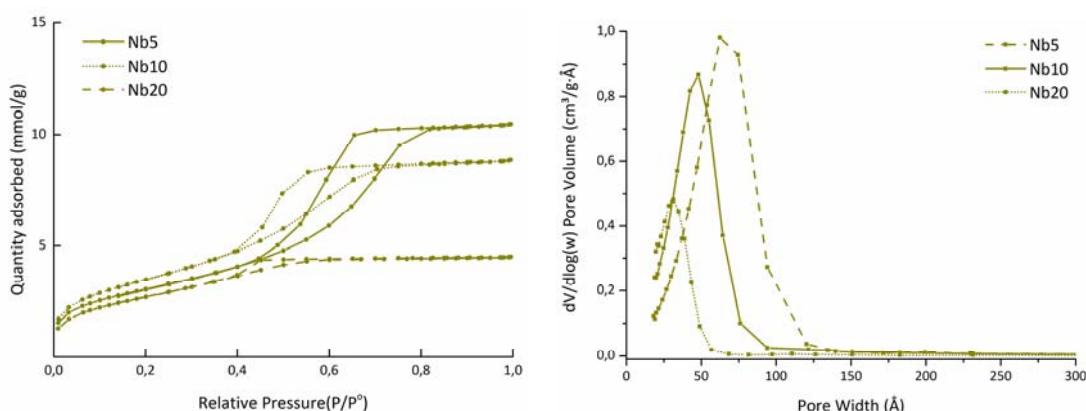


Figure S2: Nitrogen Adsorption Isotherms at 77K for the Niobium-doped series(left) and pore size distribution (right)

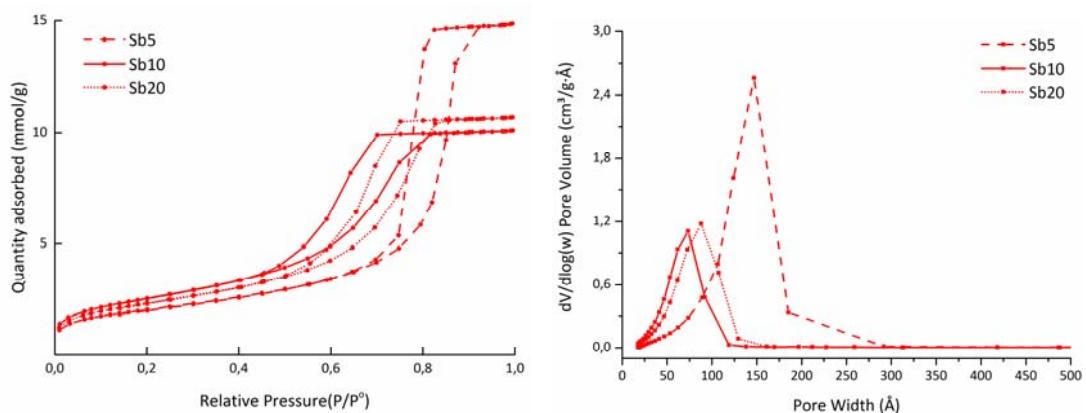


Figure S3: Nitrogen Adsorption Isotherms at 77K for the Antimony-doped series (left) and pore size distribution (right)

### Elemental Analysis

**Table S1** Elemental Analysis for selected catalysts

Entry	Catalyst	%N	%C	%H
1	<i>HS-AlF<sub>3</sub></i>	0,005	1,648	1,683
2	Precursor <i>HS-AlF<sub>3</sub></i>	-0,007	18,271	3,750
3	Nb10	-0,001	1,494	0,352
4	Precursor Nb10	-0,052	24,765	5,242
5	Sb10	0,293	2,373	1,363
6	Precursor Sb10	0,337	4,309	3,009

### MAS NMR Spectra

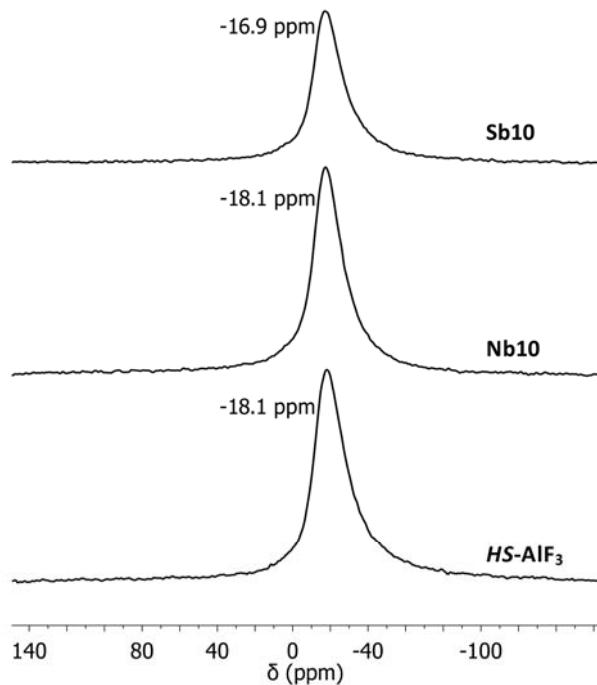


Figure S4: <sup>27</sup>Al MAS NMR spectra for Nb10 (20kHz, ns=800), Sb10 and *HS-AlF<sub>3</sub>* (25kHz, ns=800)

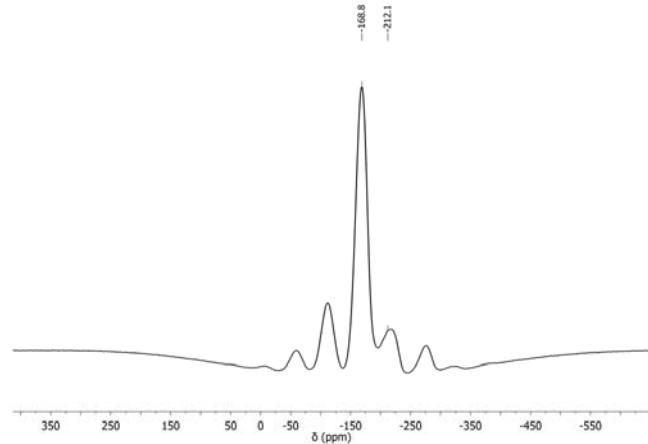


Figure S5:  $^{19}\text{F}$  MAS NMR spectra for Nb5 (20kHz, ns=64, D1=5s). Spinning side bands are depicted in asterisks

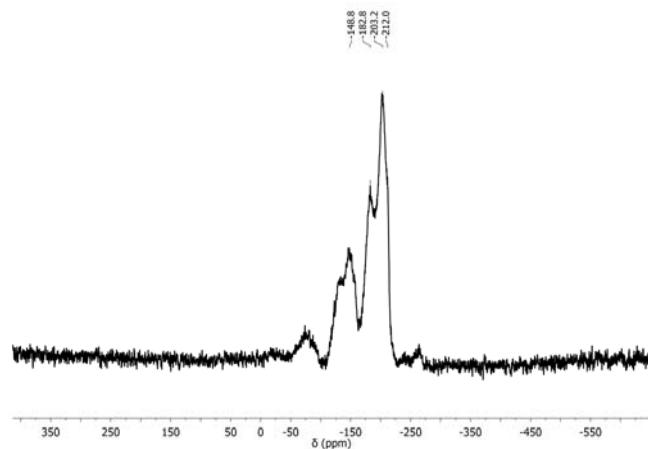


Figure S6:  $^{19}\text{F}$  Echo MAS NMR spectra for Nb5 (20kHz, ns=1280, L0=10)

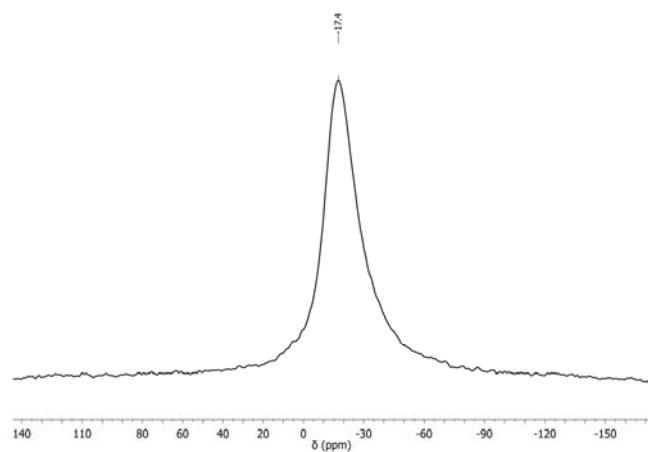


Figure S7:  $^{27}\text{Al}$  MAS NMR spectra for Nb5 (20kHz, ns=800)

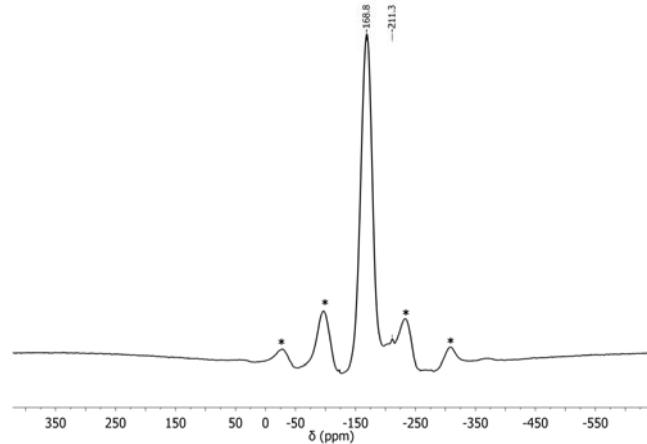


Figure S8:  $^{19}\text{F}$  MAS NMR spectra for Sb5 (25kHz, ns=64, D1=5s). Spinning side bands are depicted in asterisks

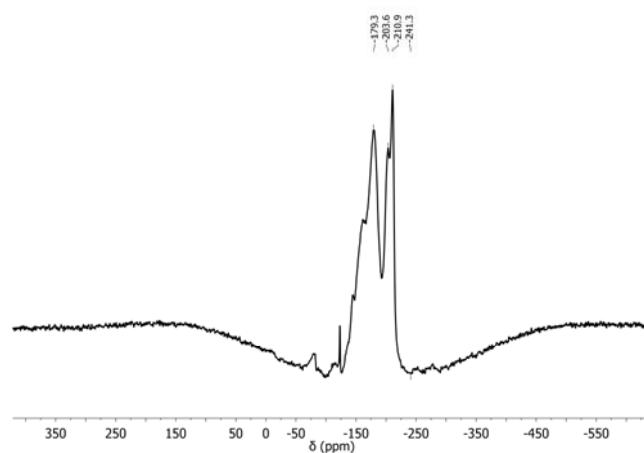


Figure S9:  $^{19}\text{F}$  Echo MAS NMR spectra for Sb5 (25kHz, ns=1024, L0=10)

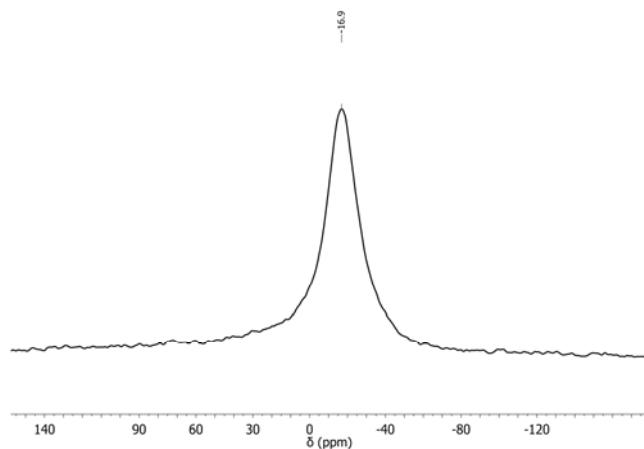


Figure S10:  $^{27}\text{Al}$  MAS NMR spectra for Sb5 (20kHz, ns=512)

### NH<sub>3</sub> TPD Profiles

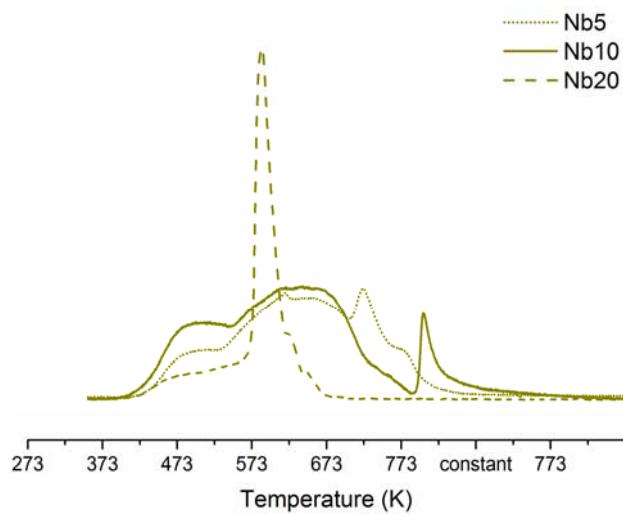


Figure S11: Ammonia TPD Profile for Nb-doped series

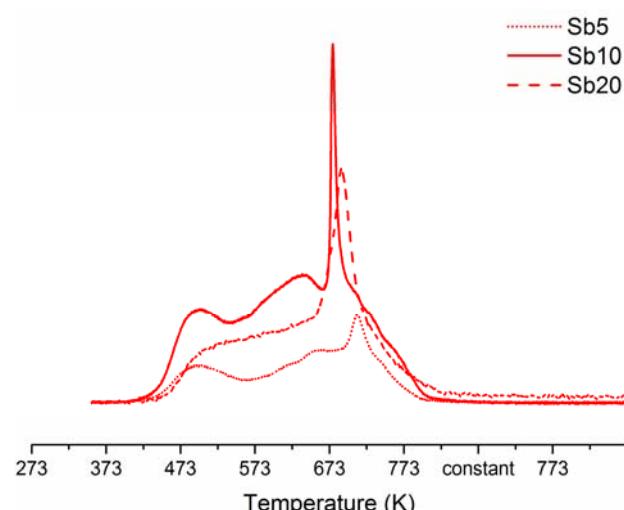


Figure S12: Ammonia TPD Profile for Sb-doped series

### PA-FTIR Spectra of absorbed pyridine (Py-PAS)

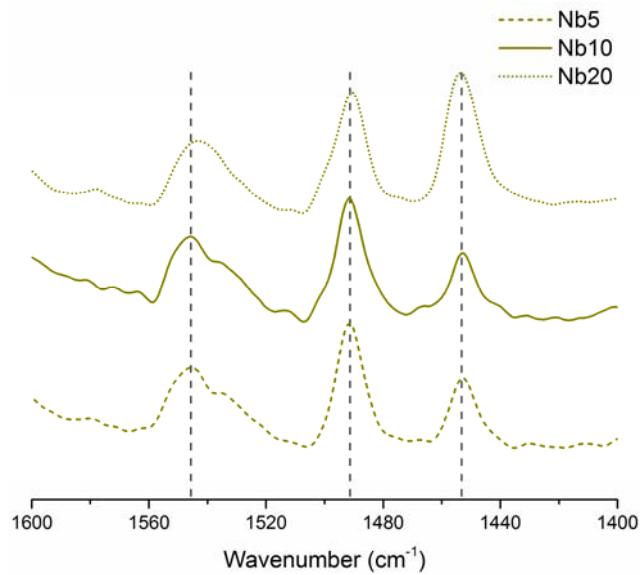


Figure S13: PAS-FTIR of absorbed pyridine on Nb-doped series

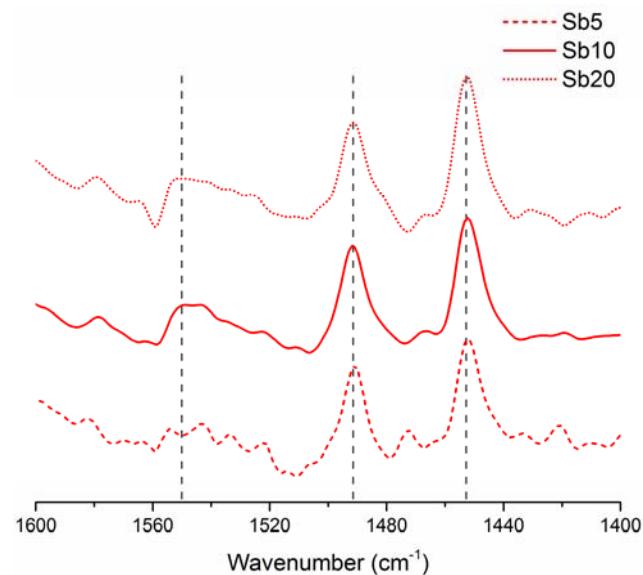


Figure S14: PAS-FTIR of absorbed pyridine on Sb-doped series

## Thermal Analysis (TG and DTA)

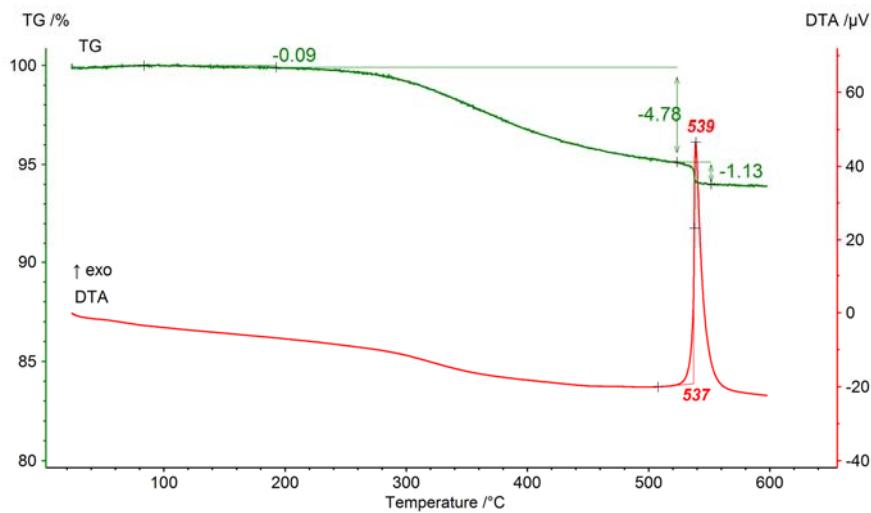


Figure S15. Thermogravimetry (green) and Differential Thermal Analysis (red) of Sb10

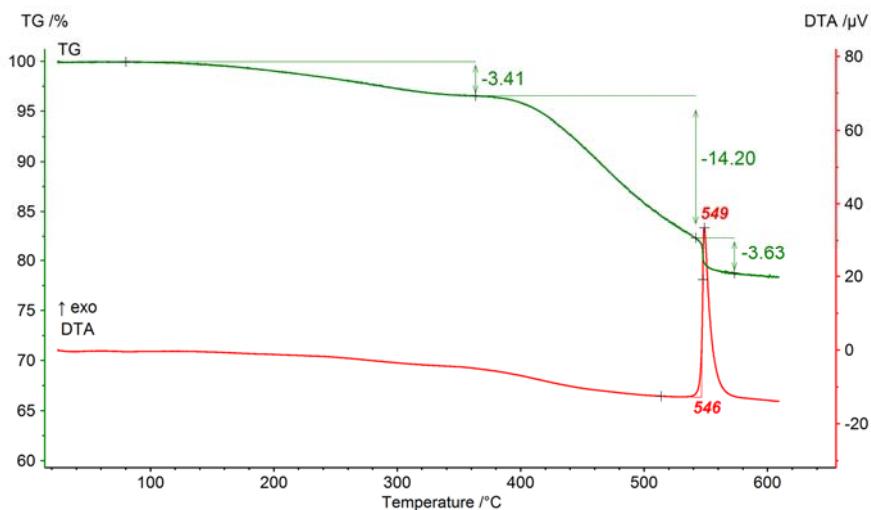


Figure S16. Thermogravimetry (green) and Differential Thermal Analysis (red) of Nb20