

Supporting information for:

Efficient and Reversible Fixation of Carbon Dioxide by NCN-Chelated Organoantimony(III) Oxide.

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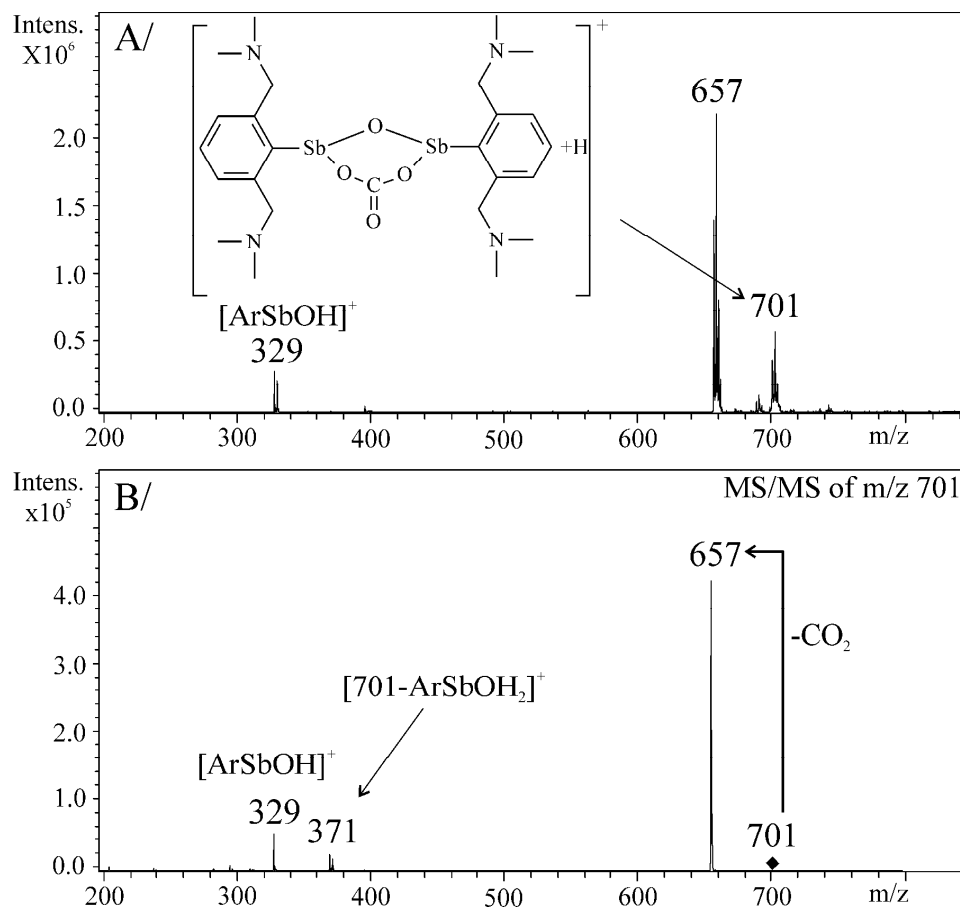


Figure S1. Positive-ion ESI mass spectra of compound **3**. A/ full scan mass spectrum, B/ MS/MS spectrum of $[\text{Ar}_2\text{Sb}_2\text{OCO}_3]^+$ ion at m/z 701.

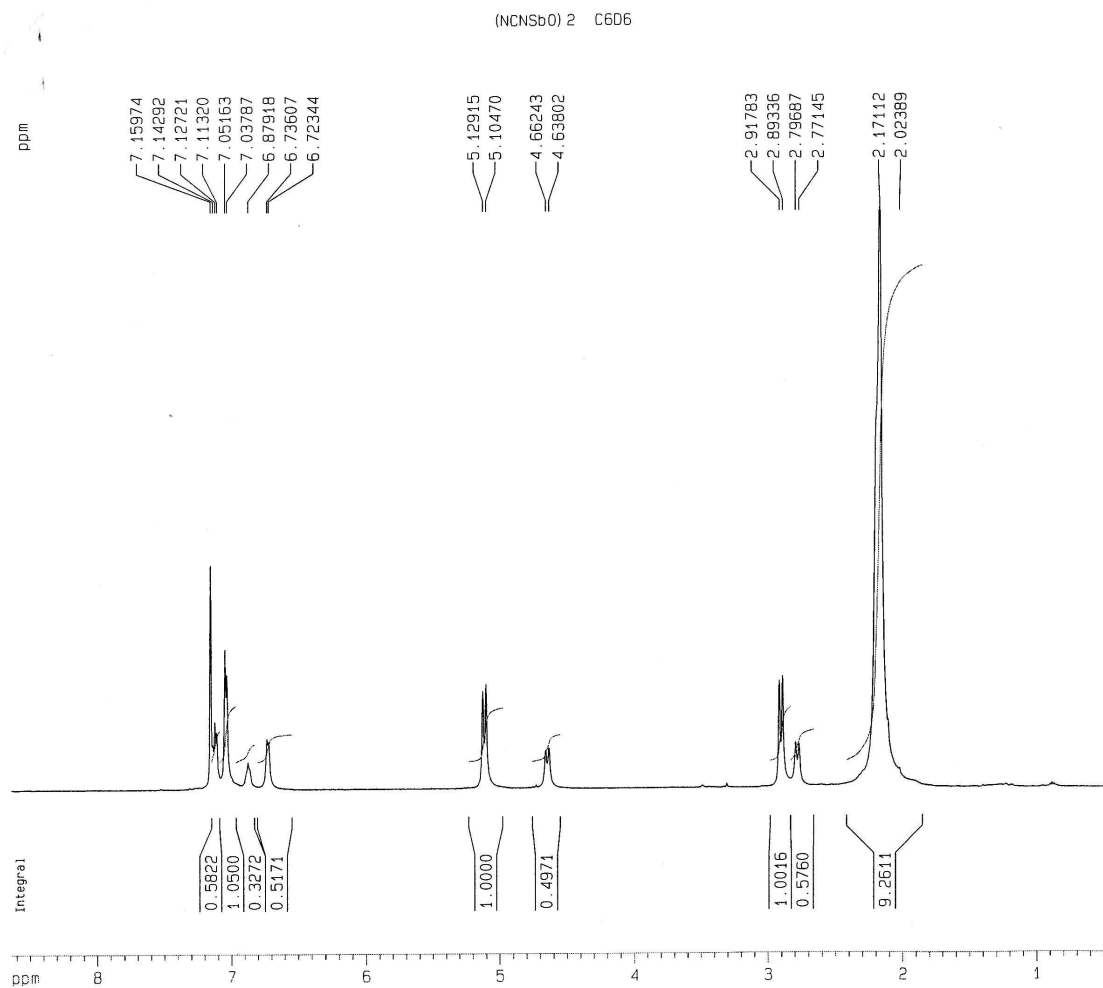


Figure S2. ^1H NMR spectrum of compound **2** in C_6D_6 .

NCNSbO C6D6

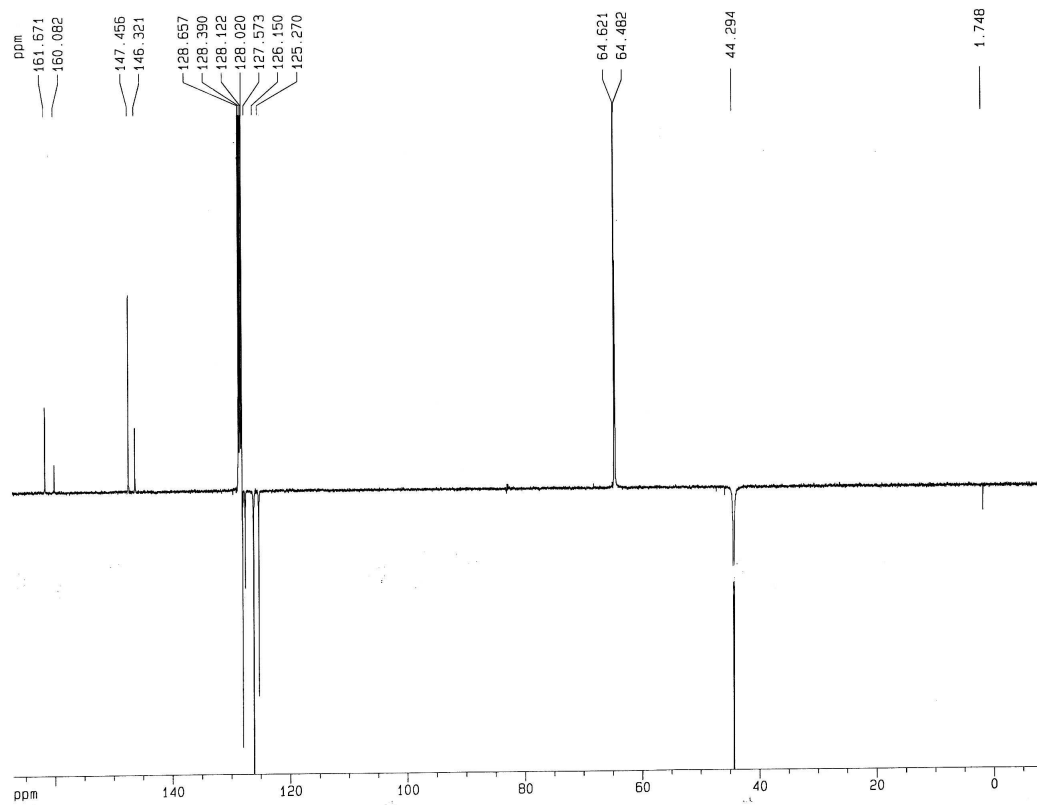


Figure S3. ^{13}C NMR spectrum of compound 2 in C_6D_6 .

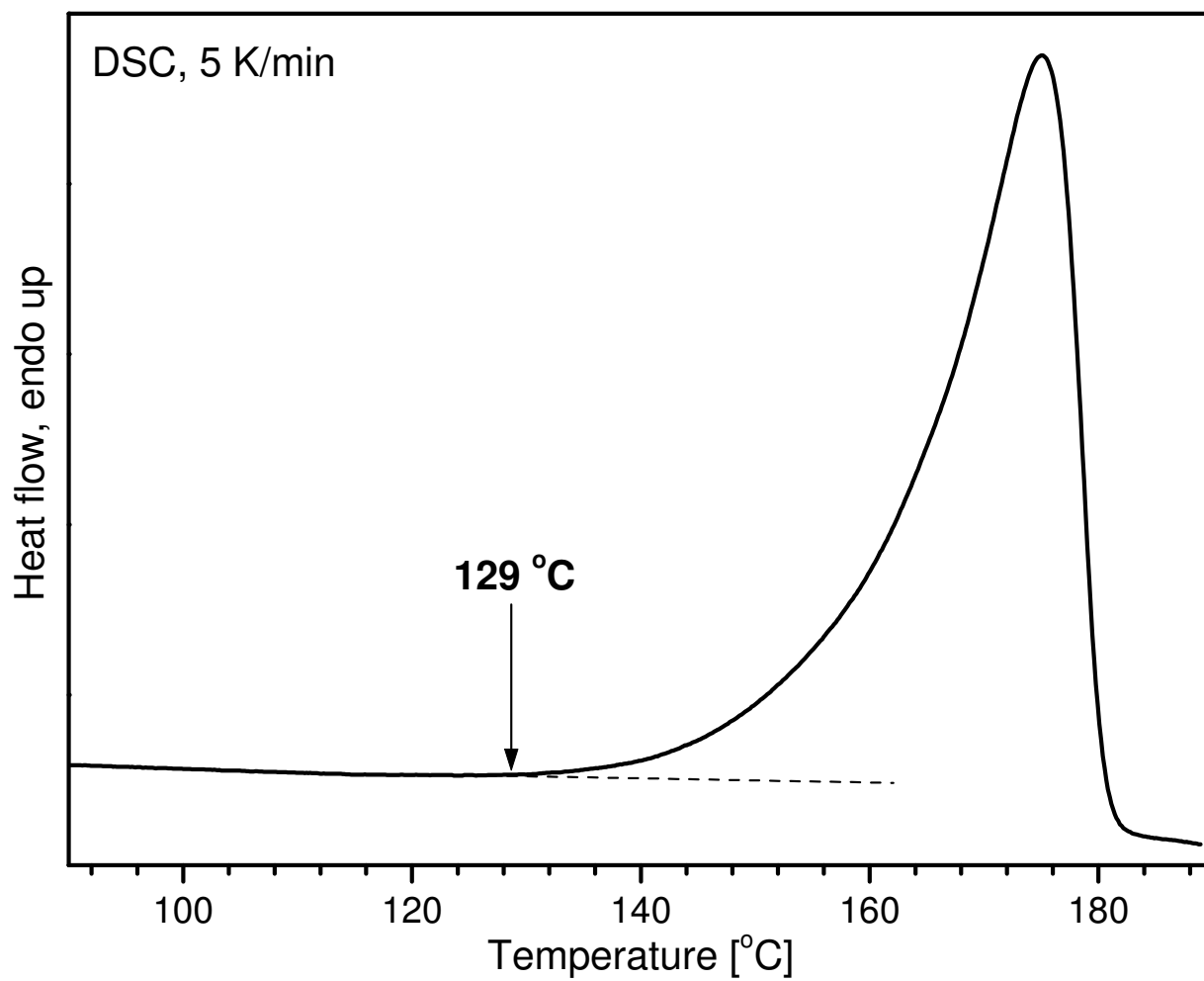


Figure S4. Example of the DSC parameters of **3** (5K/min)

X-ray crystallography

Suitable single crystal of **2** and **3** were mounted on a glass fiber with an oil and measured on four-circle diffractometer KappaCCD with CCD area detector by monochromatized MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). The numerical¹ absorption correction from crystal shape was applied for both crystals of **2** and **3**. The structures were solved by the direct method (SIR92²) and refined by a full matrix least squares procedure based on F^2 (SHELXL97³). Hydrogen atoms were mostly localized on a difference Fourier map, however to ensure the uniformity of treatment of crystal, all hydrogen were recalculated into idealized positions (riding model) and assigned temperature factors $H_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{pivot atom})$ or of $1.5U_{\text{eq}}$ for the methyl moiety with C-H = 0.96 \AA , 0.97, and 0.93 \AA for methyl, methylene, and hydrogen atoms in aromatic ring, respectively, and 0.82 \AA for O-H bonds. The water molecules of O2W and O3W moieties were localized in two symmetry constrained positions with half occupancy of oxygen atoms. The appropriate hydrogen atoms of these molecules can be split to two different (perpendicular) orientations, where the plausible short contacts with adjacent molecules from another plane can be found. The final difference maps displayed no peaks of chemical significance as the highest peaks and holes are in close vicinity ($\sim 1 \text{ \AA}$) of heavy atoms. Crystallographic data for structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC no717975 and 717976. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EY, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or [www: http://www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk)).

Crystallographic Data for 2 and 3

	2	3
empirical formula	C ₁₂ H ₁₉ N ₂ SbO	C ₁₃ H ₂₃ N ₂ SbO ₅
cryst syst	monoclinic	monoclinic
space group	<i>P2₁/c</i>	<i>C2/c</i>
<i>a</i> [Å]	6.5280(4)	11.6248(2)
<i>b</i> [Å]	17.4160(11)	16.3348(2)
<i>c</i> [Å]	12.1560(10)	18.3135(5)
β [°]	10.706(6)	106.296(12)
<i>Z</i>	4	8
μ [mm ⁻¹]	2.015	1.674
<i>D_x</i> [Mg m ⁻³]	1.609	1.628
cryst size [mm]	0.45x0.17x0.07	0.38x0.30x0.18
θ range, [deg]	1-27.5	1-27.5
<i>T_{min}</i> , <i>T_{max}</i>	0.621, 0.897	0.664, 0.806
no. of reflns measd	13 867	13 911
no. of unique reflns, <i>R_{int}</i> ^a	3095, 0.086	3803, 0.050
no. of obsd reflns [<i>I</i> >2 σ (<i>I</i>)]	2506	3095
no. of params	145	191
<i>S</i> ^b all data	1.099	1.108
final <i>R</i> ^c indices [<i>I</i> >2 σ (<i>I</i>)]	0.034	0.037
w <i>R</i> ^c indices (all data)	0.084	0.081
$\Delta\rho$, max., min. [e Å ⁻³]	0.731, -0.771	0.472, -1.017

$$^a R_{\text{int}} = \frac{\sum |F_o^2 - F_{o,\text{mean}}^2|}{\sum F_o^2}, \quad ^b S = \left[\frac{\sum (w(F_o^2 - F_c^2)^2)}{(N_{\text{diffns}} - N_{\text{params}})} \right]^{1/2}, \quad ^c R(F) = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}, \quad wR(F^2) = \left[\frac{\sum (w(F_o^2 - F_c^2)^2)}{\sum w(F_o^2)^2} \right]^{1/2}.$$

References:

S1. P. Coppens, in *Crystallographic Computing*, ed. F.R. Ahmed, S.R. Hall, and C.P. Huber, Copenhagen, Munksgaard **1970**, 255–270.

S2. Altomare, A.; Cascarone, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M.C.; Polidori, G.; Camalli, M. *J. Appl. Crystallogr.* **1994**, 27, 1045.

S3. Sheldrick, G.M. SHELXL-97, A Program for Crystal Structure Refinement. University of Göttingen, Germany 1997.