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A facile solvent free Synthesis Route for the Assembly of Highly CO_2 Selective and H_2S tolerant NiSIFSIX Metal-Organic Framework.

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Supplementary Information

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I. Structure refinement

Powder X-ray diffraction (PXRD) measurements were carried out at room temperature on a PANalyticalX'Pert PRO diffractometer 45kV, 40mA for CuKα (λ = 1.5418 Å), from 8 to 80° with a scan speed of 1.0° min^{-1} and a step size of 0.02° in 2θ.

The indexation of the PXRD diagram, carried out using the McMaille software,[1] yielded a primitive tetragonal cell (M(20)= 42.4 and F(20)= 64.2 (0.0139, 36)). The cell parameters, a= b= 7.015(1) Å, c = 7.669(1) Å, were refined by a whole powder pattern fit using the Le Bail method,[2] implemented in the FULLPROF software,[3] and were consistent with those of the previously described **SIFSIX**-3-Cu. Hence, atomic positions of the latter were used as structure solution. The final Rietveld refinement permitted access to satisfactory results: R_{Bragg} = 0.010 and R_{Factor} = 0.096 (Figure S1). The crystallographic data and refinement parameters of **SIFSIX**-3-Ni are summarized in the Table S1.

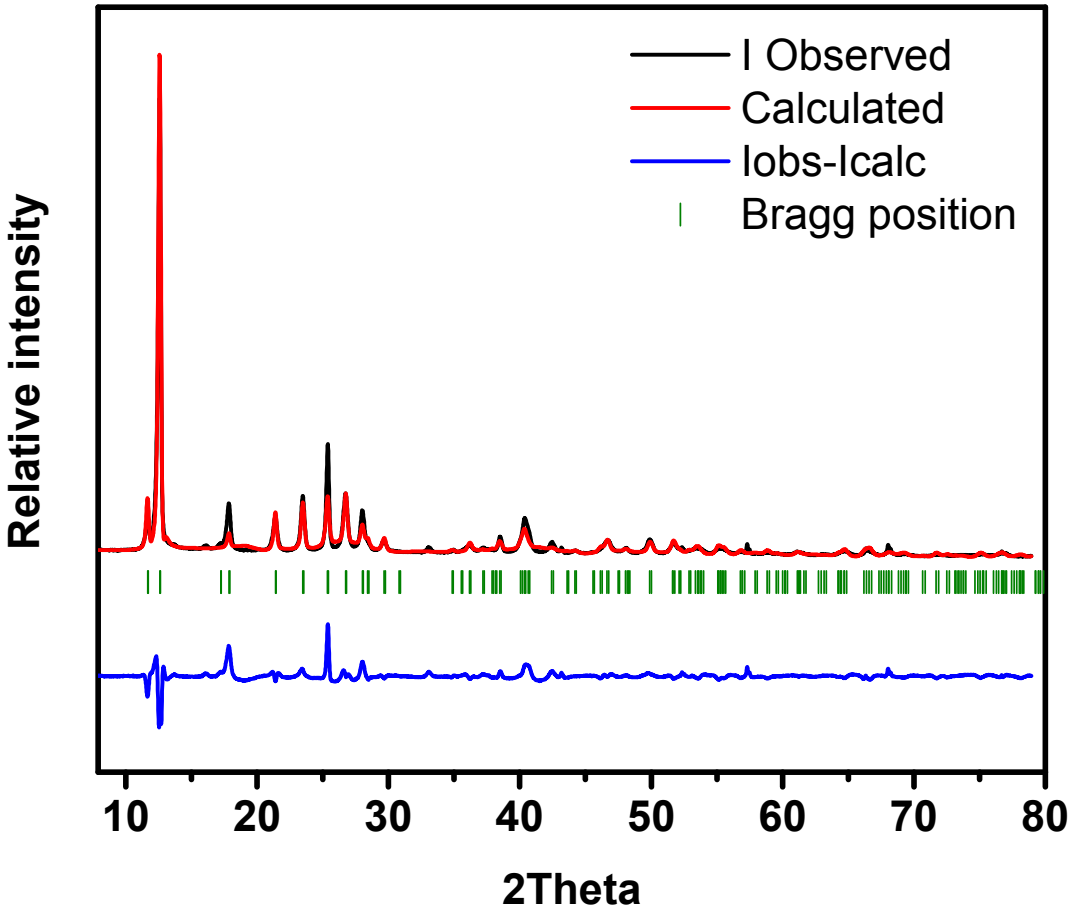

Figure S1: Rietveld refinement of **SIFSIX**-3-Ni

Table S1: Crystallographic data of **SIFSIX**-3-Ni

Compound	**SIFSIX**-3-Ni
Formula	$C_8N_4H_8NiSiF_6$
Formula weight ($g.mol^{-1}$)	360.96
Crystal system	Tetragonal
Space group	P 4/mmm (n^o 123)
a (Å)	7.0147(1)
c (Å)	7.669(1)
V ($Å^3$)	372.34
Z	1
Wavelength λ(Cu Kα)	1.5406
Temperature (K)	298
2-theta range (°)	8-80
Number of independent reflections	247
Number of intensity-dependent parameters	17
R_p, R_{wp}, R_B, R_F	0.337, 0.344, 0.104, 0.096

II. SIFSIX-3-Ni stability after H_2S or H_2O treatment

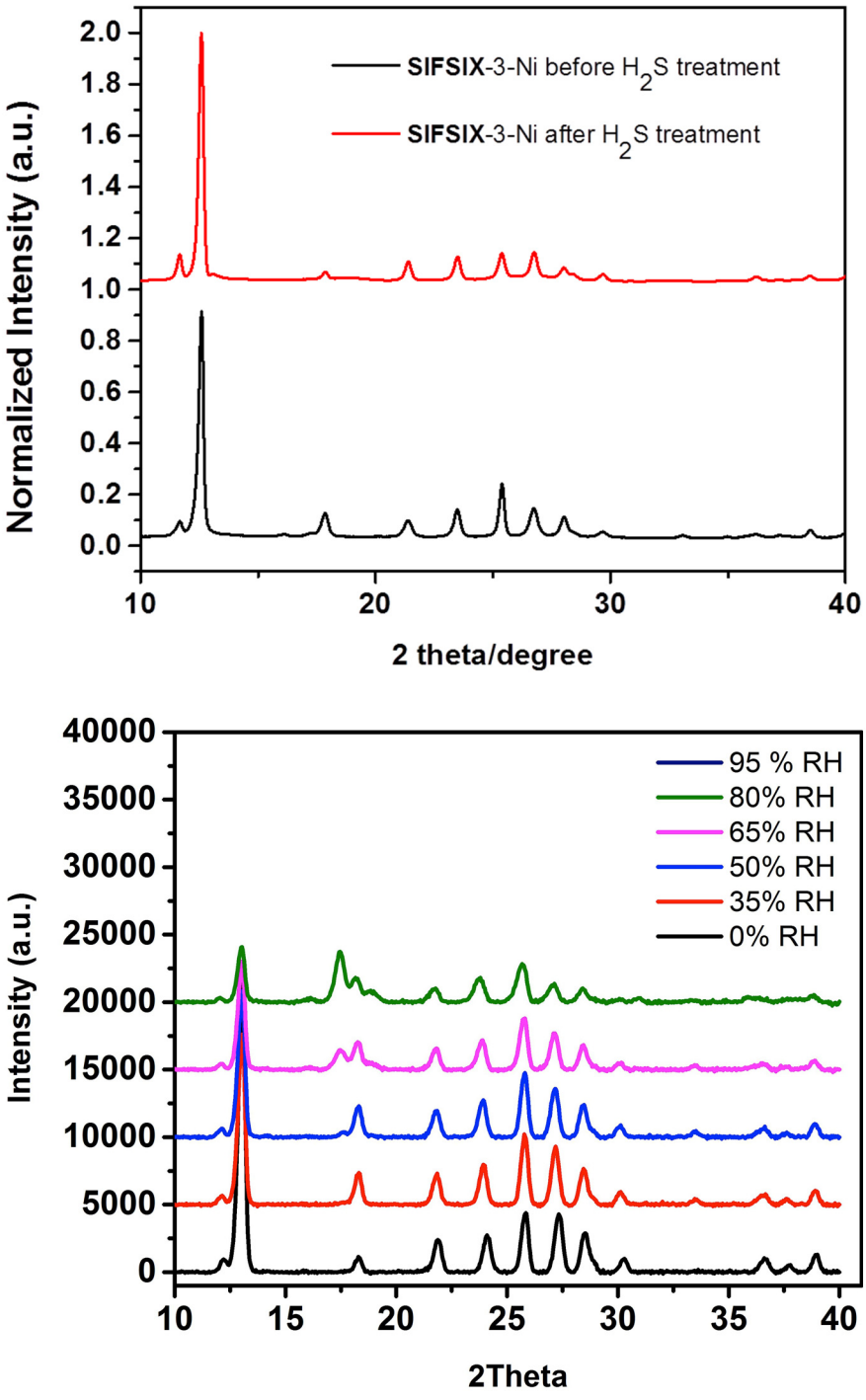

Figure S2: PXRD for **SIFSIX**-3-Ni after H_2S treatment (top) and H_2O exposure (bottom).

III. Low-Pressure Gas Adsorption Measurements

Homogenous microcrystalline sample of **SIFSIX-3-Ni** was activated by washing as-synthesized powder with 3 x 20 mL of methanol. In a typical experiment, approximately 30 mg of sample was transferred with solvent in to a 9-mm large bulb glass sample cell and dried under the dry N_2 flow. The sample was first evacuated at room temperature using a turbo vacuum pump for 8 h and then gradually heated to 105 °C, held for 8 h and cooled to room temperature.

The determination of the isosteric heats of adsorption (*Q*st) for CO_2 was estimated by applying the Clausius-Clapeyron expression using the CO_2 adsorption isotherms measured at 258, 273, 288 and 298 K.

III.1 Isosteric heats of adsorption for SIFSIX-3-Ni

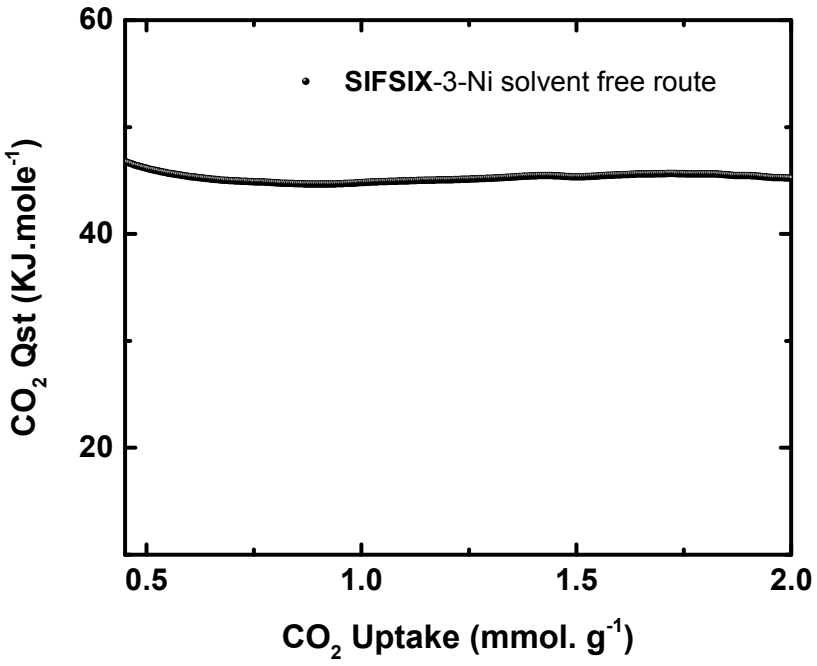

Figure S3: Isosteric heats of adsorption at low coverage for **SIFSIX**-Ni-3 prepared using solvent free route.

III.2 H_2S sorption isotherm for SIFSIX-3-Ni

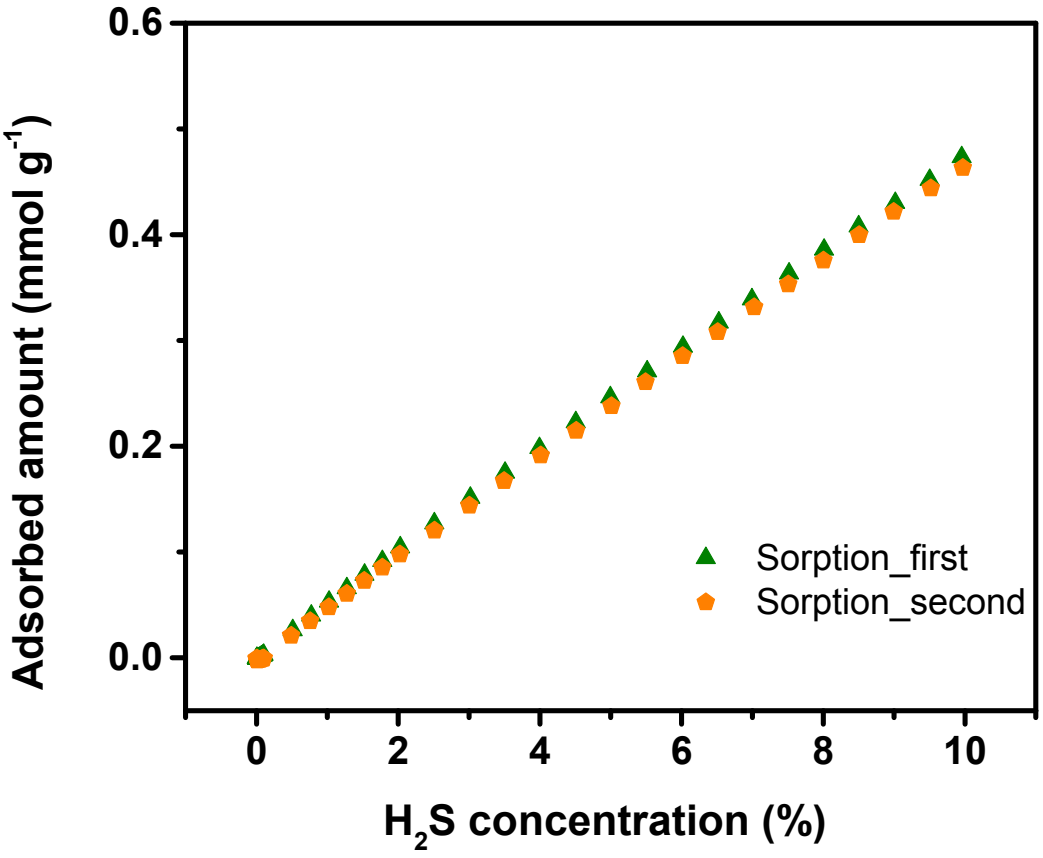

Figure S4: H_2S sorption isotherms for **SIFSIX**-3-Ni which remains same after two cycles of H_2S sorption.

III.3 CO_2 sorption isotherm for SIFSIX-3-Ni after H_2S exposure

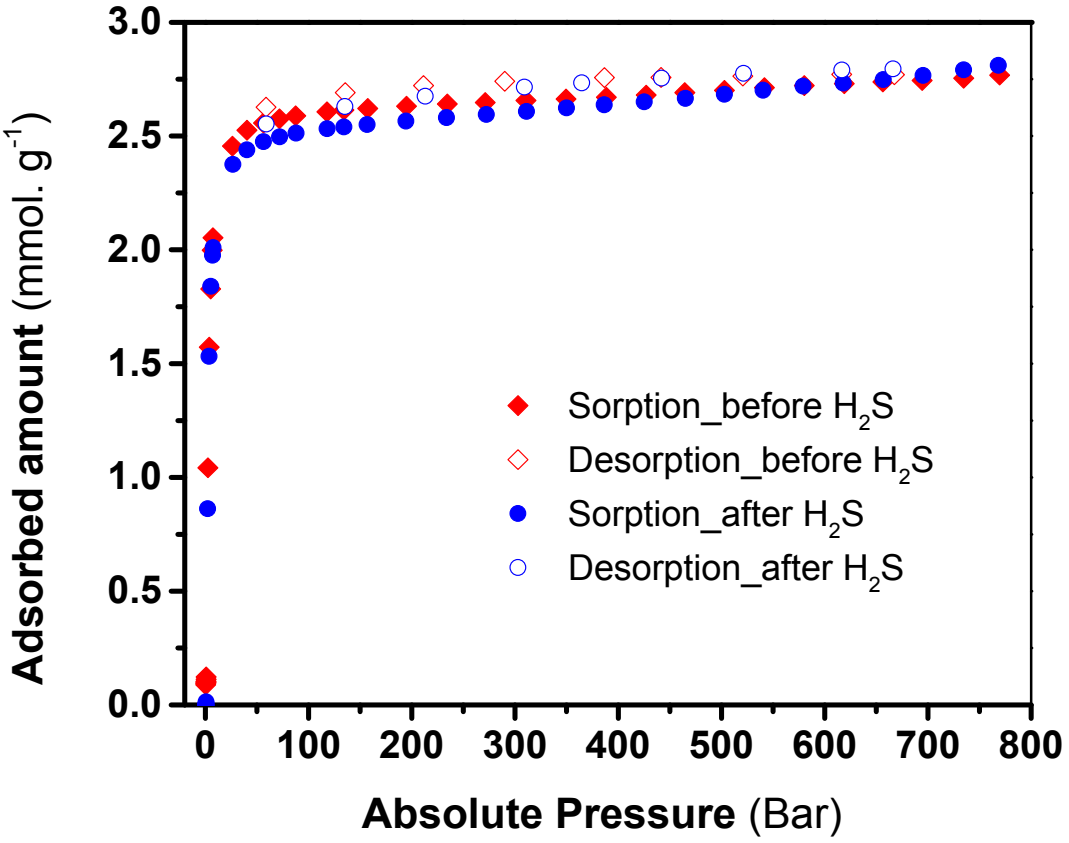

Figure S5: CO_2 sorption isotherms for **SIFSIX**-3-Ni which remains the same after H_2S sorption.

IV. References

1. Le Bail, A. Monte Carlo indexing with McMaille. Powder Diffraction **19**, 249-254 (2004).
2. Le Bail, A., Duroy, H. & Fourquet, J. L. *Ab-initio* structure determination of $LiSbWO_6$ by X-ray powder diffraction. Materials Research Bulletin **23**, 447-452 (1988).
3. Rodriguez-Carvajal, J. Abstracts of the Satellite Meeting on Powder Diffraction of the XV Congress of the IUCr, 127 (1990).