

Supporting Information

**Production and Characterization of a new
Copper(II) Propanoate-Isonicotinamide
Adduct obtained via Slow Evaporation and
using Supercritical CO₂ as an Antisolvent**

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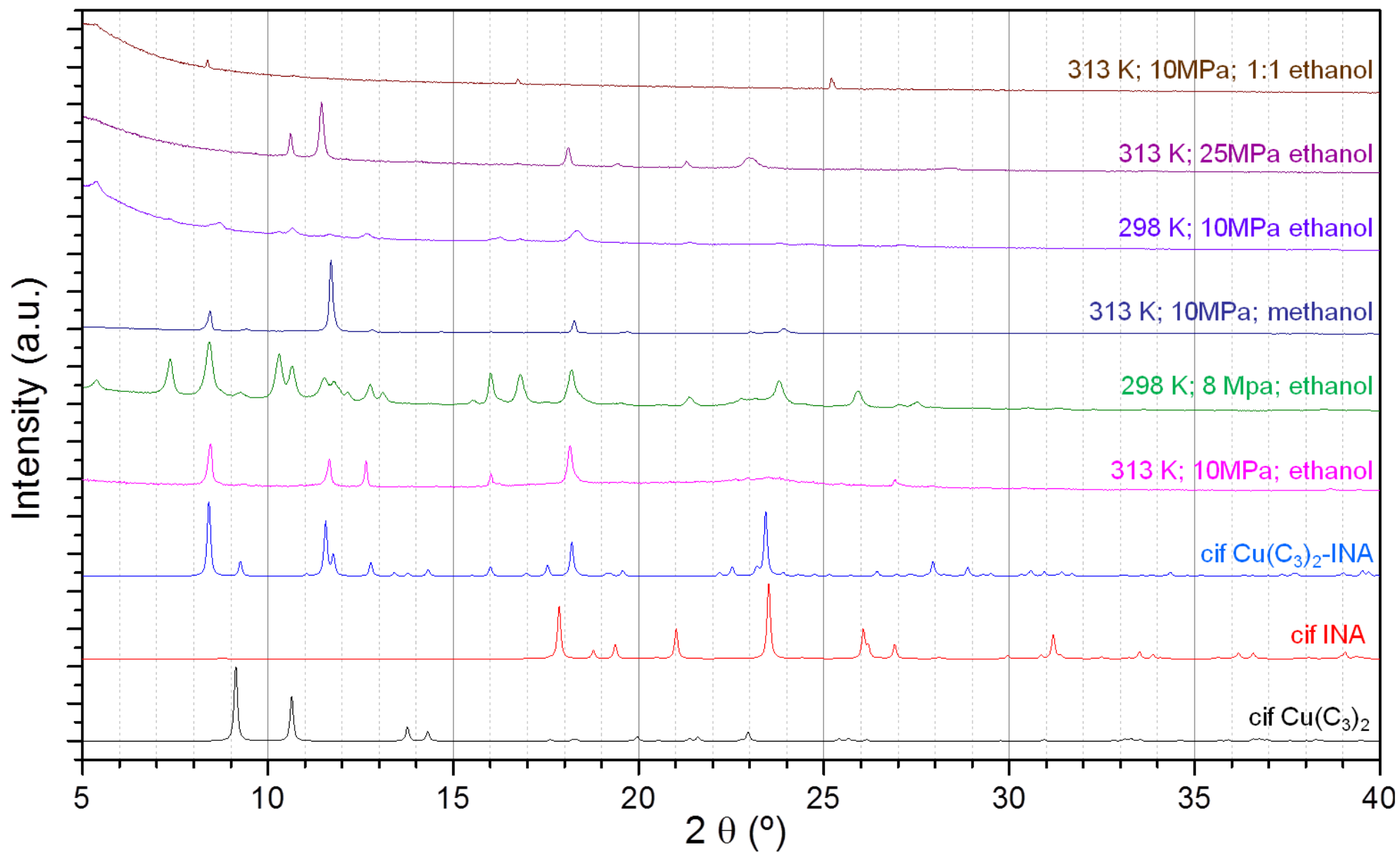
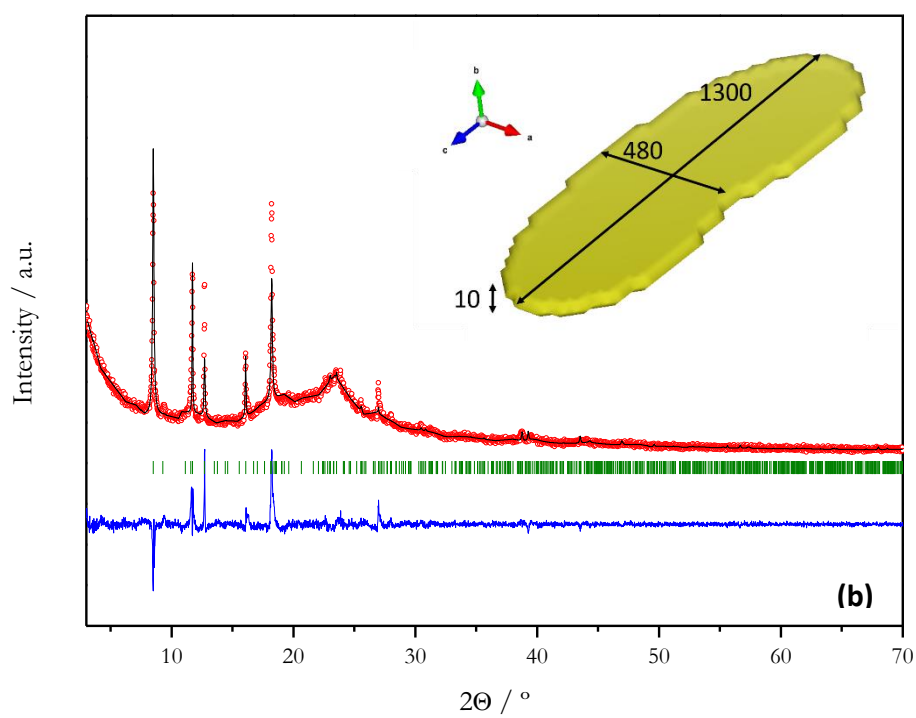
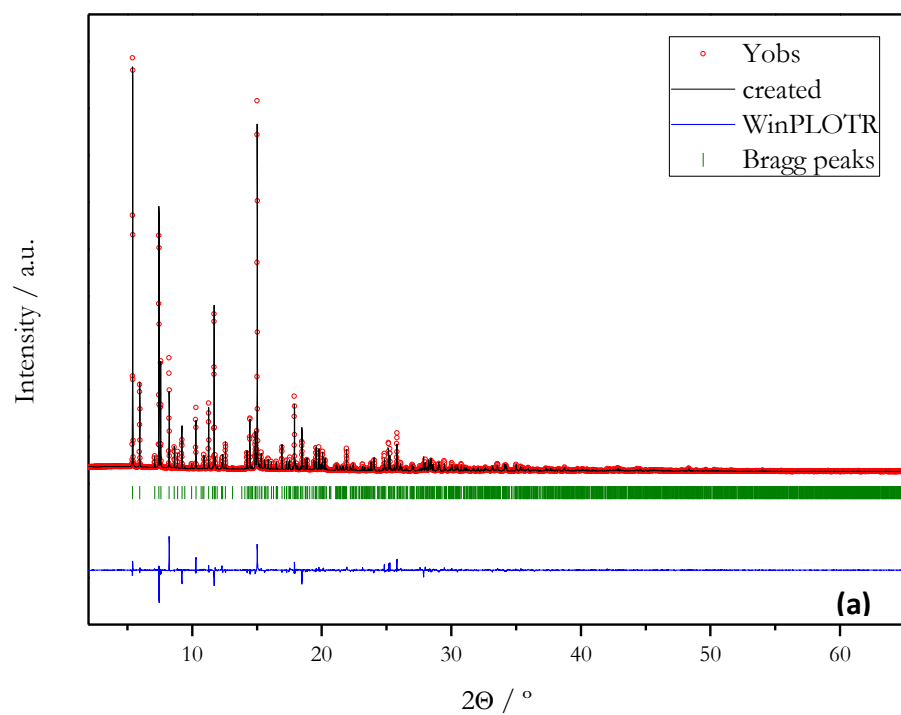


Figure S1. PXRD patterns of the samples precipitated by SAS at the different conditions with a 1:2 $\text{Cu}(\text{C}_3)_2$ to INA molar ratio unless otherwise specified and comparison with the CIF files for $\text{Cu}(\text{C}_3)_2$, INA and the $\text{Cu}_2(\text{C}_3)_4(\text{INA})_4$ adduct.



b)

Figure S2. Rietveld refinement at room temperature for the adduct obtained by slow evaporation (a) and by SAS at 313 K and 10 MPa using ethanol as solvent (b), showing in the last case the average crystal domain sizes (in Å) obtained by microstructural analysis.

Table S1. Crystallographic data. Experimental parameters and structure refinement data for $\text{Cu}_2(\text{C3})_4\text{INA}_4$ obtained by slow evaporation and SAS.

Crystal data	$\text{Cu}_2(\text{C3})_4\text{INA}_4$	
Preparation method	Slow evaporation	SAS
M_r (g·mol ⁻¹)	453.94	453.94
cell setting	monoclinic	monoclinic
space group (no.)	$P2_1/c$ (14)	$P2_1/c$ (14)
temperature (K)	298(2)	298(2)
a (Å)	10.82731(7)	10.7161(4)
b (Å)	12.38686(5)	12.9(6)
c (Å)	15.48057(9)	15.5614(6)
β (°)	103.4426(4)	104.000(4)
V (Å ³)	2019.317(19)	2086(101)
Z , D_c (g·cm ⁻³)	4, 1.493	4, 1.515
wavelength (Å)	0.9940(1)	Cu K α 1
Data collection		
diffractometer	I711 (MAX IV Laboratory)	PANalytical X'Pert MPD (U.Complutense)
specimen mounting	borosilicate glass capillary	
data collection mode	transmission	
scan mode	2θ -step scan	
2θ range (°), step size (° 2θ)	0–70, 0.008	5–60, 0.016
Refinement		
Refinement method	full-matrix least-squares on I_{net}	
R_p , R_{wp} , R_{exp}	0.0180, 0.0354, 0.0508	0.0581, 0.0844, 0.0471
R_F , R_{BRAGG} goodness-of-fit	0.0785, 0.0975 0.64	0.0834, 0.0224 1.79
profile function	pseudo-Voigt with axial divergence asymmetry	
no. contributing reflections	3863	854
no. Parameters	253	7
no. restraints	149	3 rigid bodies
CCDC deposition number	1848049	

The diffraction pattern was analyzed by Rietveld refinement using the Fullprof Suite1-2^{1,2} to evaluate the crystal size (from the broadening of the peaks in the whole pattern). The instrumental resolution function was obtained from LaB₆ and all the patterns were fit with the Thompson-Cox-Hastings pseudo-Voigt profile function through the FullProf program.

The microstructure analysis shows an average crystal domain corresponding to a platelet, which is large in the directions (100) and (001) but extremely narrow in the

direction (010); in the order of 10 Å. This direction corresponds to the stacking of the isonicotinamide aromatic rings. No sign of other crystalline phases (reactants, or new phases) was observed in this pattern.

(1) Rodríguez-Carvajal, J., Recent advances in magnetic structure determination by neutron powder diffraction. *Phys. B.* **1993**, 192, 55-69.

(2) Rodríguez-Carvajal, J., Recent developments of the program FULLPROF. *Commission on powder diffraction (IUCr). Newsletter* **2001**, 26, 12-19.

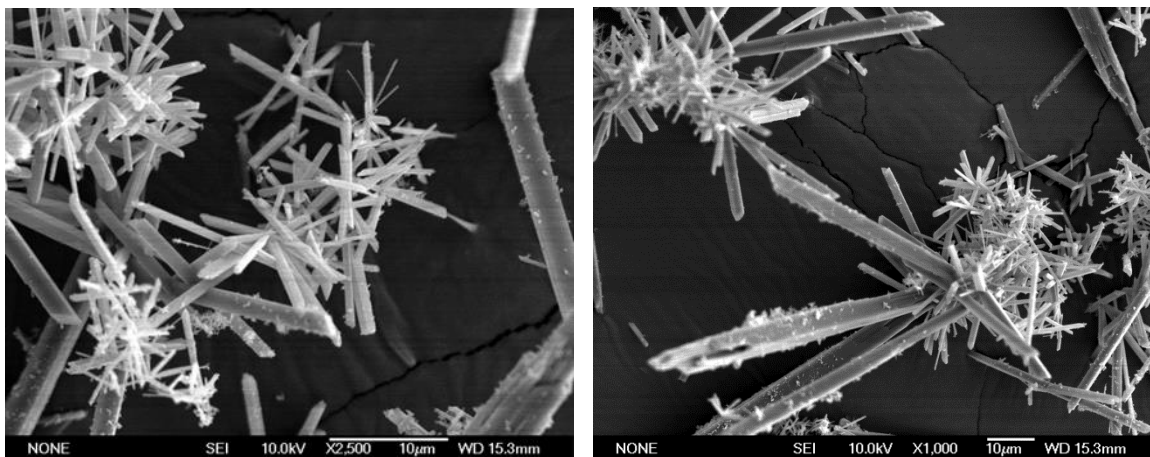


Figure S3. SEM images of the sample prepared by SAS at 313 K and 10 MPa using methanol as a solvent.

Table S2. Microelemental analysis of the samples prepared by evaporation and SAS at the different conditions.

Adduct	%C ±0.35	%H ±0.30	%N ±0.30	C:N ±0.35
Cu ₂ (C ₃) ₄ (INA) ₄ Theoretical	47.63	4.88	12.34	4.50
Evaporation	47.45	4.85	12.22	4.53
SAS at 313 K and 10 MPa EtOH - Cu(C ₃) ₂ :INA 1:2	43.75	4.26	13.17	3.88
SAS at 298 K and 8 MPa EtOH - Cu(C ₃) ₂ :INA 1:2	43.19	4.37	12.29	4.10
SAS at 313 K and 10 MPa MeOH - Cu(C ₃) ₂ :INA 1:2	43.30	4.16	12.61	4.01
SAS at 298 K and 10 MPa EtOH - Cu(C ₃) ₂ :INA 1:2	32.11	3.47	10.58	3.54
SAS at 313 K and 25 MPa EtOH - Cu(C ₃) ₂ :INA 1:2	30.79	3.59	9.24	3.89
SAS at 313 K and 10 MPa EtOH - Cu(C ₃) ₂ :INA 1:1	26.47	3.35	7.88	3.92