Electronic Supplementary Material (ESI) for CrystEngComm.

High-Pressure, High-Temperature Synthesis of Nanostructured Polydiphenyldibutadiyne Confined in the 1-Dimensional Pores of Single Crystal $AlPO_4$-54†

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Rietveld refinement of the DPB-filled VFI

Table S1. Atomic coordinates and isotropic displacement parameters from the Rietveld refinement of DPB–filled $AlPO_4$-54 ($P6_3$, a=18.9951(4)Å, c=8.133(1)Å, R_p=4.0%, R_{wp}=5.5%, R_{Bragg}=2.1%). Ow=oxygen from adsorbed H_2O molelcules.

Atom	x	y	z	U (Å^2)	occupancy
Al1	0.3847(4)	-0.0011(4)	0.2386(8)	0.00893	1.00000
Al2	0.4744(4)	-0.1727(4)	0.2042(9)	0.00893	1.00000
Al3	0.6541(4)	0.1691(4)	0.1980(10)	0.00893	1.00000
P1	0.5499(4)	-0.0008(5)	0.3583(9)	0.00893	1.00000
P2	0.3113(4)	-0.1848(4)	0.3203(9)	0.00893	1.00000
P3	0.5067(4)	0.1845(4)	0.3096(9)	0.00893	1.00000
O1	0.4700(3)	0.0009(4)	0.3386(8)	0.00893	1.00000
O2	0.4309(4)	0.0088(4)	0.0370(5)	0.00893	1.00000
O3	0.3218(4)	-0.1120(2)	0.2232(8)	0.00893	1.00000
O4	0.4302(3)	0.1109(3)	0.2548(8)	0.00893	1.00000
O5	0.5404(4)	-0.0738(3)	0.2642(10)	0.00893	1.00000
O6	0.6190(4)	0.0718(3)	0.2775(10)	0.00893	1.00000
O7	0.5120(4)	-0.2310(3)	0.2805(10)	0.00893	1.00000
O8	0.3781(3)	-0.2058(4)	0.2794(10)	0.00893	1.00000
O9	0.4747(5)	-0.1749(5)	-0.0146(5)	0.00893	1.00000
O10	0.3112(5)	-0.1680(5)	0.5000(5)	0.00893	1.00000
O11	0.5752(4)	0.1952(5)	0.1990(9)	0.00893	1.00000
O12	0.7393(3)	0.2358(3)	0.3148(9)	0.00893	1.00000
H_2O1	0.3391(4)	0.0050(5)	0.4650(7)	0.00893	1.00000
H_2O2	0.2903(3)	0.0024(4)	0.1596(7)	0.00893	1.00000
Ow1	0.1125(15)	0.3108(13)	0.908(3)	0.00893	1.00000
Ow2	0.3114(15)	0.1274(18)	0.908(3)	0.00893	1.00000
Ow3	-0.0440(3)	0.1652(3)	0.4176(9)	0.00893	1.00000
C1	0.09790	0.06025	-0.1611	0.00893	0.493(6)
C2	0.09790	0.06025	0.08895	0.00893	0.493(6)
C3	0.00000	0.12400	-0.0030	0.00893	0.493(6)

Focal Plane Array (FPA) imaging analysis

The Quasar software has been used (http://quasar.codes)[1, 2]. Spectra from gasket hole were selected by clustering with the K-means method and the spectral region of interested was cut. Baseline subtraction was performed with a rubber band method. Spectra with saturated peaks

were removed (the grey pixels inside the hole in the following images). Hyperspectral images of each region were generated by calculating the spectral integral and cluster analysis was done with the K-means method. Spectral intensities were normalized only in the unsaturated CH region 2980- 3170 cm^{-1} through a vector normalization. The other spectra were not normalized since there are areas where the peaks of interest are present and others not, so we would lose this information with a normalization. The various steps of the analysis in the different spectral ranges are displayed in Figures S1-S4. Smoothed images are shown in the TOC graphic.

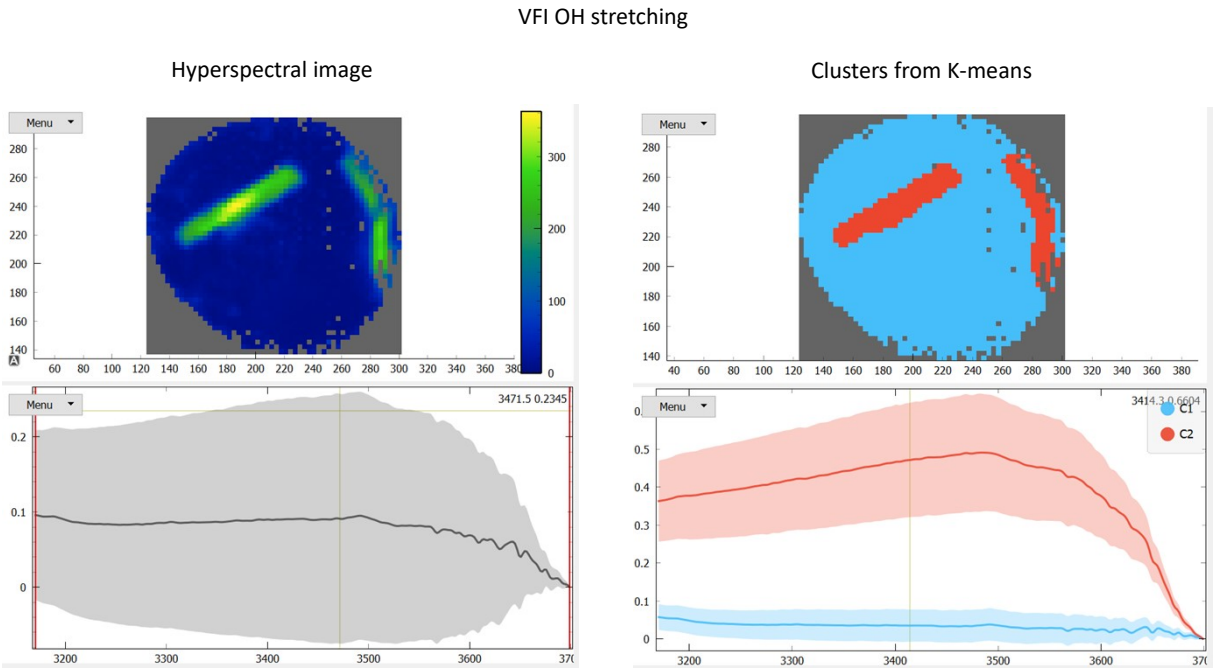

Figure S1. Hyperspectral image and clusters from K-means in the OH stretching region.

Saturated CH

Hyperspectral image

Clusters from K-means

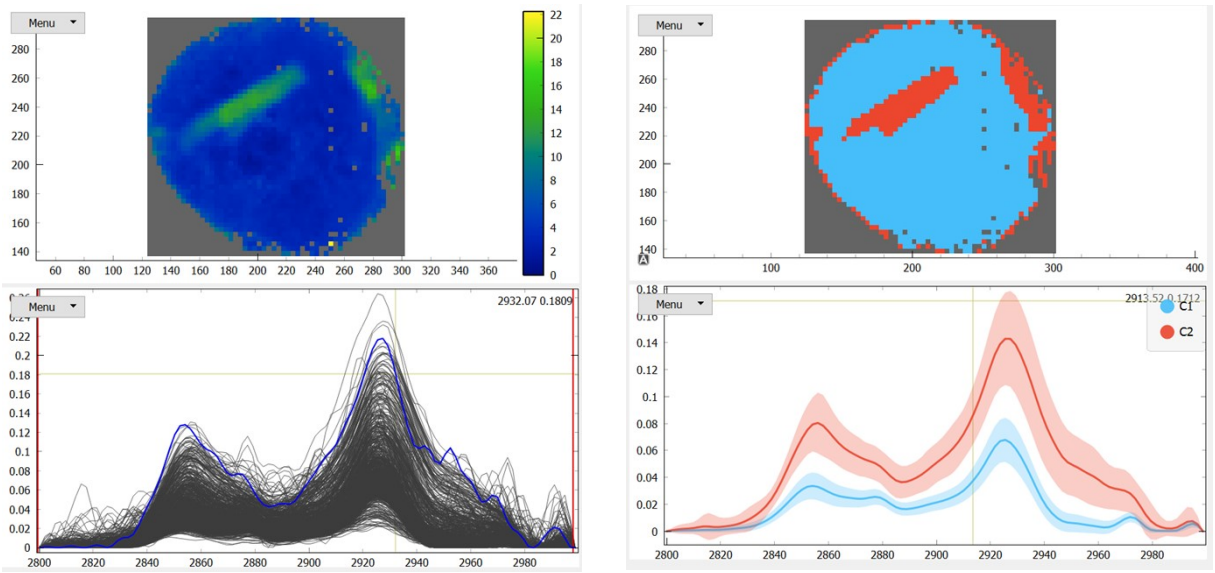

Figure S2. Hyperspectral image and clusters from K-means in the saturated CH stretching region.

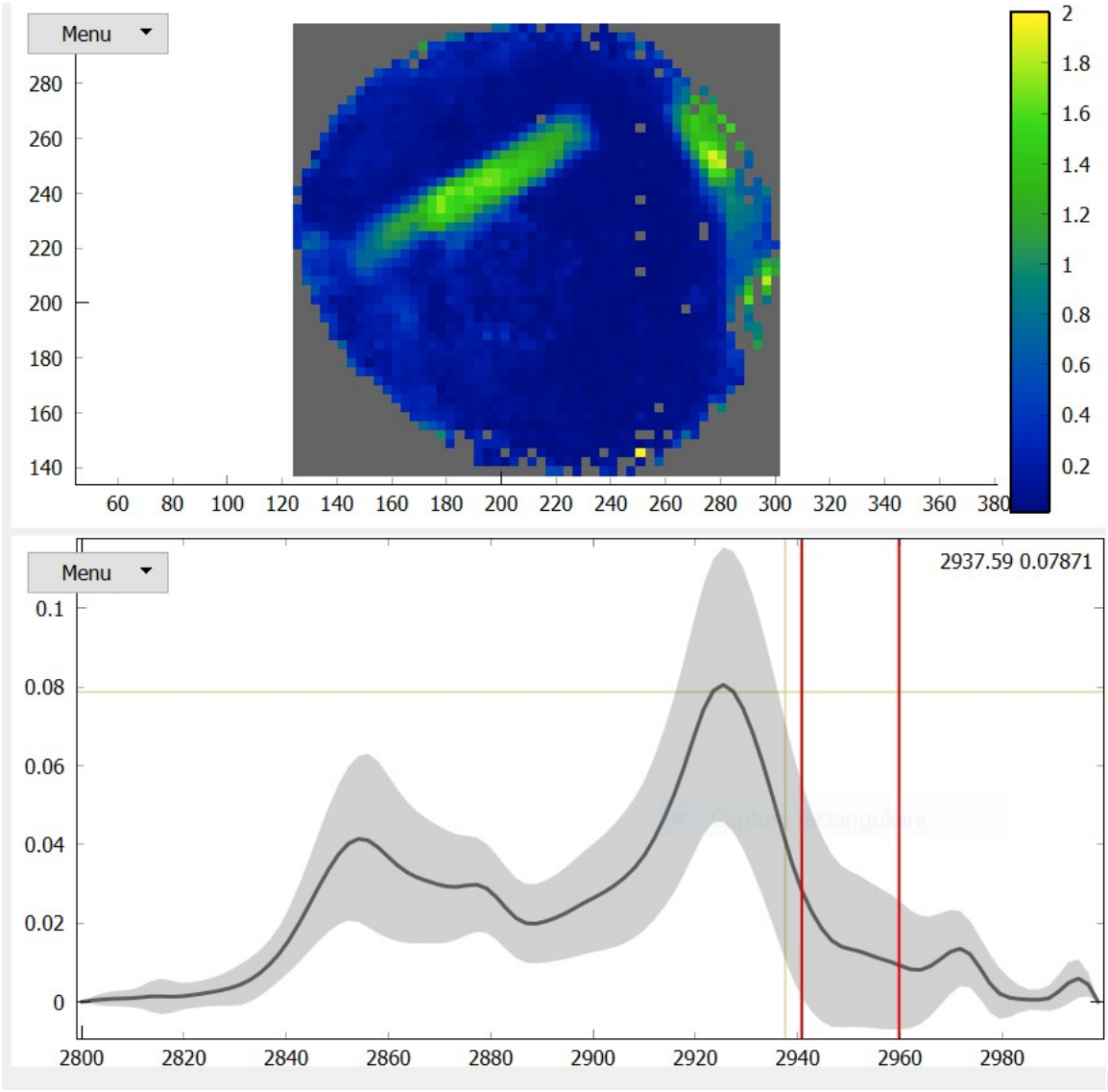

Figure S3. Hyperspectral image and clusters from K-means in the CH stretching region from 2940-2960 cm^{-1}. The spectral contrast is better and thus the spectral weight in this range is mostly due to the polymer inside the VFI.

Unsaturated CH

Hyperspectral image

Clusters from K-means

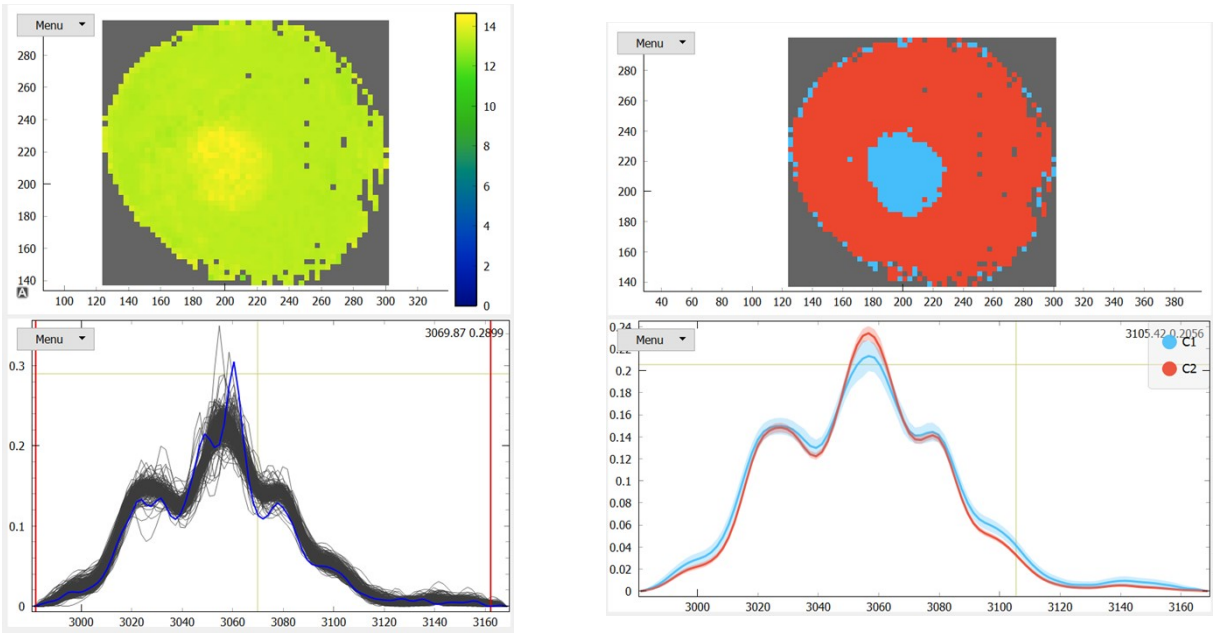

Figure S4. Hyperspectral image and clusters from K-means in the unsaturated CH stretching region. This is a kind of control. It nicely shows that the unsaturated CH are evenly distributed inside the gasket hole, even where the VFI crystals are. The cluster analysis shows a clear hole that is actually found in all the images. It correspond most likely to a region slightly burned with the laser during the ruby pressure measurements. It corresponds to lower-intensity and noisy IR spectra.

1. M. Toplak, G. Birarda, S. Read, C. Sandt, S. M. Rosendahl, L. Vaccari, J. Demšar and F. Borondics, *Synchrotron Radiation News*, 2017, **30**, 40-45.
2. M. Toplak, S. T. Read, C. Sandt and F. Borondics, *Cells-Basel*, 2021, **10**, 2300.