Liquid-assisted vortex grinding supports the single-step solid-state construction of a [2.2]paracyclophane

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SUPPLEMENTARY INFORMATION

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1. Experimental details

1.1 General remarks

^{1}H NMR spectra were collected using a Bruker 300 MHz spectrometer and DMSO-d_6 as solvent.

Powder X-ray diffraction (XRD) data were collected on a Bruker D-5000 diffractometer equipped with a Bruker SOL-X energy-sensitive detector using Cu K_α radiation (λ=1.54056 Å).

Vortex grinding was performed using a VWR vortex mixer type Vortex Genie 2. Borosilicate glass vials of dimensions 21 x 70 mm were used as sample vials along with premium grade steel BBs (5 mm diameter) washed with soap, water, and acetone.

All photoreactions were carried out in a photoreactor chamber equipped with a broadband, low-pressure UV lamp with a quartz Hg arc, Hanovia lamp, model number PC451.050 and ACE Glass Inc. Power supply: 230 V, 50 Hz, 450 W.

1.2 Manual grinding

In a typical dry grinding experiment 50 mg of **1,4-bpeb** and 35 mg of **4-benz-res** (1:1 molar ratio) were placed in an agat mortar-and-pestle and ground for up to 1 h.

In a typical LAG experiment 50 mg of **1,4-bpeb** and 35 mg of **4-benz-res** (1:1 molar ratio), and a single drop of methanol (50 μl) were placed in an agat mortar-and-pestle and ground for up to 1 h.

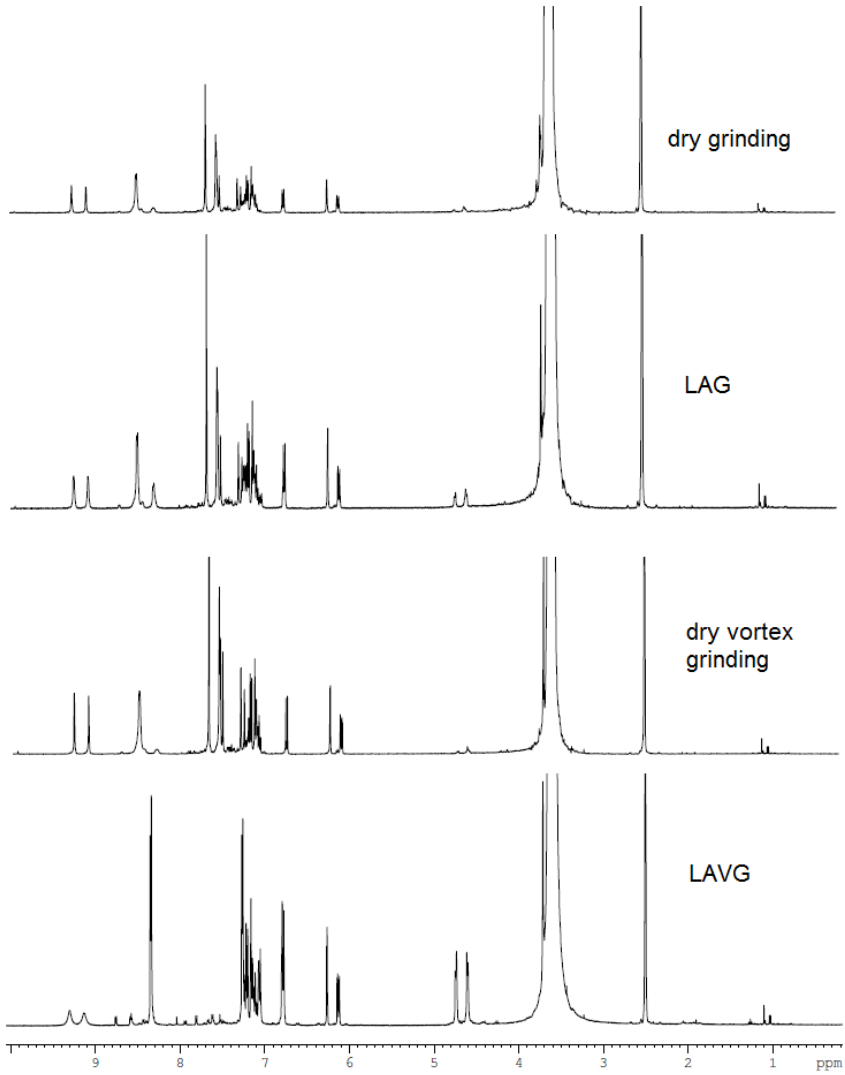
1.3 Vortex grinding

In a typical dry vortex experiment 50 mg of **1,4-bpeb** and 35 mg of **4-benz-res** (1:1 molar ratio)) were placed in a borosilicate glass vial (dimensions of the vial were 21 x 70 mm) with two premium grade steel BBs (5 mm diameter). Prior to use, BBs were washed with soap, water and acetone and dried. The capped vial was mounted atop of the vortex mixer and secured with the test tube holder attached to the ring stand rod. Upon initiation of the vortex mixer, the vial was vigorously shaken using the vortex mixer. Motion of the vial was transmitted to the BBs which exhibited rapid, chaotic movement and collisions.

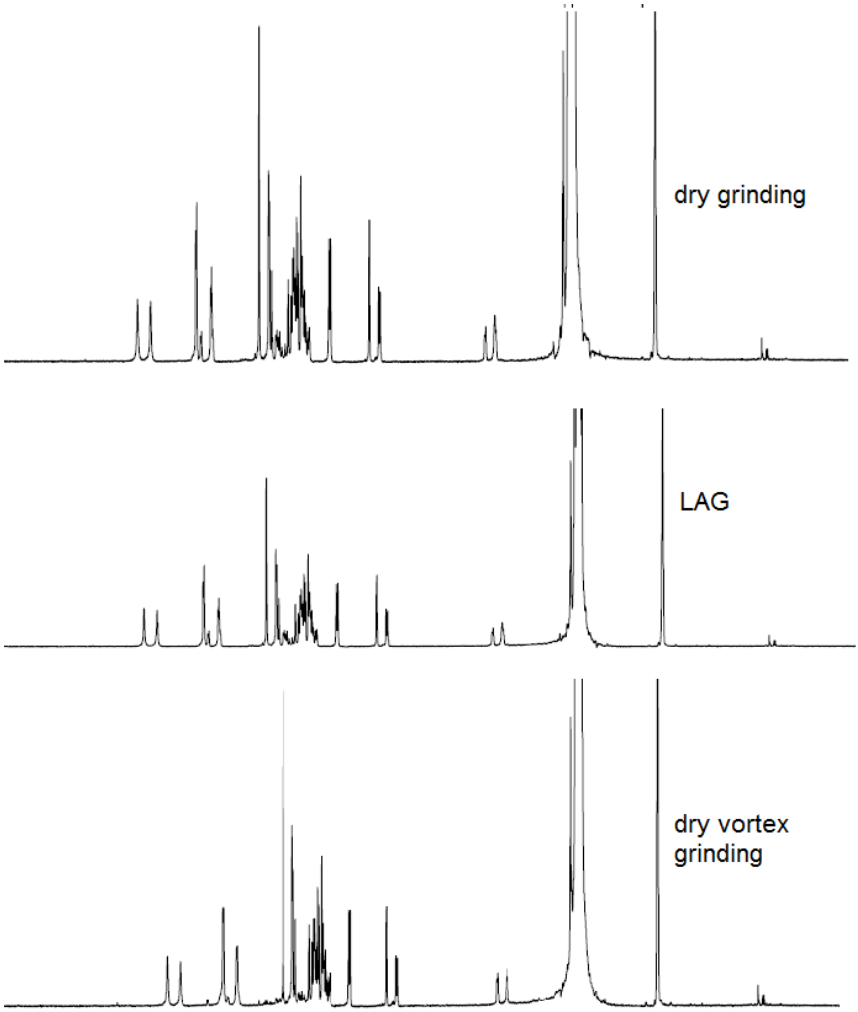
In a typical LAVG experiment, virutally the same procedure was followed as in the vortex grindig experiment, with addition of a single drop of methanol or nitromethane (50 μl) in the vial.

2. ^{1}H NMR spectroscopy

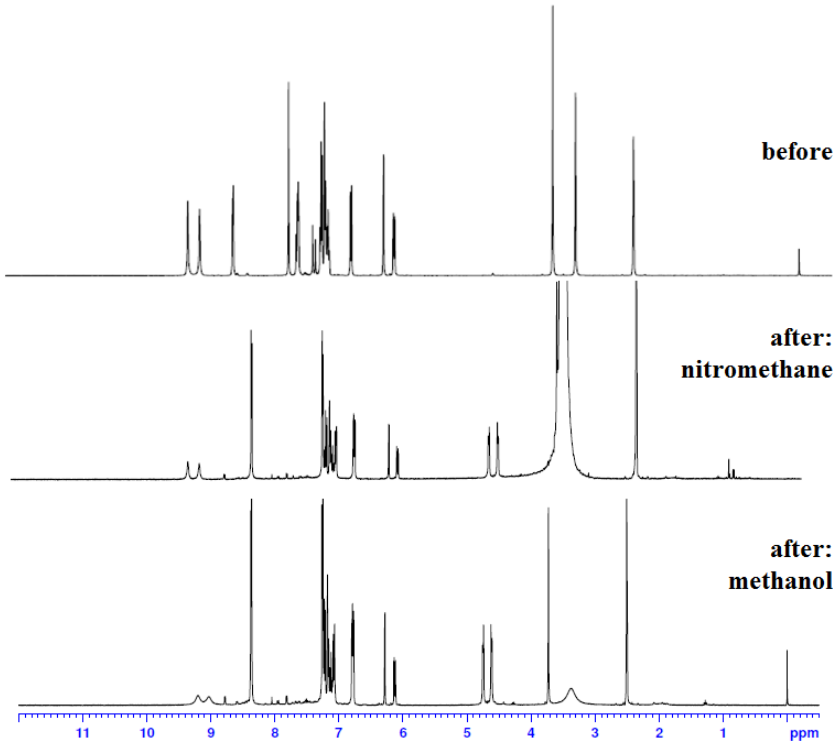
2. 1 ^{1}H NMR data after 20 h of UV radiation



2. 2 ^{1}H NMR data after 60 h of UV radiation.

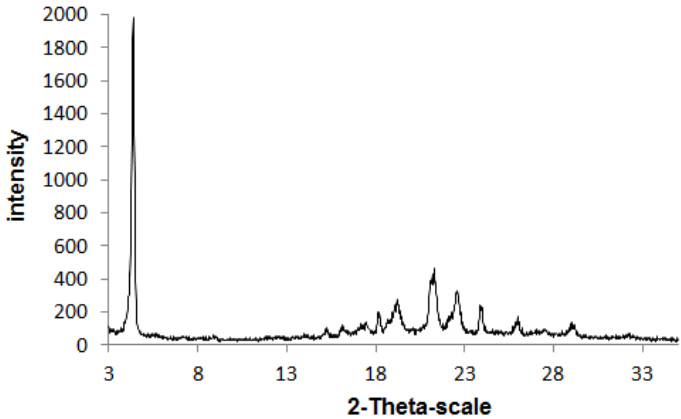


2. 3 ^{1}H NMR data before and after 10 h of simultaneous LAVG and UV radiation.

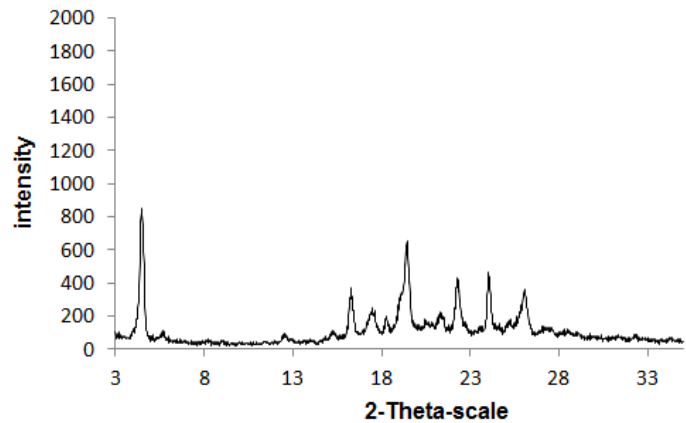


3. Powder X-ray diffraction

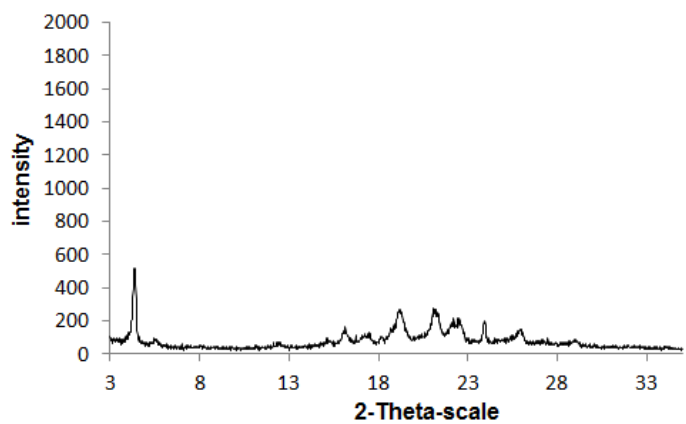
3. 1 X-ray powder pattern upon 1 h of dry grinding



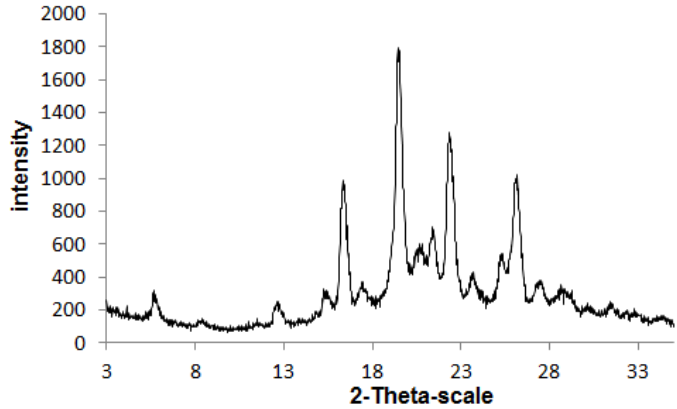
3. 2 X-ray powder pattern upon 1 h of LAG



3. 3 X-ray powder pattern upon 1 h of dry vortex grinding



3. 4 X-ray powder pattern upon 1 h of LAVG



3. 5 Comparison of X-ray powder patterns obtained after 10 h of simultaneous UV-radiation and LAVG, and cocrystals grown from solution after 20 h of UV-radiation