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Supporting information for article:

Four interpenetrating hydrogen-bonding three-dimensional networks in divanillin (6,6′-dihydroxy-5,5′-dimethoxy-[1,1′-biphenyl]-3,3′-dicarbaldehyde)

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S1. NMR and EA results

1H-RMN (400Hz, DMSO-d6): δ=3.93 (s, 6H); 7.43 (s, 4H); 9.81 (s, 1H).

13C-RMN (DMSO-d6, 100 MHz): δ=56.0 (CH3O); 56.5 (CH3O); 109.5 (Ar); 111.1 (Ar); 115.8 (Ar); 125.0 (Ar); 126.6 (Ar); 128.1 (Ar); 128.7 (Ar); 129.1 (Ar); 148.7 (Ar); 151.0 (Ar); 153.5 (Ar); 191.1 (CHO); 191.2 (CHO).

C and H analyses were carried out with a Thermo Scientific Flash 2000 elemental analyzer.

Anal. Calc. for $C_{16}H_{14}O_6$ (%) C, 63.57; H, 4.67. Found for 5 min sample: C, 62.24; H, 4.39. Found for 15 min sample: C, 62.74; H, 4.59

S2. Powder diffraction data collection conditions

Powder diffraction data collection was performed with a Rigaku ULTIMA IV diffractometer in θ-θ geometry with diffractometer radius 285 mm. Radiation form a water-cooled Cu-anode sealed tube provided CuKα (λ_{ave}=1.5418 Å) X-rays that were monochromated with a diffracted beam curved Ge(111) crystal monochromator.

The short patterns in Fig. S1 were collected in the 5 to 50° range with a step of 0.04° in 2θ and 2 s/step counting time.

The high-quality pattern used for structural determination and refinement was obtained in the 8 to 108° 2θ range with a step of 0.01° in 2θ and 20 s/step counting time.

S3. Scanning electron microscopy image collection conditions

SEM images were obtained with a Jeol JSM 59900-LV instrument.

Table S1 Fractional atomic coordinates and isotropic displacement parameters of the Rietveld-refined divanillin structure. Standard uncertainties are shown in parenthesis.

	x	y	z	Uiso (Å^2)
C1	0.27806(12)	0.41290(11)	0.6785(6)	0.0470(13)
C2	0.21080(10)	0.48780(18)	0.5520(10)	0.0493(11)
C3	0.26177(13)	0.56830(16)	0.4107(11)	0.0598(14)
C4	0.37574(11)	0.57070(11)	0.4036(7)	0.0607(14)
C5	0.44038(11)	0.49460(17)	0.5	0.0375(12)
C6	0.38971(11)	0.41765(11)	0.6453(6)	0.0532(13)
C7	0.23099(9)	0.32760(10)	0.8444(7)	0.0808(14)
C8	0.08772(11)	0.64343(9)	0.33827(34)	0.0567(9)
O1	0.13485(8)	0.31375(11)	0.8931(7)	0.0784(9)

O2	0.20440(9)	0.64551(12)	0.2917(7)	0.0663(8)
O3	0.42399(10)	0.64420(7)	0.2402(5)	0.0707(7)
H2	0.13565(13)	0.4859(6)	0.5785(23)	0.0592(14)
H3	0.4013(4)	0.70692(13)	0.2997(16)	0.0848(9)
H6	0.43316(23)	0.37013(17)	0.7397(10)	0.0638(16)
H7	0.27949(21)	0.27727(16)	0.9036(17)	0.0969(17)
H8A	0.0593(3)	0.70318(14)	0.2656(12)	.0681(11)
H8B	0.0718(4)	0.6328(5)	0.5720(4)	0.0681(11)
H8C	0.0553(3)	0.59394(16)	0.2069(8)	0.0681(11)

Table S2 Fractional atomic coordinates of the divanillin structure obtained from DFT-D calculations.

	x	y	z
C1	0.279541	0.406423	0.679591
C2	0.212317	0.483522	0.572461
C3	0.258312	0.565034	0.425705
C4	0.373290	0.571940	0.382718
C5	0.440226	0.495466	0.499045
C6	0.391929	0.413605	0.641167
C7	0.239698	0.320389	0.846470
C8	0.086619	0.643572	0.362250
O1	0.143854	0.302742	0.925752
O2	0.201718	0.644328	0.314654
O3	0.420908	0.648623	0.238364
H2	0.125239	0.477093	0.607862
H3	0.373866	0.702017	0.138414
H6	0.443330	0.355508	0.737337
H7	0.302875	0.266482	0.910388
H8A	0.056914	0.713511	0.269031
H8B	0.065122	0.636221	0.632785
H8C	0.048534	0.584483	0.216034

Figure S1 X-ray powder diffraction patterns of divanillin samples after 5 min and 15 min reaction time.

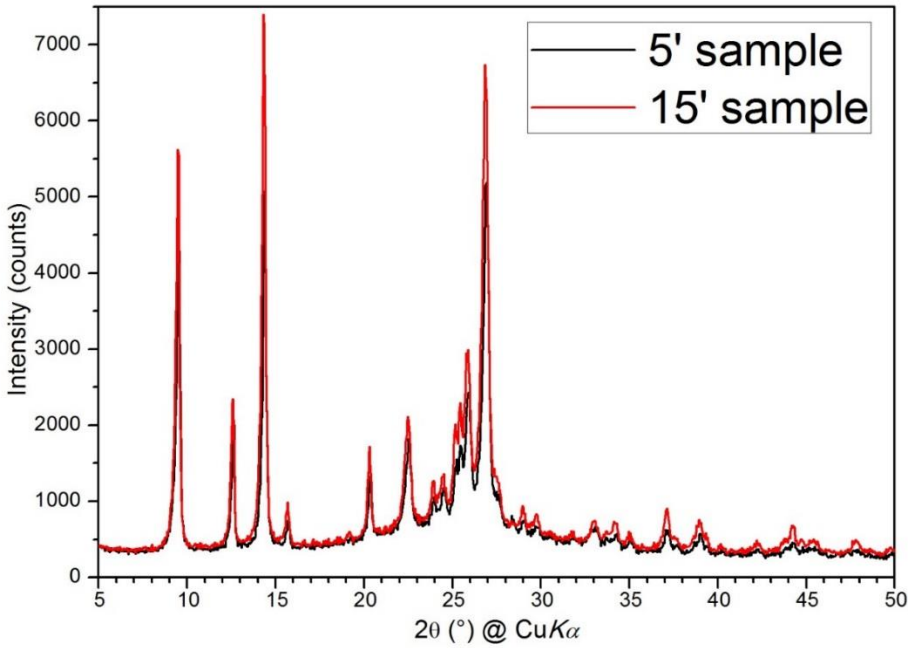

Figure S2 Rietveld fit of divanillin model. The black × symbols represent the observed data, the red line is the calculated diffraction profile, and the green line the background intensity. The blue line at the bottom is the difference between observed and calculated diffraction data. Pink vertical symbols indicate diffraction peak positions.

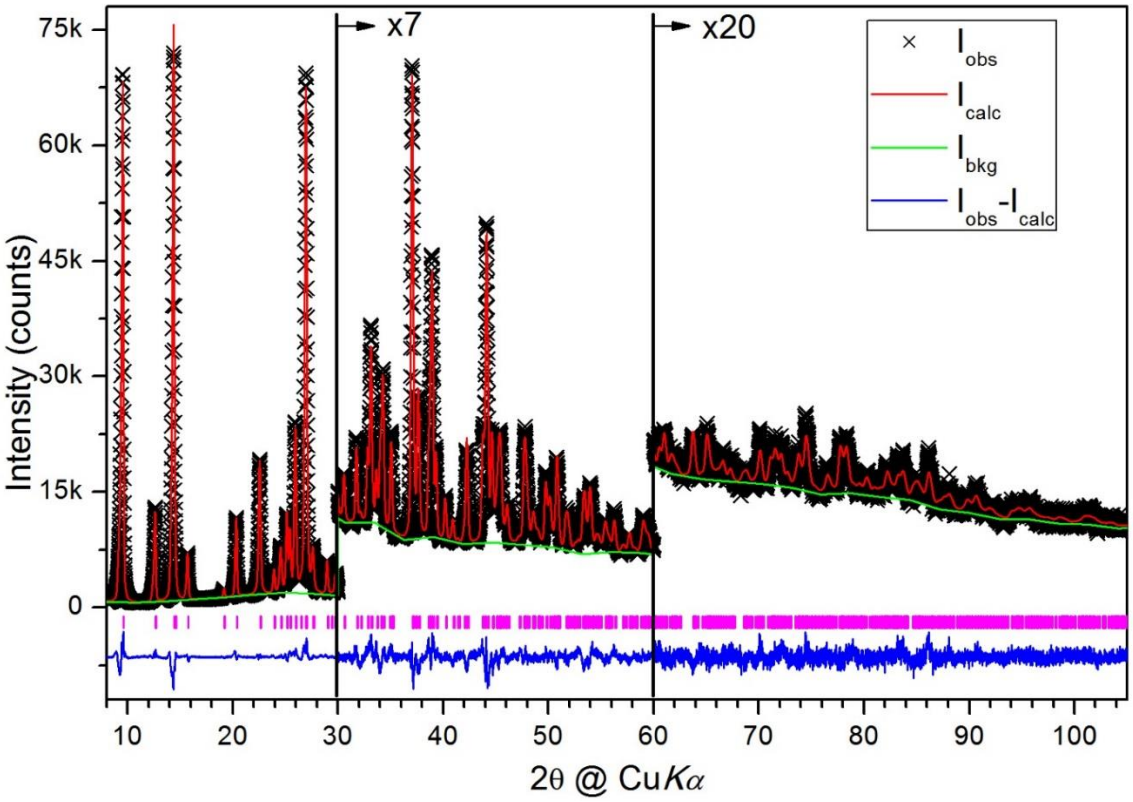

Figure S3 Overlay of the DFT-D (blue) and refined (red) crystallographic models for divanillin.

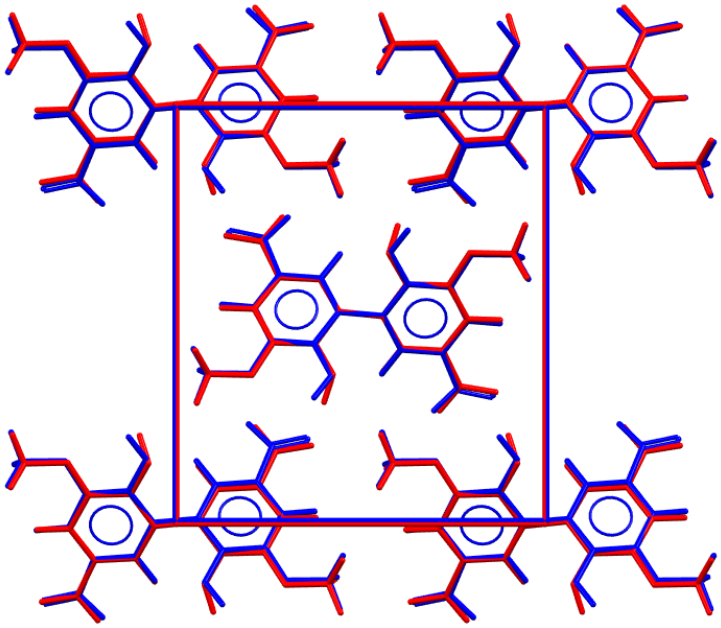

Figure S4 Scanning Electron Images of divanillin prepared using 5 min reaction time at (a-top left) x5000, and (b-top right) x12000 magnifications, showing long needle crystals with some curved edges; and prepared using 15 min reaction time at (c-bottom left) x5000 and (d-bottom right) x12000 magnifications, showing plate crystals.