Supporting Information

Solid-state ^{1}H, ^{13}C, ^{17}O NMR characterization of the two uncommon polymorphs of curcumin

Yizhe Dai,[1] Victor Terskikh,[1,2] Andreas Brinmkmann,[2] and Gang Wu[1]*

[1]Department of Chemistry, Queen's University, 90 Bader Lane, Kingston, Ontario,
Canada K7L 3N6;
[2]Metrology, National Research Council Canada, 1200 Montreal Road, Ottawa, Canada
K1A 0R6.

*Corresponding author: wugang@queensu.ca

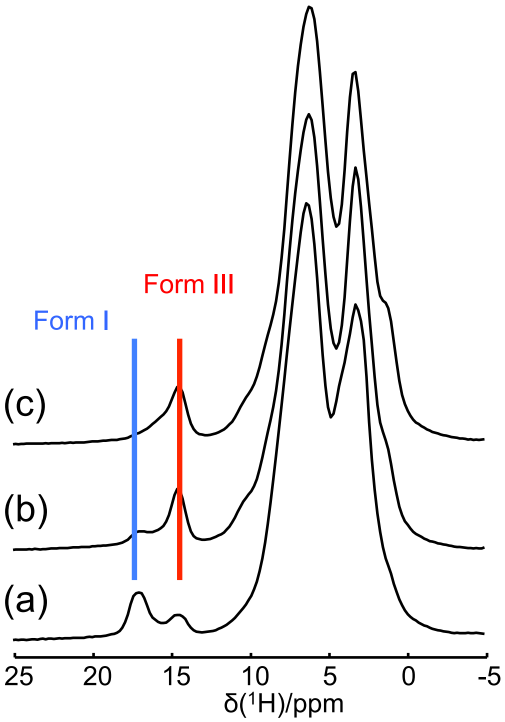


Figure S1. ^{1}H MAS NMR spectra of curcumin samples at different stages of the preparation for form III. (a) After quickly drying from a MeOH solution with a stream of N_2(g). (b) After another dissolution/drying cycle from (a). (c) Two more dissolution/drying cycles from (b). All ^{1}H MAS spectra were recorded at 16.4 T (700 MHz for ^{1}H) with a sample spinning frequency of 30 kHz. The sample temperature was controlled at 293 K.

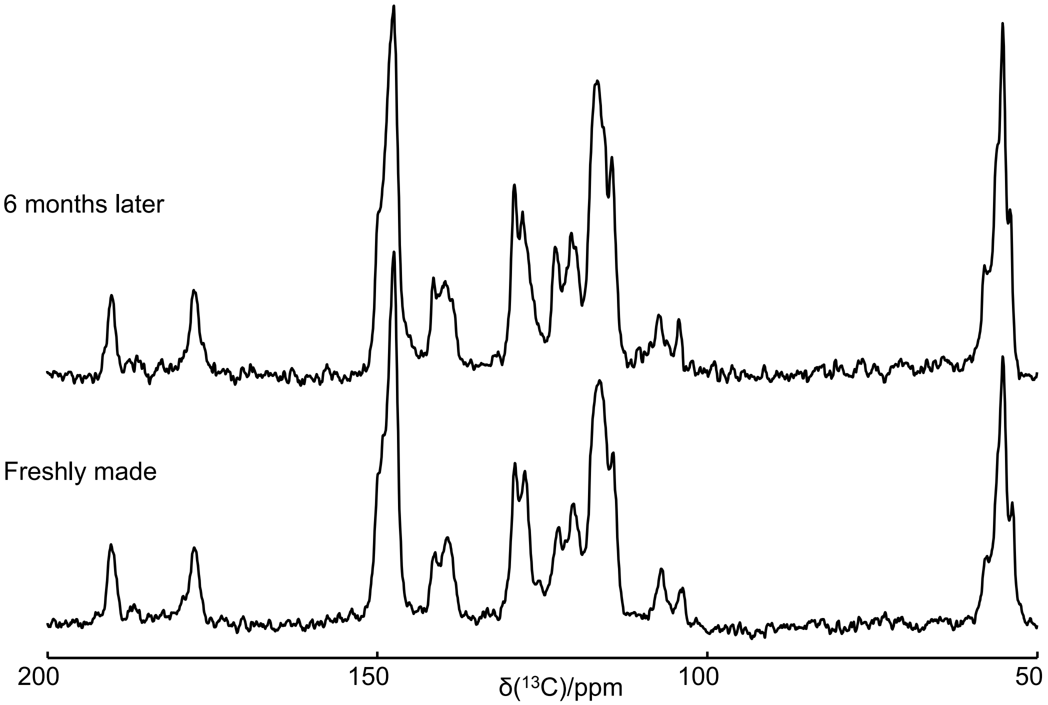


Figure S2. ^{13}C CP/MAS NMR spectra showing the stability of the form-III curcumin crystals. All spectra were recorded at 16.4 T (700 MHz for ^{1}H and 176 MHz for ^{13}C) with a sample spinning frequency of 20 kHz. The sample temperature was controlled at 293 K.

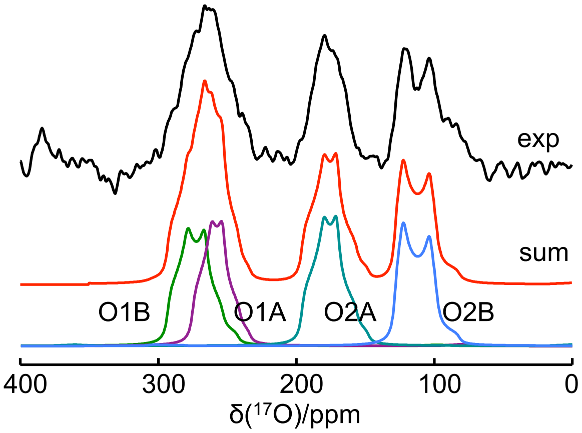


Figure S3. Experimental (black traces) and simulated (colored traces) ^{17}O MAS NMR spectra of a form-II curcumin sample recorded at 21.1 T (900 MHz for ^{1}H and 122 MHz for ^{17}O) with a sample spinning frequency of 22 kHz. The sample temperature was controlled at 293 K.

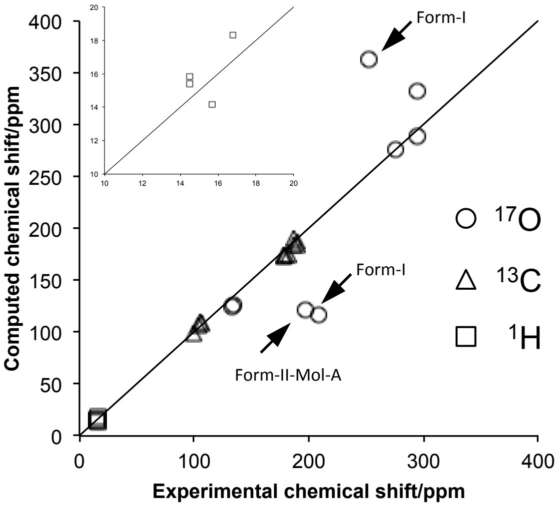
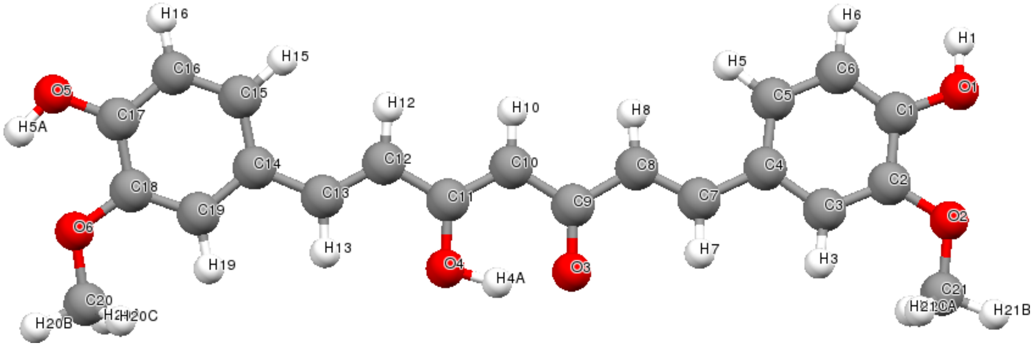


Figure S4. Comparison between experimental and computed ^{1}H, ^{13}C, and ^{17}O chemical shifts for the three polymorphs of curcumin. The diagonal line is shown to aid for visual inspection of data. The inset shows the expanded region around the ^{1}H chemical shift data points. All calculations were done with dispersion corrected DFT-D2. Data points that deviate from the diagonal line are highlighted.

Table S1. Complete list of computed ^{13}C and ^{1}H chemical shifts for curcumin in Form III.



Atom	CASTEP	σ_{iso}/ppm	δ_{iso}/ppm
C1	C15	16.71	153.7
C2	C3	19.26	151.1
C3	C2	51.79	118.6
C4	C1	43.31	127.1
C5	C14	50.21	120.2
C6	C16	50.55	119.9
C7	C7	27.40	143.0
C8	C5	43.47	126.9
C9	C9	-15.56	186.0
C10	C4	59.67	110.7
C11	C6	-7.29	177.7
C12	C8	50.16	120.2
C13	C11	26.67	143.7
C14	C10	40.59	129.8
C15	C12	51.58	118.8
C16	C21	54.84	115.6
C17	C13	17.20	153.2
C18	C19	19.61	150.8
C19	C18	53.98	116.4
C20	C20	112.42	68.0
C21	C17	113.23	57.2
H1	H19	19.8	9.2
H3	H1	24.29	4.7
H4A	H18	13.54	15.5
H5	H8	23.72	5.3
H5A	H20	22.90	6.1
H6	H9	23.73	5.3
H7	H4	24.57	4.4

H8	H3	24.75	4.3
H10	H2	25.09	3.9
H12	H5	23.81	5.2
H13	H6	24.02	5.0
H15	H7	23.41	5.6
H16	H17	23.45	5.6
H19	H13	24.59	4.4
H20A	H14	26.54	2.5
H20B	H15	26.45	2.6
H20C	H16	27.46	1.5
H21A	H10	25.95	3.1
H21B	H11	26.97	2.0
H21C	H12	28.16	0.8