Polymorphism in acesulfame sweetener: Structure-property and stability relationships of bending and brittle crystals

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## Electronic Supplementary Information (ESI)
(10 pages)

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S1. Neutralization of Acesulfame potassium (Ace K).

It was obtained from Sigma Aldrich at a stated purity of 99% and no attempt was made at further purification. Ace K (5 g) was dissolved in water (5 mL), neutralized with concentrated HCl (5 mL) and achieved the highly acidic solution of ~ pH =2 and extracted with ethyl acetate (15 mL). Up on routine work up afforded a white solid of salt free acesulfame. It was crystallized from EtOAc by slow evaporation at the ambient conditions. Needle crystals were yielded. Mp: 122-124 °C.
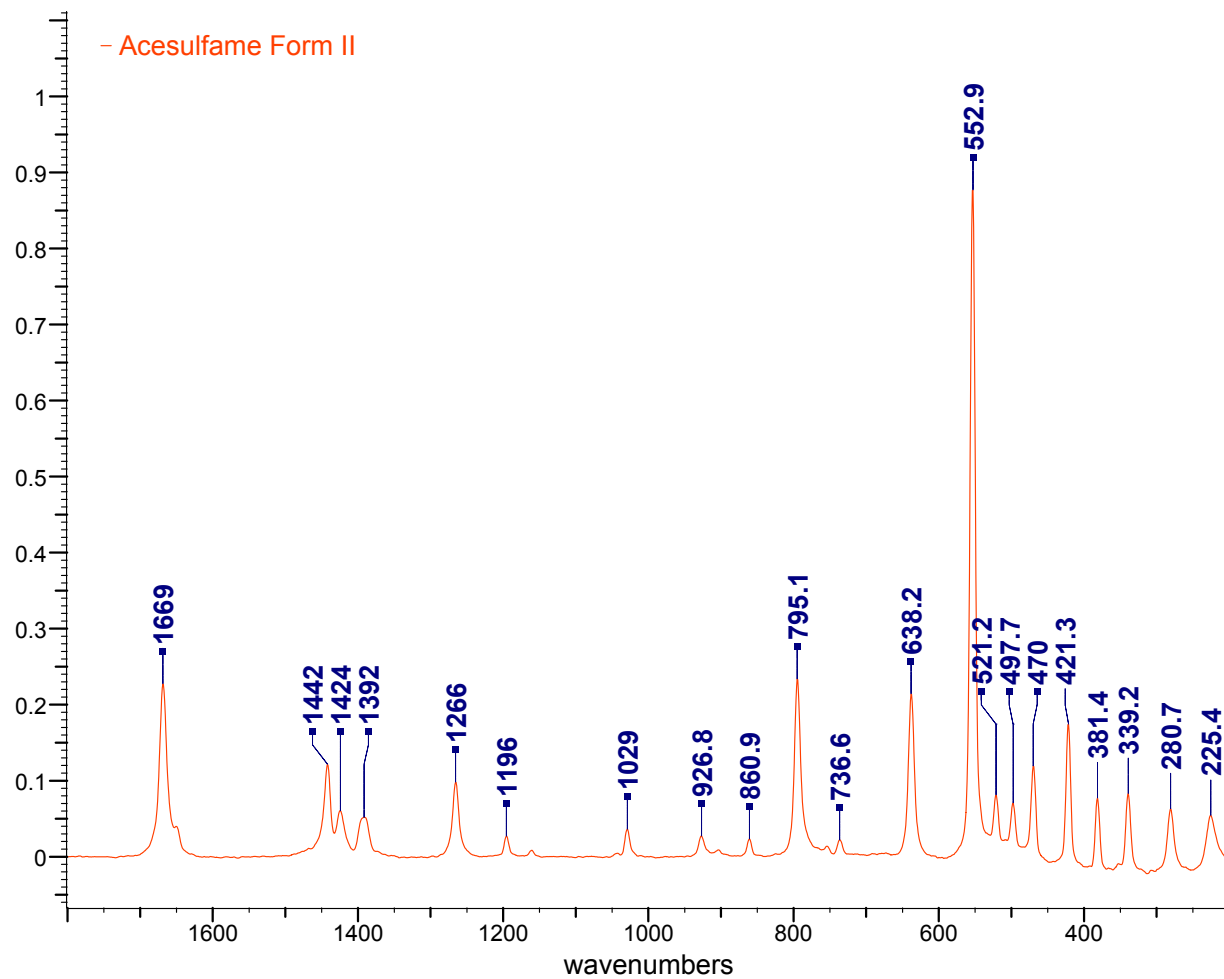
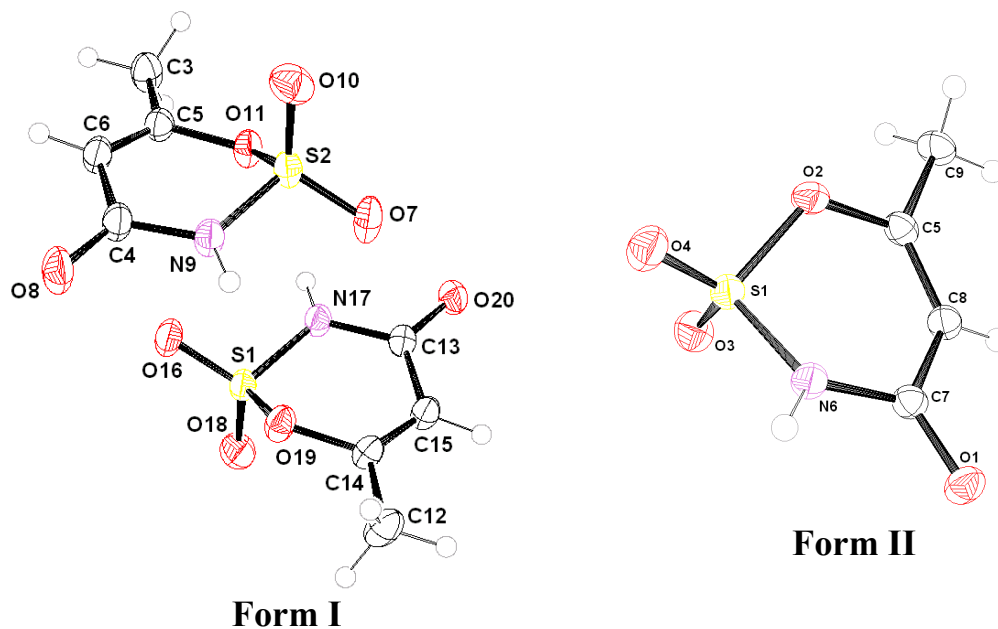**S2** DSC and TGA for the needle and the prismatic crystals.
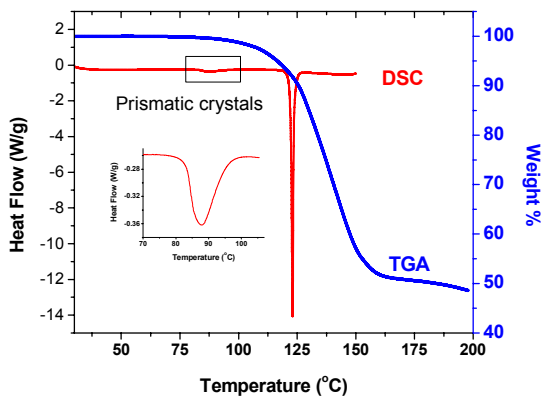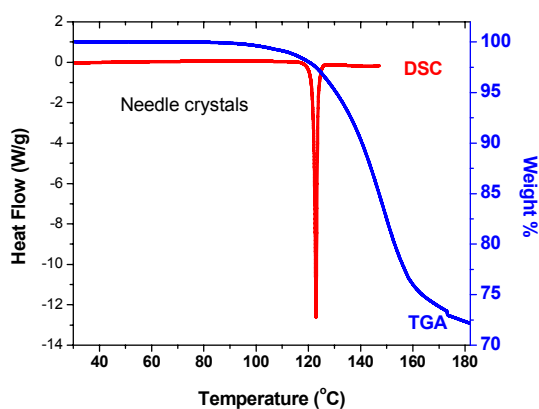
**S3a.** Raman vibrational spectroscopy for acesulfame Form I.
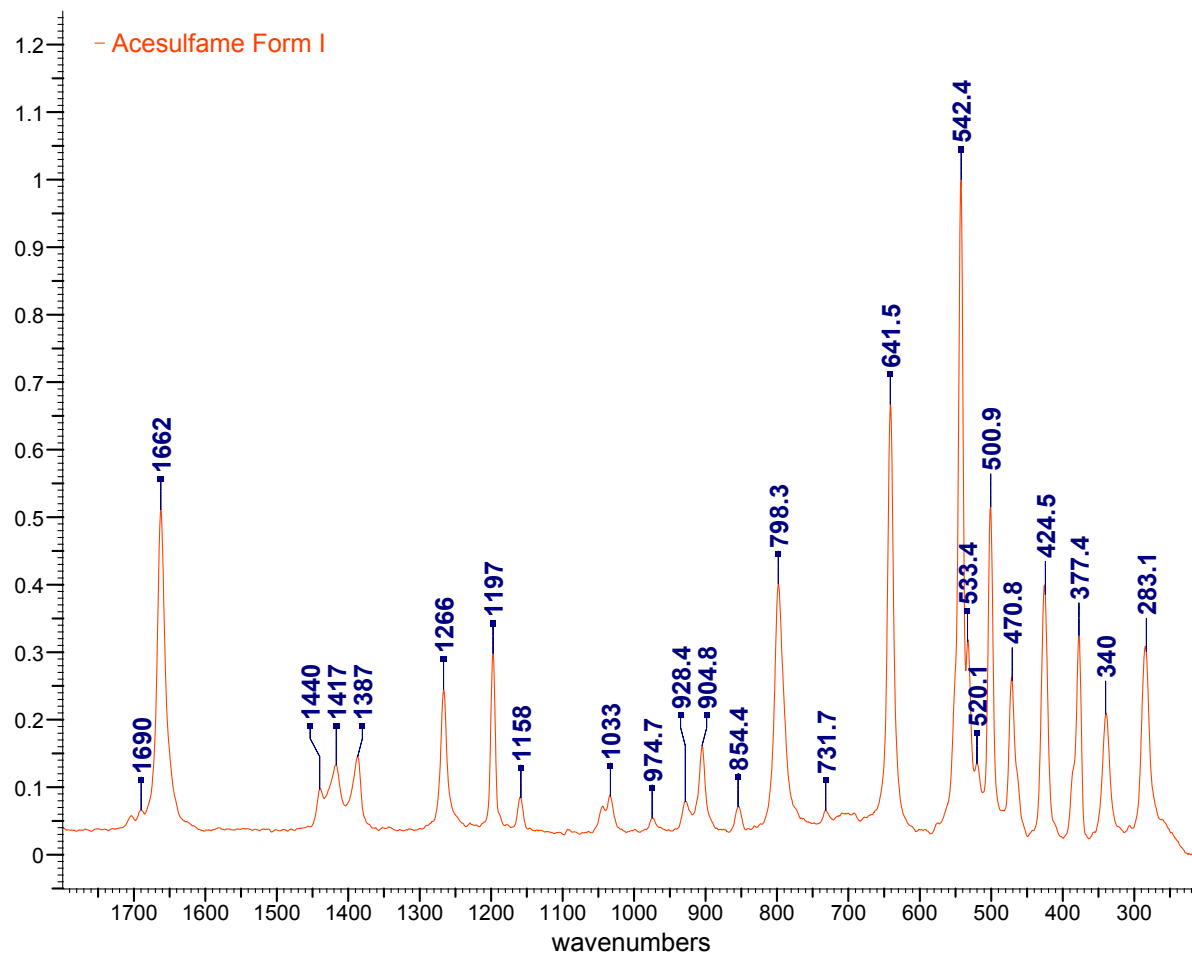
S3b Raman vibrational spectroscopy for acesulfame Form II.

S4a ORTEP plots at the 50% probability level. Two and one independent molecules in the asymmetric unit of Forms **I** and **II** respectively.

Form I

Form II

S4b Molecular conformations of acesulfame Forms I and II. While Form I adopts two different conformations (Blue and Green), Form II conformation (pink) was similar to one of the indpentent molecules of Form I (Blue).

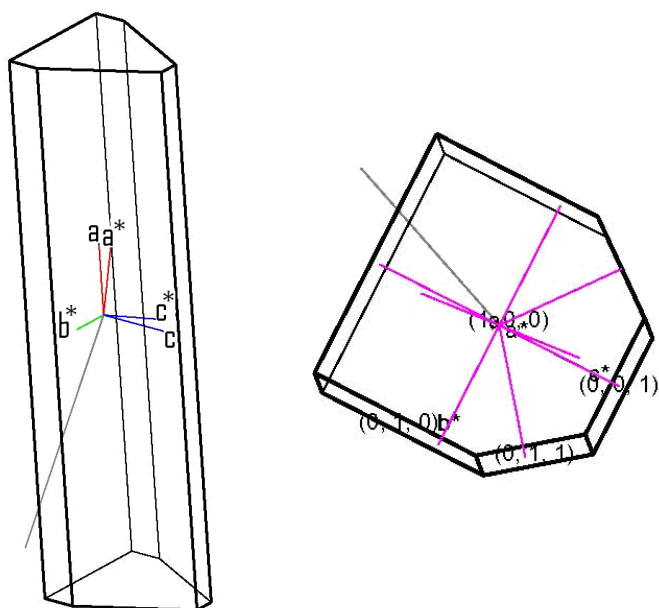Form **I**   Form **II**
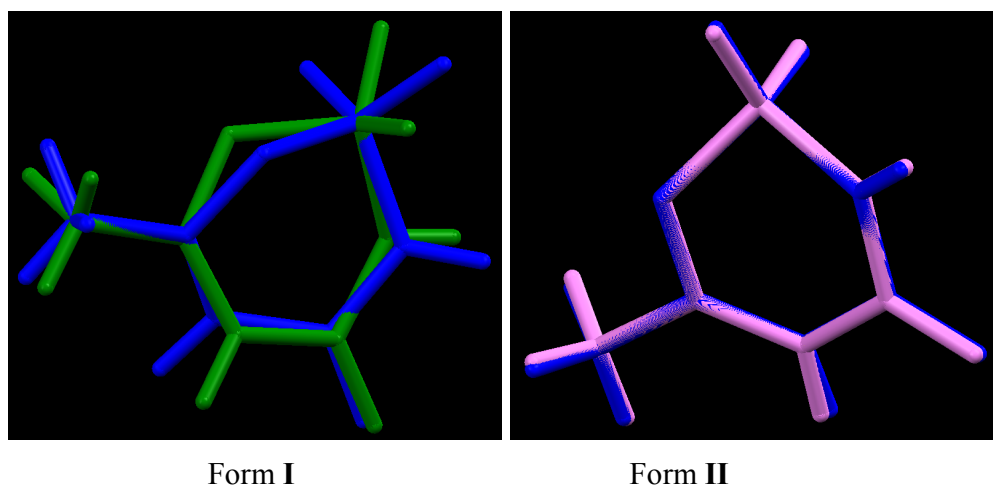
S4c Hydrogen bond geometries for Forms I and II.[a]

D–H···A	d(H···A)/ Å	d(D···A)/ Å	∠(D–H···A)/ °	Symmetry code
Form I				
N–H···O	1.74	2.7349(19)	169	2-x,-1/2+y,1/2-z
N–H···O	1.77	2.717(2)	156	1-x,1/2+y,1/2-z
C–H···O	2.29	3.341(2)	163	-1+x,y,z
C–H···O	2.37	3.372(2)	153	x,1/2-y,1/2+z
C–H···O	2.39	3.338(3)	145	2-x,-1/2+y,1/2-z
C–H···O	2.41	3.436(3)	157	1+x,y,z
C–H···O	2.63	3.634(3)	155	1-x,1/2+y,1/2-z
C–H···O	2.66	3.552(2)	140	1+x,y,z
C–H···O	2.69	3.583(2)	139	-1+x,y,z
C–H···O	2.70	3.547(2)	135	1-x,-y,1-z
Form II				
N–H···O	1.77	2.7722(18)	169	1-x,1-y,-z
C–H···O	2.49	3.516(2)	157	x,-1+y,z
C–H···O	2.65	3.395(2)	125	1-x,-y,1-z
C–H···O	2.67	3.604(2)	145	-x,-y,-z
C–H···O	2.68	3.665(2)	151	-x,-y,1-z

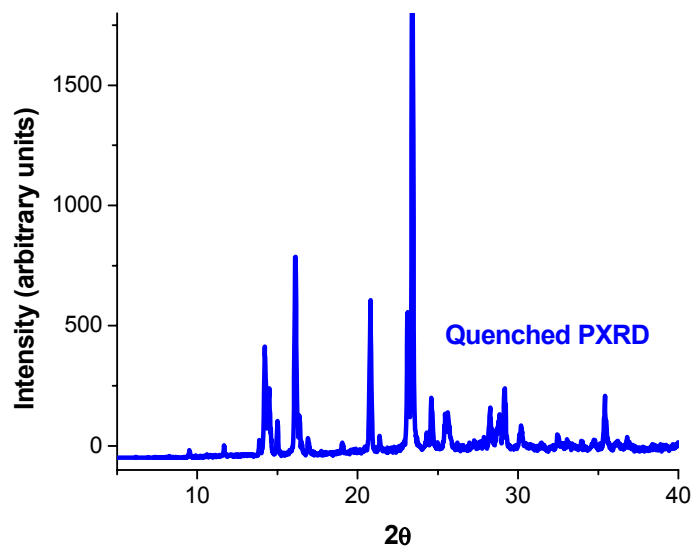[a]N-H and C-H geometries were normalized.

S5 Face indexing of acesulfame Forms I.

S6 Simulated X-ray powder patterns obtained from the single crystal structures of Forms I and II.
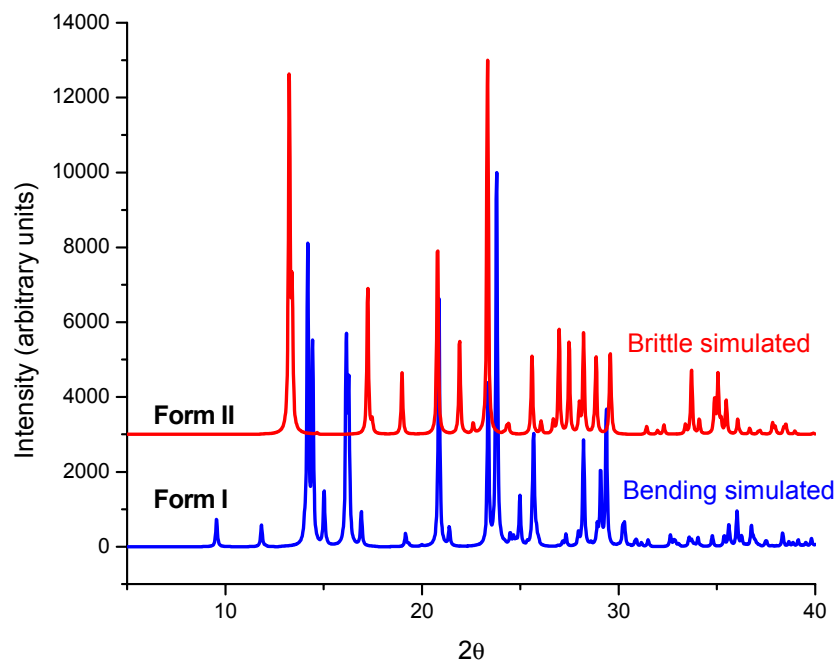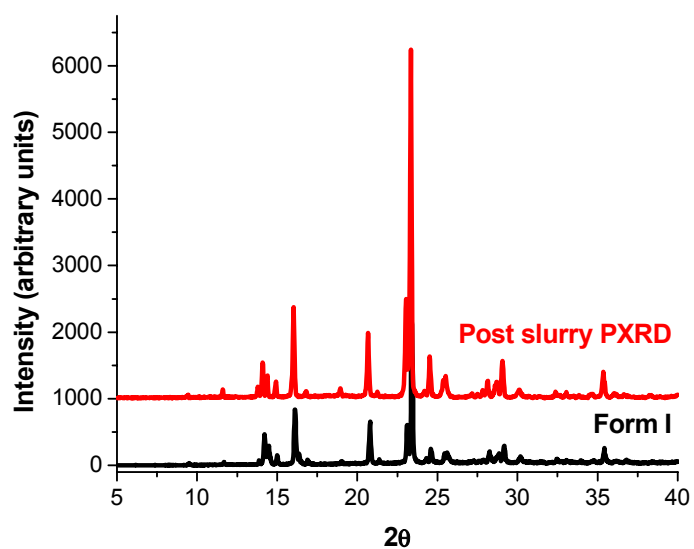
**S7** Form II quenched at 110 °C and recorded the PXRD. It was shown to be Form I.

**S8a** Slurry of Forms I and II. Product phase PXRD shown to be Form I.

A 50:50 (w/w) mixture of Forms I and II was stirred in CH_3OH at room temperature RT for 72h and the filtrate was dried at 30 °C for 24h

S8b DSC trace for the product phase from the slurry experiment. Note the complete transformation to Form I.
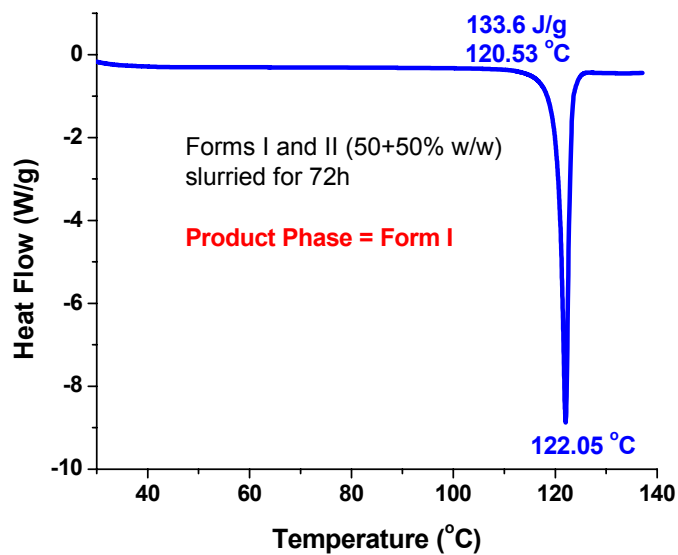
S9 DSC profile of heat–cool–heat for Form II. It suggests that enantiotropic transformation is non-interconvertible under the investigated conditions.

S9
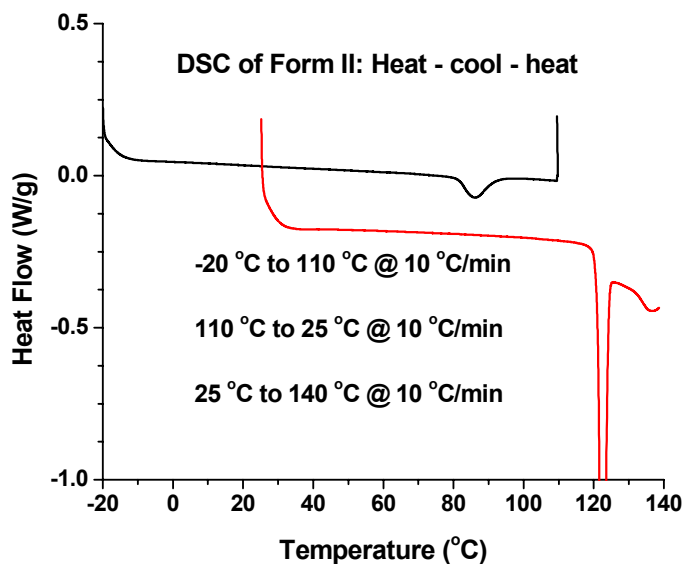
S10. Lattice energy calculations for Forms I and II

Lattice energy calculation for Forms I and II was performed using the Forcite module in the Materials Studio. Single crystal X-ray structure co-ordinates were minimized by the COMPASS force field. The charges were assigned by the force field. The Ewald summation employed to compute the non-bonded interactions that include van der Waals and electrostatic interactions. Finally, lattice energies were computed per molecule based on the number of molecules present in the unit cell (See also: A. Nangia, *Acc. Chem. Res.* 2008, **41**, 595-604 and references cited there in).