

Electronic Supplementary Information

Using fast scanning calorimetry for detecting guest-induced polymorphism by irreversible phase transitions in nanogram scale

Karina V. Gataullina^a, Aleksey V. Buzyurov^a, Marat A. Ziganshin^a, Pavel L. Padnya^a, Ivan I. Stoikov^a, Christoph Schick^{ab} and Valery V. Gorbachuk^{*a}

^a*A.M. Butlerov Institute of Chemistry, Kazan Federal University, Kremlevskaya 18, 420008 Kazan, Russia*

^b*University of Rostock, Institute of Physics, Albert-Einstein-Str. 23-24, 18051 Rostock, Germany*

Contents

Figure S1. Curves of simultaneous TG/DSC for the initial form **I** and the product of its saturation with acetone vapor.

Figure S2. PXRD data for products of form **I** saturation with acetone and acetonitrile.

Figure S3. PXRD data of form **III**, of form **I** prepared by heating of **III**, and of the initial form **I**.

Figure S4. Optical microscope pictures of forms **I**, **II** and **III** in polarized light at RT.

Figure S5. Fast scanning calorimetry (FSC) cooling curves of the melted microcrystalline **I**, **II**, and **III** samples.

Figures S6, S7. Visible changes of microcrystalline **I**, **II** and **III** samples at heating.

Figure S8. Cooling curves of microcrystalline **III** sample from cyclic heating/cooling FSC experiment.

Figure S9. Visual changes of the initial microcrystalline **III** sample after the heating/cooling runs in the same FSC experiment as in Figure S8.

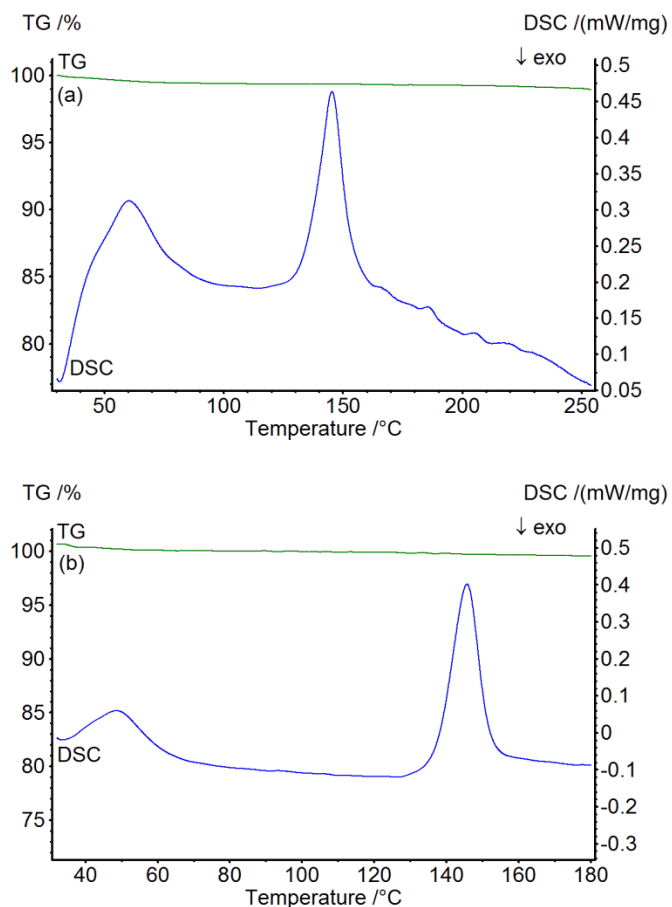


Figure S1. Curves of simultaneous TG/DSC for (a) initial form **I** and (b) form **I** after its powder in initial state was saturated with vapor of $(\text{CH}_3)_2\text{CO}$ at $P/P_0=1$ and 25°C .

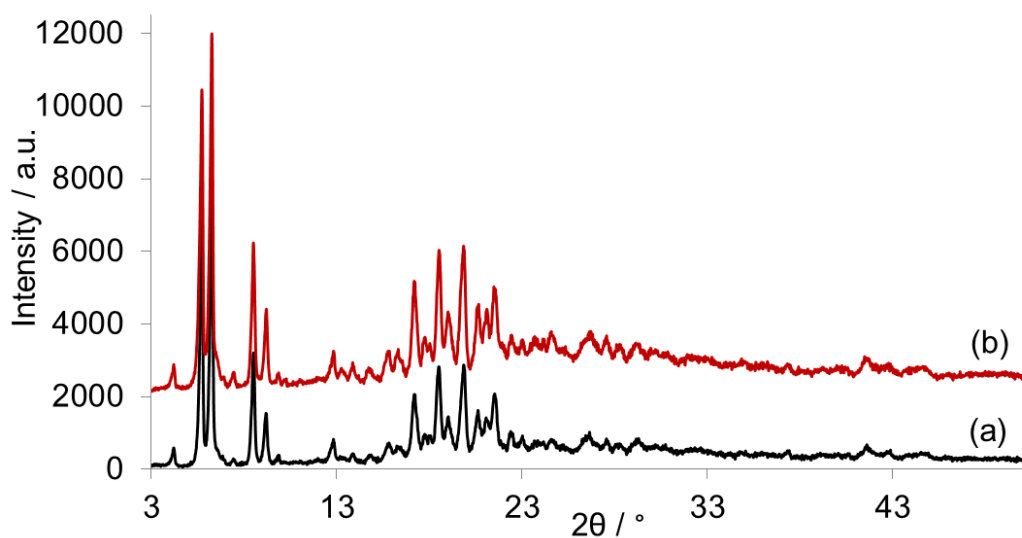


Figure S2. PXRD data for saturated products of form **I** with (a) acetone, (b) acetonitrile.

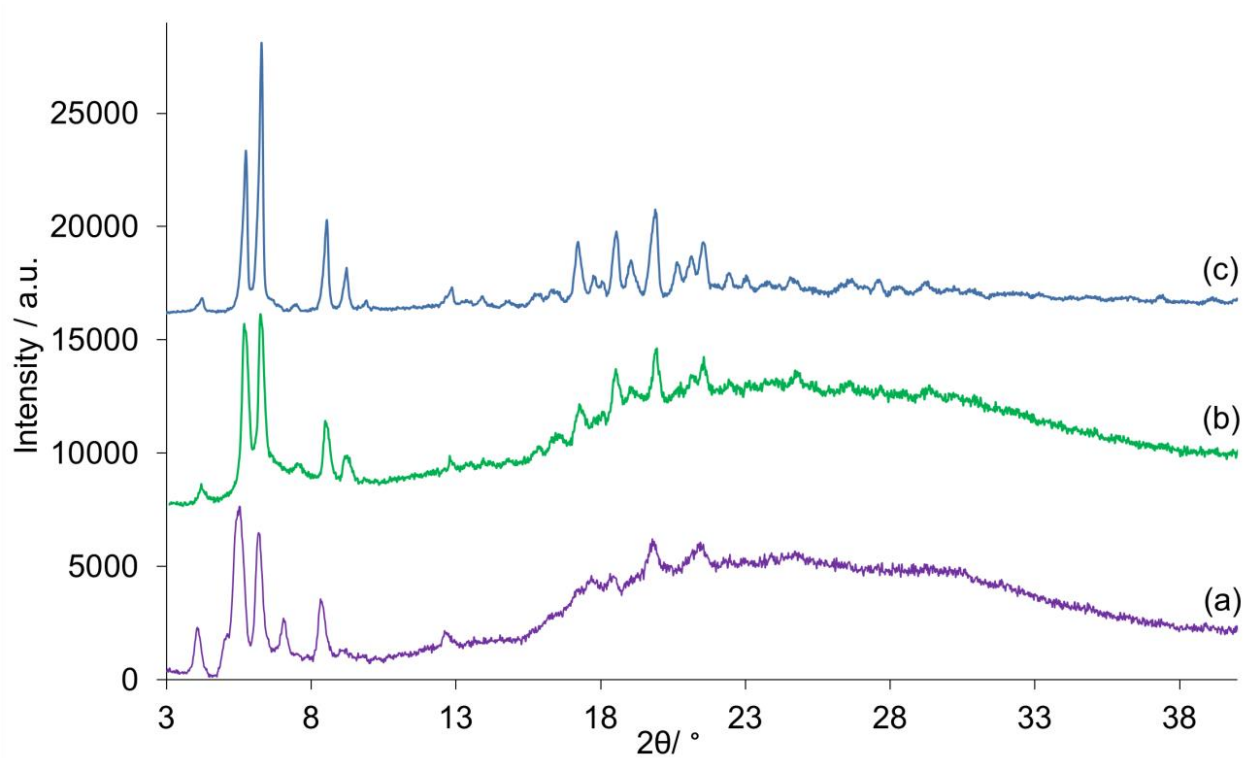


Figure S3. PXRD data of (a) form **III**, (b) form **I** prepared by heating form **III** to 130°C and cooling to RT, and (c) initial form **I**.

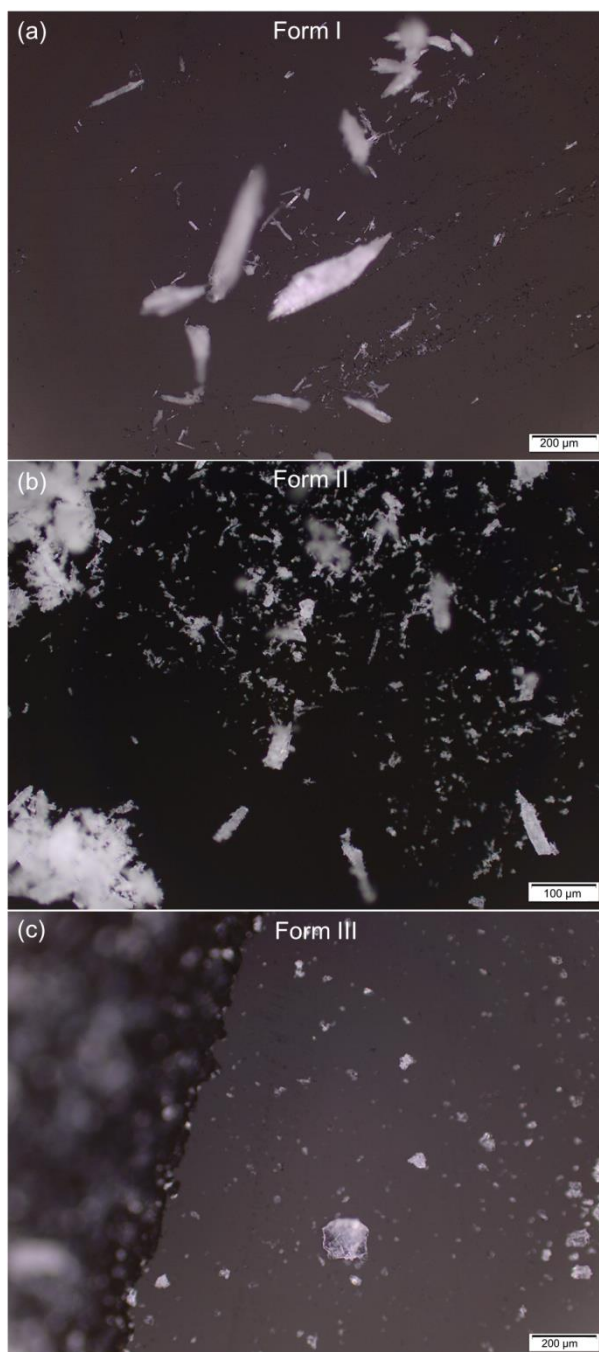


Figure S4. Optical microscope pictures at RT in polarized light of (a) initial form **I**, and samples of (b) form **II**, (c) form **III** prepared by saturation of form **I** with propionitrile and ethanol, respectively, and subsequent drying in vacuum for 3 hours at RT.

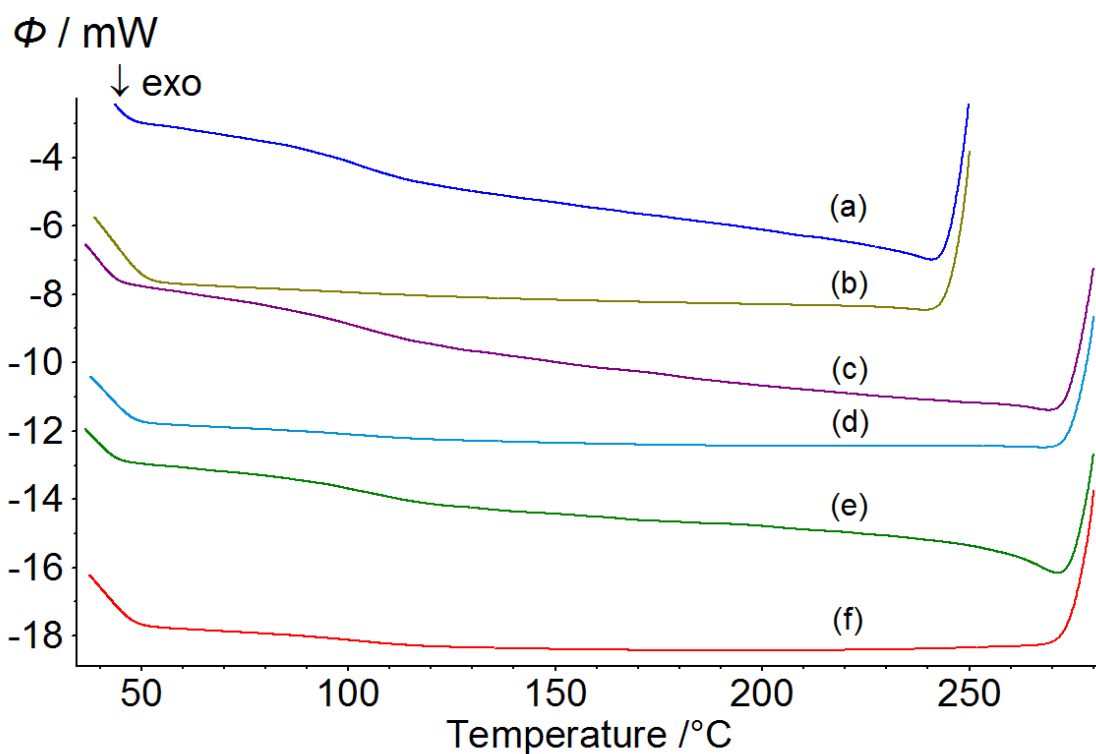


Figure S5. FSC cooling curves of the melted microcrystalline samples of (a, b) **I**, (c, d) **II**, and (e, f) **III**. FSC curves (b, d, f) were determined with a film of silicone oil between crystalline sample and sensor surface. Cooling rate is 1000 K s^{-1} rate. The samples were melted in the first heating run with the same rate. Melts of forms **I** (a curve), **II** (c curve), **III** (e curve) exhibit a glass transition at (T_g) 102, 103, 99°C, respectively.

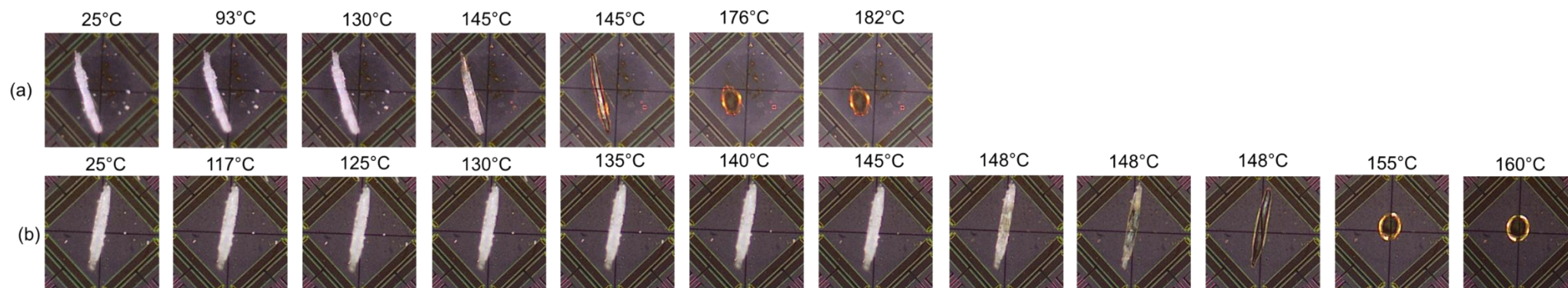


Figure S6. Visible changes of microcrystalline samples in polarized light of (a) initial form **I** and (b) form **II** at heating with a rate of 1000 K s^{-1} and isothermal steps of 30 s to take pictures at given temperatures. The images shown at the same temperatures were taken at the beginning and the end of the isothermal periods. A nitrogen atmosphere is used in the experiment.

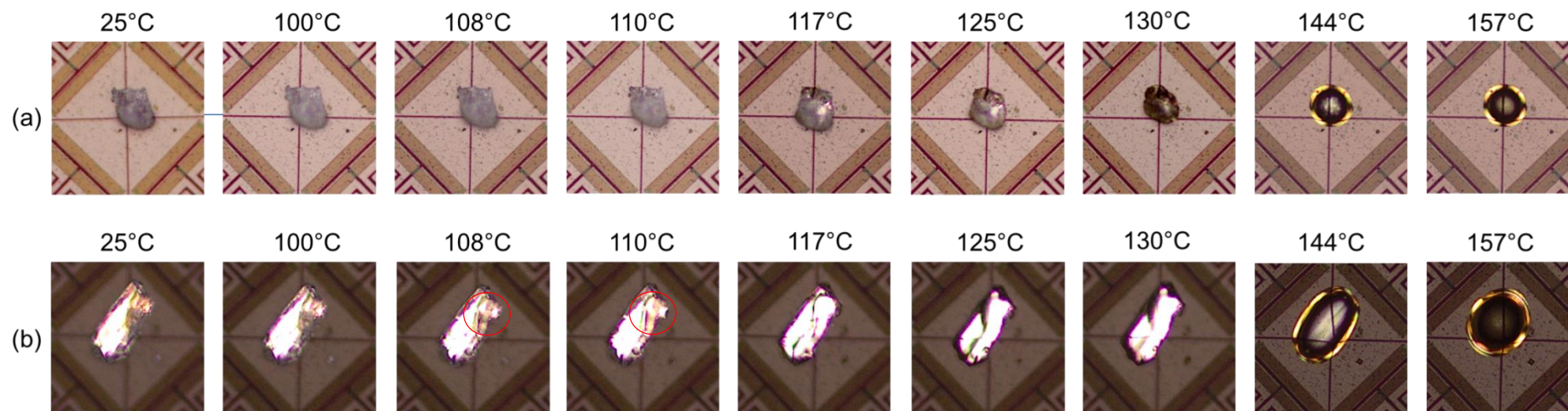


Figure S7. Visible changes of microcrystalline **III** samples in polarized light at heating with a rate of 1000 K s^{-1} and isothermal steps of (a) 30 s and (b) 60 s to take pictures at shown temperatures. A nitrogen atmosphere is used in the experiment. Red circles mark the place where a change in the morphology and shape of the crystalline aggregate is observed at heating from 108 to 110 °C.

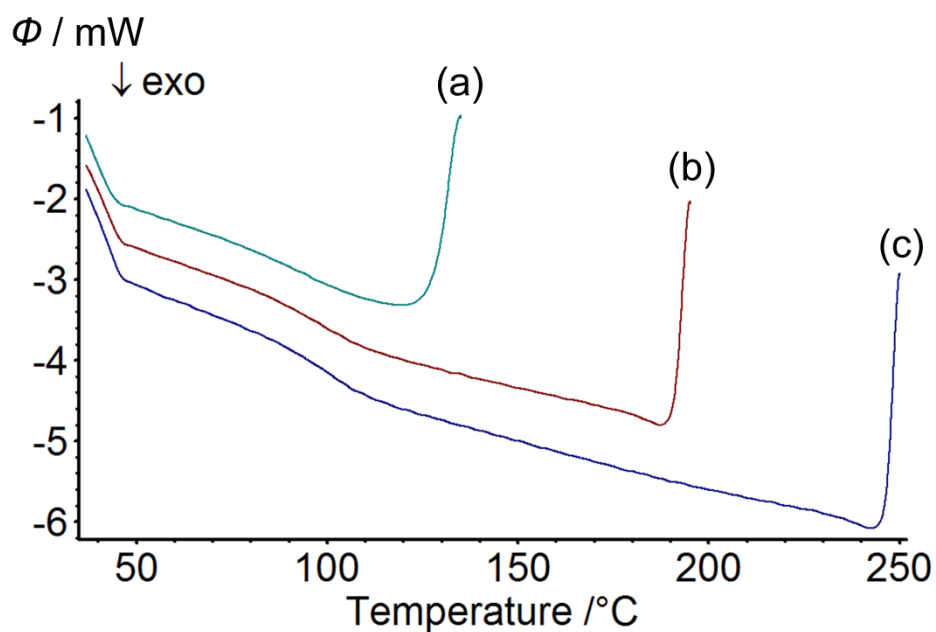


Figure S8. Consecutive FSC cooling curves for initial form **III** after (a) the first heating run to 135°C; (b) the second heating run to 195°C; (c) the third heating run to 250°C. Cooling rate is 1000 K s⁻¹.

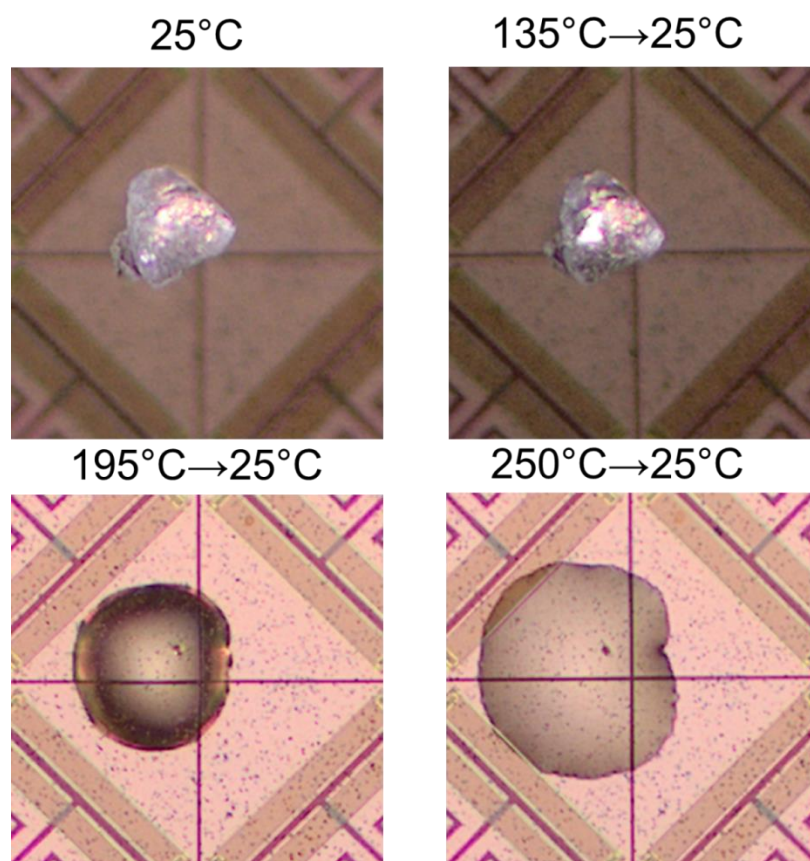


Figure S9. Optical microscope pictures of the initial microcrystalline sample of form **III** in polarized light at RT= 25°C, of the same sample after the first, second and third heating run to the temperatures shown in the same FSC experiment as in the Figure S8.