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In situ mechanochemistry of hybrid materials

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Mechanochemistry is increasingly used for solid state reactions because of its advantages like high yields, high conversion rates, the small produce of waste and the good energy consumption, for which it belongs to green chemistry.[1] Nevertheless, the mechanisms behind mechanochemical reactions are still under investigation which is why in situ setups are needed.[2]
Here we present the in situ investigation of the formation of a series of organic-inorganic hybrid materials from mechanochemical synthesis. The compounds are constructed by guanidinium-, lead(II)- and iodide-ions, with the formula (C(NH2)3)nPbI2+n (n = 1, 2, 3, 4).[3] For the in situ investigations a new setup, developed at the MS beamline (PSI, Switzerland) is used.[4] Due to the gained high quality data an automatic quantitative analyses of the time-resolved powder X-ray diffraction patterns was possible and revealed intermediate formations, solid-solid phase transitions and reactions between the guanidinium lead(II) iodides during the syntheses. We consider these discovered pathways to be linked to the respective structural features of the different compounds. Recent results suggest that 3 is a necessary intermediate for the formation of 4 and that both steps have very different energy dependencies.[5]

[1] James et al., Chem. Soc. Rev. 2012, 41, 413-447.
[2] Užarević et al., J. Phys. Chem. Lett. 2015, 4129-4140.
[3] Wilke et al., Chem. - Eur. J. 2018, 24, 17701-17711.
[4] Ban et al., Anal. Chem. 2017, 89, 13176-13181.
[5] Wilke et al., in preparation.

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