Mechanochemical Reactions and X-ray Powder Diffraction as a Teaching Tool

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Topics in solid state chemistry for first year college students is typically limited to the unit cell contents of a few inorganic structures with the Bragg equation representing about crystallography. Expanding this to presenting the X-ray powder diffraction patterns of the series isostructural alkali halides (AX A = Na, K, X = Cl, Br) provides experimental evidence for the periodic trends of sizes of atoms and ions that we teach all first year students. Using mechanochemistry to convert crystalline reactants to crystalline products within minutes is much faster than solution based reactions followed by crystallization.

Reactions of hydrated transition metal halides with the halide salts of protonated organic amines (e.g enH_2Cl_2) affords dramatic color changes that accompany the chemical reactions taking place while the reactants are ground together using a mortar and pestle. X-ray powder diffraction allows for rapid verification of products.

$MX_2{\bullet}nH_2O$ + 1 or 2 enH_2X_2 —> $(enH_2)MX_4$ or $(enH_2)_2X_2[MX_4]$

Acid-base chemistry can then be explored by grinding the products with sodium or potassium acetates. The simple metathesis reaction of enH_2X_2 with NaOAc will afford $enH_2(OAc)_2$ + NaCl where the appearance of the XPD pattern of is evidence of a reaction. Brønsted acid-base reactions will result in the release of acetic acid and this occurs when a transition metal is present. Detecting the odor of the acid and a color change when the ethylenediamine coordinates to the metal (e.g. Cu^{2+}) is evidence of a reaction and XPD verifies the structure of the product.

Recrystallization of the products from solution connects the mechanochemical reactions to traditional equilibria that occur in solution based chemistry and also affords purification and evidence for the stoichiometry of reactions or identification of limiting reagents.