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**Metastable (Bi, M)<sub>2</sub>(Fe, Mn, Bi)<sub>2</sub>O<sub>6+x</sub> (M: Na or K) Pyrochlores from Hydrothermal Synthesis.** — (Na<sub>0.60</sub>Bi<sub>1.40</sub>)(Fe<sub>1.06</sub>Mn<sub>0.17</sub>Bi<sub>0.77</sub>)O<sub>6.87</sub> and (K<sub>0.24</sub>Bi<sub>1.51</sub>)(Fe<sub>1.07</sub>Mn<sub>0.15</sub>Bi<sub>0.78</sub>)O<sub>6.86</sub> are hydrothermally synthesized from aqueous solutions of NaBiO<sub>3</sub>, Fe(NO<sub>3</sub>)<sub>3</sub>, MnCl<sub>2</sub>, and NaOH or KOH (autoclave, 200 °C, 6 h). The samples are characterized by powder XRD and neutron diffraction, X-ray fluorescence spectroscopy, XANES, <sup>57</sup>Fe Moessbauer spectroscopy, TEM, and magnetic measurements. The pyrochlores crystallize in the cubic space group Fd $\bar{3}$ m. Disorder is present in both the metal and coordinating oxygen positions, along with metal-mixing across both the A and B sites of the structure. The materials exhibit a lack of long-range magnetic ordering that is typical of geometrically frustrated pyrochlores. The compounds are metastable and collapse on heating at about 395 °C. — (DANIELS, L. M.; PLAYFORD, H. Y.; GRENECHE, J.-M.; HANNON, A. C.; WALTON\*, R. I.; Inorg. Chem. 53 (2014) 24, 13197-13206, <http://dx.doi.org/10.1021/ic502411z>; Dep. Chem., Univ. Warwick, Coventry CV4 7AL, UK; Eng.) — W. Pewestorf