A 2D interdigitated [Ni2(adipate)2(spy)4(H2O)2] (spy = 4-styrylpyridine) was transformed to a 3D coordination polymer through [2 + 2] photodimerization between the olefin bonds in spy upon UV-irradiation in a single-crystal-to-single-crystal (SC–SC) manner. During this transformation, a coexisting flexible adipate ligand changes its conformation, and helps in withstanding the conversion strain and retaining the single crystallinity.


**Keywords:** MOF, photoreaction, [2+2] cyclization