Redesign of a Grignard-Based Active Pharmaceutical Ingredient (API) Batch Synthesis to a Flow Process for the Preparation of Melitracen HCl - DTU Orbit (11/08/2019)

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A Grignard-based batch process, for the preparation of Melitracen HCl, has been redesigned to fit a continuous reactor system. The Grignard addition is carried out at room temperature, with subsequent hydrolysis of the magnesium alkoxide intermediate followed by dehydration of the resulting alcohol. The product undergoes further workup by simple gravimetric phase separation and then crystallization with 2 M HCl in diethyl ether to afford pure Melitracen HCl. All steps in the laboratory setup were concatenated, and the setup was proven capable of producing a significant portion of the commercial quantities of Melitracen HCl. The flow setup profits from a reduced footprint, lower energy consumption, fewer synthetic steps, and reduced raw material usage compared to the batch process.

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