Production of active pharmaceutical ingredients (APIs), fine chemicals, food products, and so on has in recent years been focused on implementing process intensification and process optimization tools. Lower costs and higher selectivity as well as better sustainability and competitiveness are the main benefits. A good approach to achieve this is to perform continuous manufacturing together with satisfying process analytical technology (PAT) requirements. The example studied here is the dehydration reaction of 9-allyl-2-chlorothioxanthen-9-ol ("N714-allylcarbinol") to give a mixture of cis- and trans-9H-thioxanthene, 2-chloro-9-(2-propenyldiene)-(9CI) ("N746-butadienes"). A simplified procedure for designing mesoscale tubular reactors is demonstrated together with performance outside of the normal operation windows (higher pressures and temperatures above normal boiling points of solvents). Noninvasive in-line real-time monitoring was established by using Fourier transform near-infrared (FT-NIR) spectroscopy and a suitable partial least squares (PLS) model. High prediction accuracy was achieved and additionally validated by using at-line FT-NIR spectroscopy and off-line HPLC analysis. The presence of impurities was noticed and partly identified in the form of polymers. It is important to note that substrates and products in this work are API intermediates in the production of zuclopenthixol, a product of H. Lundbeck A/S.

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