547f Multistep Synthesis of Tetrazole Compounds in Microchemical Systems

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Microchemical systems are used for kinetic, optimization, and productivity studies of the multi-step synthesis of sodium nitrotetrazolate (NaNT). NaNT is a useful intermediate in the manufacture of specialty energetic materials whose annual demand is on the 1-100 kilogram scale. Aminotetrazole is regarded as safe and is generally used as a starting material to make many other tetrazole derivatives via Sandmeyer-type diazotization and substitution or coupling reactions but even lab-scale production is hazardous due to the high heats of reaction and mixing, high acid concentrations needed, and toxic gas formation. In addition, explosions in solutions containing just 1% diazotetrazole have been documented, posing significant risk to manufacturing personnel. To manage these hazards in lab-scale operations, metal salts are used to prevent formation of the diazotetrazole, adding additional reaction steps and reducing overall yield. Thus, high heat and mass transfer rates inherent in microreactors offer a unique opportunity for safe study and for efficient production.

We have adopted a modular design of micro-mixers and capillary tubing to vary residence times and allow precise control of reaction quenching at low concentrations (~0.05 M) for mechanistic insight and accurate measurements of kinetic parameters of each reaction step. The system was designed so that multiple reactors could be run in parallel to expedite the data collection process while minimizing reagent consumption. The results were then used to optimize the system so that nearly full conversion takes minutes rather than hours. By converting NaNT production from a batch to continuous process, only small, relatively safe amounts of diazonium intermediates are present in the system at any time. Increasing concentrations improved productivity so that a number of reactors can be run in parallel to increase output as needed.