AM Technology
Engineering Chemistry

AMT TN-10

Exothermic control in the Coflore® ACR

Overview

Typically, dropwise addition of reagent is required in batch processes involving exotherms, such as nitrations, in order to control temperature and avoid potential thermal runaway. At scale, dropwise addition steps are extremely time-inefficient, often taking several hours. A key advantage of Coflore flow reactors is their greatly improved heat-transfer coefficient compared to batch reactors, meaning that exothermic reactions can be performed much more efficiently by avoiding the need for time-limiting dropwise addition steps. This technical note summarises feasibility testing performed at AM Technology for an exothermic nitration reaction. Calorimetric data showed the enthalpy of reaction to be -3250 J/g. The nitrated product is formed as a precipitate within the reactor, therefore requiring that any flow reactor can suitably handle solids without blocking.

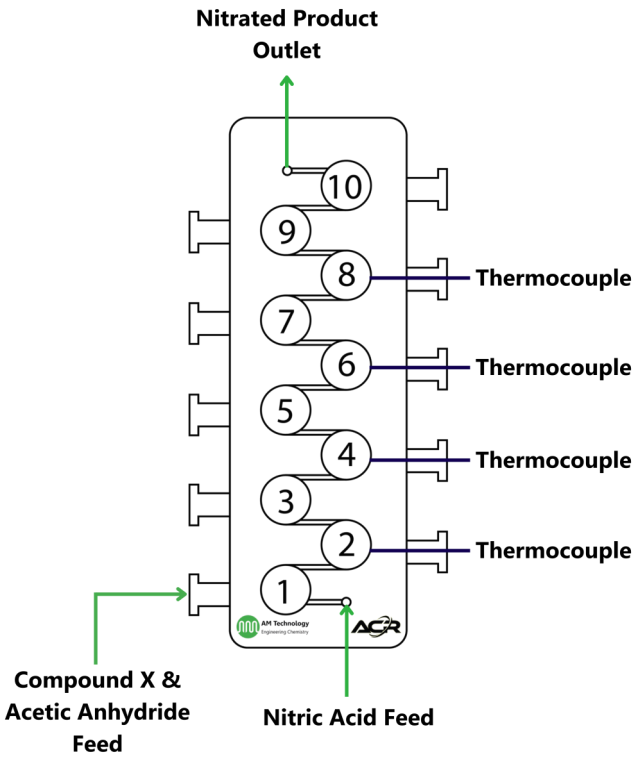

Figure 1: A schematic of the ACR reactor cell block setup.

Experimental Overview

The Coflore ACR was set up as shown in Figure 1, with continuous feed of both the nitrating agent and compound X into cell 1. Thermocouples were positioned at cells 2, 4, 6 and 8 to monitor the reaction temperature over time. Three residence times were tested - 2, 3 and 4 minutes, with the 2-minute residence time representing the harshest conditions in flow (i.e. less time to control the exotherm).

For the 2-minute residence time run, the steady-state temperature values across the ACR reactor cell block are shown in Figure 2, demonstrating the ability of the ACR to handle the reaction exotherm without issue. Three runs were performed, varying the feed temperature to investigate the effect on conversion.

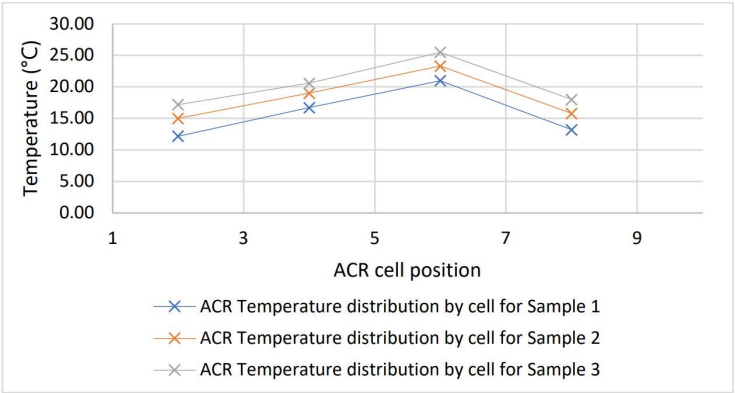

Figure 2: Temperature gradient across the ACR for the 2-minute RT run.

Results

Reports in the literature state that preparation of the nitrated product *via* batch methods takes 6-12 hours with dropwise addition accounting for 3 hours, and at temperatures up to 80°C. The Coflore® ACR was successfully able to control the heat evolution of the nitration reaction at all residence times tested (2, 3 & 4 minutes), with full reaction conversion determined to require an 11-minute residence time. The Coflore ACR therefore offers reaction time efficiency savings equivalent to multiple orders of magnitude. In addition, much lower reaction temperatures were observed (25°C in flow compared to 80°C in batch) and the ACR successfully handled the precipitated product continuously without blocking.

AM Technology Berkeley Court, Cheshire, United Kingdom +44 1928 237 420 info@amt.uk

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