

ADVANCES IN SUPERCRITICAL REACTION PROCESS DEVELOPMENT USING LABORATORY AND PILOT HIGH PRESSURE EQUIPMENT

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Abstract

The primary goal of laboratory supercritical fluid reaction unit testing is to assess technical feasibility of a potential supercritical fluid reaction application.

Initial screening with a Phase Equilibrium analyzer should be carried out to determine the processing conditions in which the reagents and products of interest solubilize and/or precipitate from the supercritical fluid. Experimentation then moves to the use of a supercritical fluid reaction bench top laboratory unit. The Laboratory Unit typically has a 50ml to 4 liter reaction vessel fitted with the appropriate reagent addition modules, mixing, flow meters, and sensors. Product samples and data from the feasibility testing are used to assess product quality, and to research the following process variables: 1) Preparation and solubility of reagents 2) Reaction conditions (temperatures, pressures, use of Co-Solvents to enhance reagent or product solubility. 3) Collection conditions. The reaction product is analyzed to determine how changes in these parameters change yield, purity, and economics of the proposed process.

This information can then be utilized to fine tune the reaction to maximize key parameters for a commercial scale supercritical fluid reaction process. Examples demonstrating the use of both and laboratory SFR unit and supercritical fluid phase equilibrium instrument will be shown.