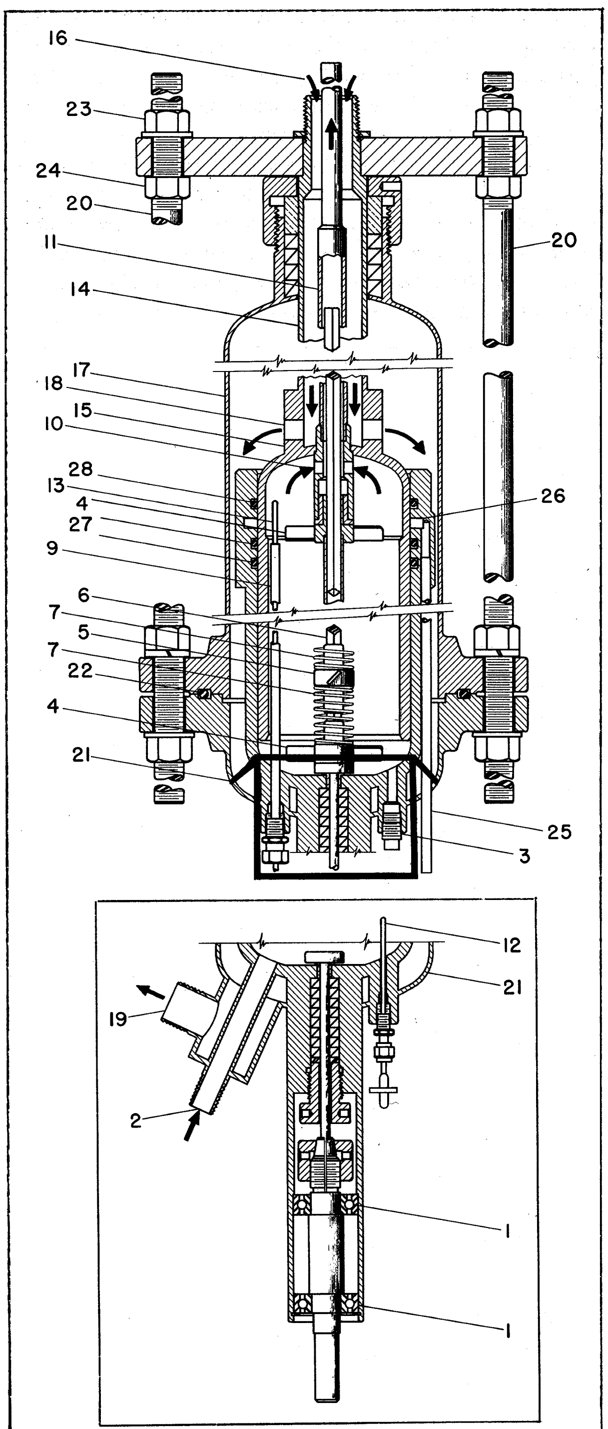
Experimental Adjustable-Volume Reactor



VARIABLE-VOLUME reactor in cross section. The operating parts designated by the numbers are defined in the text. The lower diagram is a view of the bottom, turned 90 degrees.

A new reactor for studying batch or continuous chemical reactions has been designed, built and tested at the U.S. Dept. of Agriculture's Eastern Regional Research Center near Philadelphia. The reactor is presently operating as part of a continuous experimental process for making isopropenyl stearate by reacting stearic acid with methyl acetylene (propyne), and zinc stearate as a catalyst.

Pluses for Variable Volume

This process demonstrated the practicability of all design objectives, which included variable internal volume, working pressures to 750 psi, working temperatures from -20°F to +400°F (for limited time periods to 500°F)—all in a fully jacketed, stirred, longitudinal-flow reactor.

In an experimental reactor, variable volume provides three important features. First is the capability of operating at a critical reaction volume when combining the reactor's volume with that of associated piping; second, the possibility of operating first at minimum volume and later at double this volume when conducting scaleup studies; third, because this reactor can be operated full only, elimination of the need for a liquid level control, which is not readily available for such a small reactor.

How It Works

The reactor (see diagram) has an internal volume variable from 2 to 4 l. Fabricated from standard commercial sizes of type 304 stainless-steel welding-fittings, pipe, rod and plate, the reactor is a vertical unit driven from the bottom by an air motor and a V-belt drive. A stirrer drive at the bottom of the reactor provides for a stationary drive when changing volume and keeps the heat flowing to the spindle bearings (1, in diagram) to a minimum.

Component A, or a preheated premix of components A&B enters the reactor through port (2) and the other component enters through port (3) (shown plugged). As the component mix flows upward, five sets of stirrer blades (4 and 5) with alternating pitches provide mixing action. One of the sets (5) floats on the square stirrer shaft (6), automatically centered by two compression springs (7). Three vertical surfaces on an intermediate stirrer shaft-support, together with thermocouple support (9), act as vortex breakers.

The reacted component mix leaves through four ports (10) and an exit tube (11). A short thermocouple (12) provides for measurement of the component-mix entrance temperature, and a long thermocouple (13) measures exit temperature when the reactor is set for minimum volume. At all other volume settings, this thermocouple is either replaced with one corresponding to the internal length of the reactor, or the exit temperature is measured at the top of the exit tube (11).

Heating and Adjusting

Heating (or cooling) of the reactor is arranged in "counterflow" fashion. Surrounding the product exit tube (11), the heating medium enters the exit tube jacket (14) of the upper chamber (15) at location (16). Flowing downward, it enters the reactor jacket (17) through four ports (18) and leaves through port (19), next to port (2).

Eight rods (20) provide a means for adjusting the internal volume of the reactor and for keeping the reactor jacket (17) bolted to the lower chamber (21), where an O-ring (22) seals between heating medium and atmosphere. Enlargement of the internal volume is accomplished by presetting eight nuts (23) followed by pressurizing the reactor chamber. Decreasing this volume is done by presetting two nuts (24), followed by pressurizing the jacket chamber. Galling between mating surfaces of the reactor halves is avoided by hard chromeplating and grinding the outside diameter of the upper chamber (15).

To prevent cross-contamination between reaction components and heating medium, the two chambers are not sealed against each other; rather, they are sealed against the atmosphere. This is accomplished by having a blowout tube (25) to the outside connected to an annular groove (26). Two O-rings (27) seal the reaction chamber, and one O-ring (28) seals the jacket. O-ring material selection must be based on temperature stability and chemical inertness. A fluoroelastomer O-ring with a durometer hardness of 75 is a good first choice.—*Wolfgang K. Heiland, U.S. Dept. of Agriculture, Eastern Regional Research Center, 600 E. Mermaid Lane, Philadelphia, PA 19118.* *(Mr. Heiland will be glad to furnish detailed fabrication drawings to those who request them.*—Ed