Design of an Enhanced Throughput Catalytic Test System Capable of Rapid Heating and Cooling

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Introduction

High-throughput techniques are used in combinatorial chemistry, for example to permit preparation and screening of hundreds of catalysts simultaneously [1]. The principle of conducting more than one experiment at the same time is generally desirable. Here we present a system allowing three concurrent fixed bed reactor tests, to be conducted on the laboratory scale (2 ml bed volume). This enhancement in throughput is achieved without loss of reaction analysis information.

System Requirements

The system is designed to investigate low temperature alkane isomerization (butane, pentane) on sulfated zirconia catalysts and therefore must fulfill the following conditions:

(I) Isothermal over a wide temperature range, from 0°C (*n*-pentane isomerization) to 650°C (*in situ* calcination of the catalyst material [2])

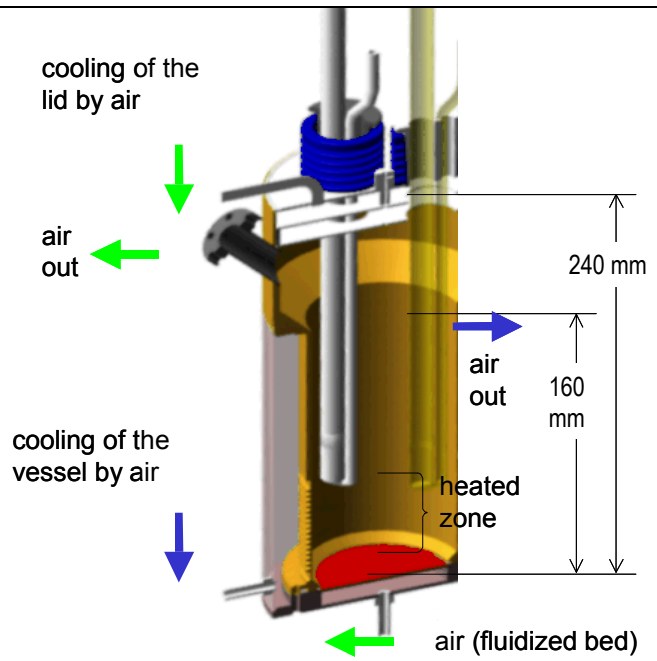
(II) Rapid heating and cooling to reduce time loss

(III) Fast and quantitative gas phase analysis

Design and Test Results

The requirements led to the construction of a reaction vessel in which three U-shaped tubular quartz reactors (inlet Ø 12 mm, outlet Ø 6 mm) are positioned symmetrically. These tubular reactors each contain a quartz frit in the inlet tube to hold the catalyst powders. They are fixed at the top by seals made of polytetrafluorethylene. Cooling the lid by an air flow avoids thermolysis of the PTFE. Isothermal heating is possible using a fluidized sand bed. The bottom of the vessel is heated electrically. It contains a frit of metal wire that supports the sand (50-70 mesh, ca. 500 ml). The sand is fluidized by air flowing through the frit (ca. 12 l/min). A 25 K/min heating ramp is possible. For experiments below room temperature the air can be cooled, e.g. by

liquid nitrogen. The reaction vessel is enclosed by a cylindrical shell that can be purged by air for cooling. Thus a fast return to lower temperatures after activation/calcination is guaranteed (from 450°C to 50°C in ca. 45 min). The temperature of the reactor is monitored by a thermocouple positioned in the center of the vessel and controlled by a second thermocouple close to the heating wire. A four position valve selects the outlet of either one of the three reactors, or the bypass, for analysis. Analysis of the gas phase is performed using a Micro GC (Varian CP 4900) equipped with a thermal conductivity detector, which allows separation of *n*-butane and isobutane within ca. 1 min.



Cross section through the reaction vessel containing the U-shaped reactors

Reaction vessel without cylindrical shell

References

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[2] A. Hahn, T. Ressler, R.E. Jentoft, F.C. Jentoft, *Chem. Comm.*, **2001**, 537-538.