

Adaptation of standard instruments to reproduce specific process conditions - development of a high temperature fixed bed calorimetric cell

INTRODUCTION

Some studies may not be possible with standard thermal analyzers or calorimeters because of some limitations in terms of pressure, temperature, resistance to corrosive atmospheres, etc. But in many cases, the adaption of a few elements of these analyzers can change the situation and allow fitting them to the needs of such studies.

A very large number of reactions now involve solid catalysts. Their evaluation has become a major concern for industrial processes. The Calvet calorimeters are particularly well adapted to help in understanding heterogeneous catalytic processes. Thanks to the large number of calorimetric cells that can be used, they allow matching many industrial conditions.

In the following application, the goal was to develop a cell enabling the possibility of measuring the heat of conversion of gaseous 2-butene to 1-butene flowing through a catalytic bed.

Key elements

Our skills in customized instrument design, our expertise in calorimetric solutions development, and the organization of our R&D service being structured to manage specific projects have been applied for a long time in such cases.

A specific cell allowing the reactive gas to flow through a catalytic bed had thus to be designed. This cell was meant to be used with the CALVET HT calorimeter enabling measurement up to 600°C. The cell had to be integrated in the gas feeding and pressure control system provided by a third party (figure 1).

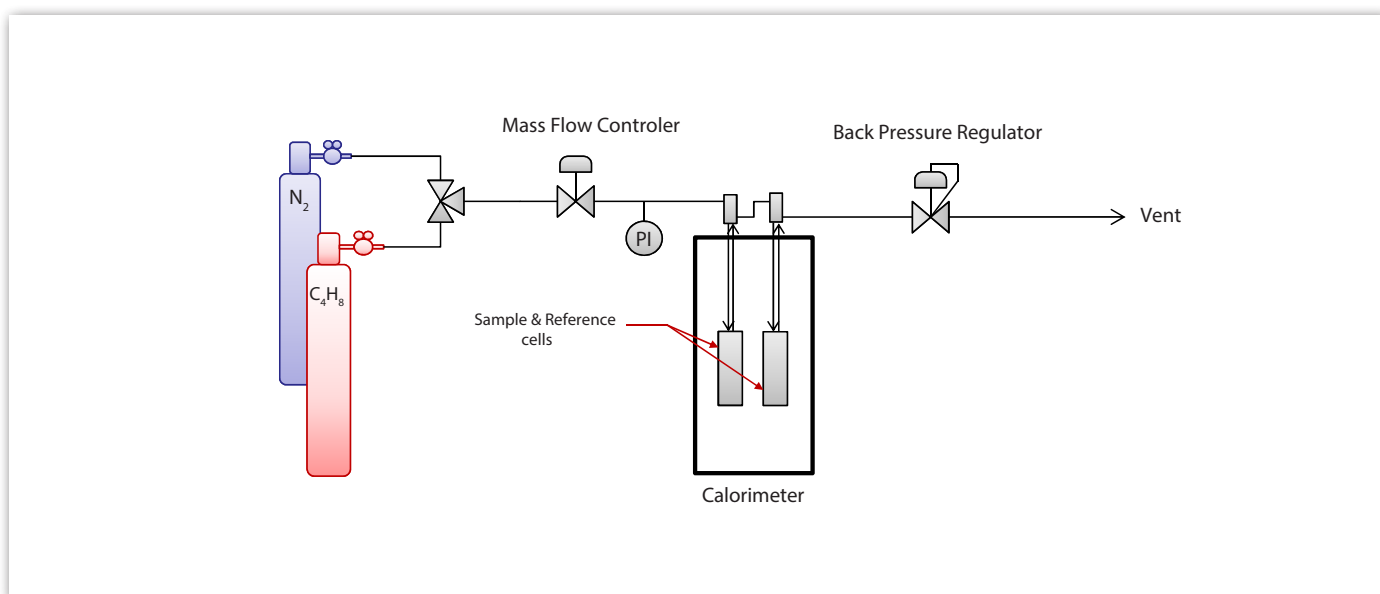


Figure 1: Schematic of the experimental setup

Technical achievements

The provided solution consists in high temperature and high pressure cells (Figure 2) equipped with :

- A gas inlet
- A gas outlet
- A metal filter attached to an inner chamber

The solid catalyst can be poured inside the chamber, on the metal filter. The flow of reactive gas is forced to percolate through the catalyst bed thanks to the long inlet tubing which is soldered to the upper part of the chamber (Figure 3).

The gaseous products of the reaction are then pushed through the coaxial outlet tube.

The heat effect generated by the gas flow is compensated by placing a cell of the same type in the reference well of the calorimeter. They are connected in series to the gas supply line so as to make sure the same gas flowrate is applied in both cells.

Going further

The CALVET HT percolation cell specially designed for the this specific application of butene is perfectly adapted to any type of heterogeneous reaction involving a solid catalyst and a flowing fluid.

A number of calorimetric cells have been adapted to cover specific needs:

- Measurement of pressure generated by a decomposition
- Semi-batch cell with a mechanical stirring
- ...

Characteristics

Material:

- Inconel 625

O-ring:

- Nickel

Dimensions of the cell:

- Inner diameter: 13mm
- Inner height: 58mm

Pressure capabilities:

- 100bar up to 500°C
- 30bar between 500°C and 600°C

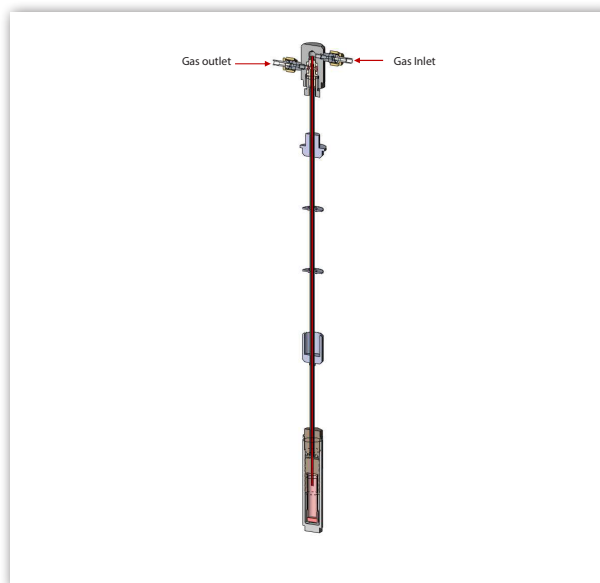


Figure 2: Drawing of the percolation cell

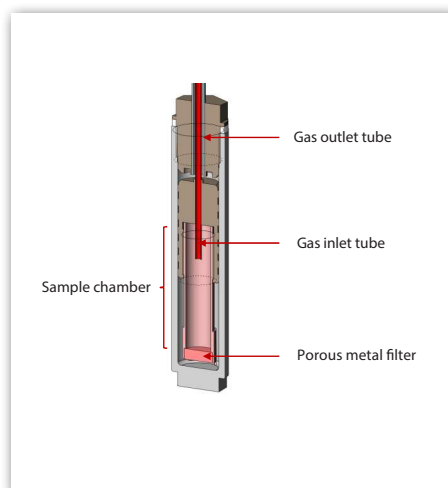


Figure 3: Enlarged view of the body of the cell