SEQENS 'Lab

Process Safety

Seqens'Lab's process safety department supports our teams and our customers in the prevention of chemical reaction risks. Seqens'Lab's methodology is based on the evaluation of risks related to chemical reactions. The Seqens'Lab is equipped with experimental means to test the stability and identify the thermal phenomena of various materials. Our characterization tests of exothermic chemical reactions allow to control the risks of runaway concerning existing processes or help to develop new processes.

1. Thermal scanning analysis

Screening tests are a preliminary step in understanding the behavior of a product subjected to a flow of heat. These tests consist mainly in the study of the stability, over time, of a sample as a function of temperature. The different screening methods offer two modes of analysis:

- Temperature ramping: a temperature gradient (between a start and end temperature) is imposed on the sample,
- Isothermal exposure: the temperature imposed on the sample is constant throughout the analysis. This last method allows to highlight the auto-catalytic character of a reaction.

Most screening tests are characterized by a small amount of sample, ranging from 0.01 to 10 grams, as well as the choice of ramping velocity and temperature range.

Performing the screening tests allows:

- A preliminary analysis of a large number of samples, tests over a wide temperature range (generally from - 80°C to + 450°C),
- Tests on the products of a reaction during its different stages (reagent, intermediate, medium or final product) tests on samples of distillation residues or recycled products,
- Identification of thermal phenomena (endothermic or exothermic),
- An indication of the amplitude of the thermal phenomena and the starting temperature,
- Highlighting the effects of aging on the stability of products (temperature variations).

These screening tests are performed by Differential Scanning Calorimetry which is a technique that determines the variation of the heat flux lost or given by the sample by monitoring the temperature under controlled atmosphere. During heating or cooling, any transformation of the product is accompanied by a heat exchange. Differential Scanning Calorimetry (DSC) allows to determine the temperature at which the transformation occurs and to quantify the heat released or absorbed.

The Seqens'Lab is equipped with

- **TA INSTRUMENT**
- **METTLER**

In addition to the screening analysis by DSC, Seqens'Lab is able to carry out analyses of thermogravimetry (ATG) to characterize the phenomena of loss of mass resulting from a temperature increase.

2. Characterization of planned synthesis reactions

Reaction calorimetry is the method of choice for all processes that require stirring, addition or withdrawal of reagents, or if thermal data must be extrapolated to an industrial scale, such as heat transfer data.

The typical reaction calorimeter consists of a reactor with a volume of 1 to 2 liters, having a similar geometry to the industrial reactor. In such a calorimeter, all the usual operations such as addition, pH control, heating, cooling, distillation can be performed, with simultaneous measurement of heat exchange.

The collected data makes the studied process transparent and allows its optimization.

The RC1 calorimetric reactor, developed by CIBA GEIGY and commercialized by METTLER, is both an automatic laboratory reactor and a reaction calorimeter allowing the calculation of the heat balance of the reaction. Small laboratory installations allow to realize very close to reality, entire processes or only a few steps, on a liter scale.

The reaction calorimeter provides quantitative information on:

- The thermal efficiency of the reaction as a function of time,
- The heat of reaction,
- The specific heat of the reaction mass
- The heat transfer of the stirred reaction mass to the tempered wall of the semi-batch reactor.

The Seqens'Lab is equipped with

- **2 CALORIMETRIC REACTORS RC1**
- **3 DIRECT FLOW CALORIMETRIC REACTORS DEVELOPED BY THE COMPANY ALGOCHEM**

3. Characterization of decomposition reactions

Decomposition reactions can be studied by various specific calorimetric methods. The knowledge of the course of a decomposition reaction allows the estimation of the domain of stability or instability of a reaction, the estimation of the temperature of beginning of decomposition and also allows to estimate the consequences in terms of temperature and maximum pressure reached during the decomposition, of the quantity of released heat. These data allow access to the characteristics necessary for the sizing of the safety devices adapted to the implemented reaction.

The study of the decomposition reactions allows to obtain some characteristic parameters such as:

- The ONSET temperature which is the tem perature at which decomposition is detec ted by the equipment used,
- The heat released by the decomposition reaction,
- The kinetics of the decomposition reaction,
- The maximum temperature reached during
- The decomposition reaction,
- The maximum pressure reached during the decomposition reaction,
- The rate of pressure and temperature rise.

The Seqens'Lab is equipped with

- **PHI-TEC REACTOR**

The PHI-TEC corresponds to the VSP2 adiabatic calorimeter. The acquired information (pressure and temperature levels as well as their evolution rates) helps in the dimensioning of overpressure vents (valves, rupture discs).

3.1 – Pseudo-adiabatic calorimetry

Pseudo-adiabatic calorimeters use a technique where the temperature of the test cell surroundings is controlled to be equal to the temperature of the sample or the temperature of the test cell walls in order to exclude any heat loss from the test cell. With this technique, the heat released by the sample is used to increase its own temperature as well as to increase the temperature of the test cell. The thermal inertia of the experiment is characterized by the Phi factor.

This technique is suitable for obtaining data on the kinetics of runaway reactions. The use of pseudo-adiabatic techniques for the study of thermal runaway is based on the possibility of recalculating the characteristics of the runaway under adiabatic or near adiabatic conditions from the results obtained with pseudo-adiabatic devices. Another important element is the possibility to recalculate the kinetics of a runaway with an initial temperature different from the experimental one.

3.1.1 – PHI TEC

The PHI-TEC is an apparatus allowing the study of exothermic reactions under experimental conditions reproducing those observed during industrial scale reactions. These conditions are made possible with samples having a capacity of only 10 to 120 ml. Information concerning the kinetics and some thermodynamic parameters of the decomposition reactions are thus obtained. The data obtained can be directly exploited in order to define the safety conditions of storage, transport and packaging of the synthesized product.

The reactions performed in the PHI-TEC reactor enable the comportment assessment of the reactional mass in conditions similar to conditions of the industrial reactor. Data extrapolations are therefore easier and more reliable.

3.1.2 – ARC: Accelerating Rate Calorimeter

The ARC reactor allows the search for the starting temperature of an exothermic reaction of the sample by raising the temperature of the thermostat in successive steps followed by a period of observation of the cell temperature, in order to see if the latter does not rise in an autothermic way. When an exotherm is observed, the temperature and pressure are recorded as a function of time by the apparatus, while the external temperature is controlled by the cell temperature to ensure pseudo-adiabatic conditions in the cell.

Seqens'Lab plans to equip itself with an ARC reactor by 2022.

3.2 – Isothermal Calorimetry

Isothermal calorimetry is a technique in which the sample contained in a test cell is maintained at a constant temperature imposed by the apparatus. When a temperature change is recorded, the heating system automatically adjusts to keep the temperature of the chamber constant.

The thermal flux released or absorbed by the mixture of reagents is dissipated through an assembly of thermocouples constituting a fluxmeter enabling a direct flux recording. The reagent mixing devices are directly linked to the choice of the cell:

- A mixing cell by overturning : the cell has two compartments separated by a piston. The two compartments are put in communication by the turning of the apparatus.
- A membrane mixing cell: the two compartments are connected by piercing a separation membrane.

The C80 isothermal calorimeter is characterized by a high sensitivity, the possibility of agitation by continuous inversion of the apparatus and the use of tight cells limiting the phenomena of vaporizations.

The Seqens'Lab is equipped with

C80 ISOTHERMAL

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