

# C80 Versus DSC

## Comparison and specific applications



Ecole d'ingénieurs et d'architectes de Fribourg  
Hochschule für Technik und Architektur Freiburg

laurent.brandinu@edu.hefr.ch

**Laurent Brandinu, Charles Guinand, Pierre Brodard**

University of Applied Sciences of Western Switzerland CH-1705  
Fribourg, SWITZERLAND

### INTRODUCTION

The Calvet-C80 is one of the best calorimeter for the study of complex reactions, thanks to a chamber of reaction/reference surrounded by more than 300 thermocouples, a cell with a much bigger volume and very large choice of them (for in-line pressure measurement, mixing, gas introduction, etc).

However, extremely slow heating and cooling rates (not more than 1°C/min) makes an experiment last one or two days.

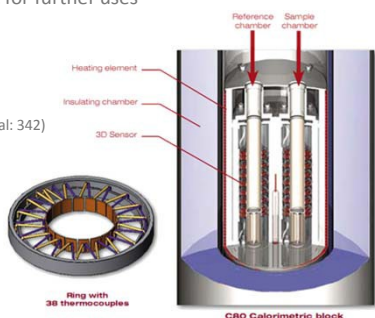
The software is easy to use and designed to treat the enormous amount of data sent by the C80 (Pressure, HeatFlow, both temperatures of the furnace and the sample, etc).

### Goal of the project

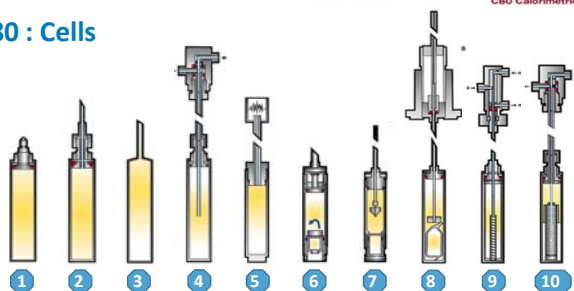
- Restart the C80 after a complete review by Setaram
- Try different compounds, reactions, decompositions,...
- Make a comparison between DSC and C80 for a chosen reaction
- Elaborate a standard procedure for further uses

### C80 : Specifications

- Measurement precision: 94%  
9 rings of 38 thermocouples each (Total: 342)
- Temperature max: 300°C
- Pressure max: 100 – 1000 bar  
(Depending on the cell)
- Heating rate: 0.001 – 1°C/min
- 10 different cell types



### C80 : Cells



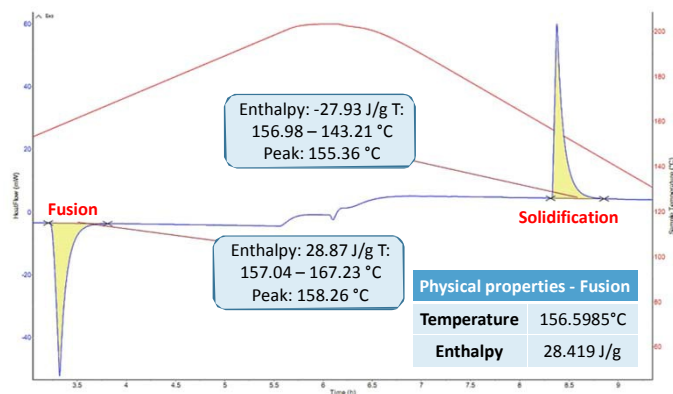
- Standard / high pressure
- Vacuum
- Liquid Cp
- Gaz circulation cell
- Pressure transducer cell
- Mix by inversion
- Mix by membrane perforation
- Mix by bulb breakage
- Liquid circulation
- Liquid percolation

### Comparison – Qualities

| C80   | DSC                                      |
|---|--|
| • Large choice of cells                       | • High heating rates                     |
| • Software Calisto (by AKTS)                  | • Cooling more efficient                 |
| • Solid, liquid and gas phases can be studied | • Autosampler                            |
| • Reactions also can be studied               | • Few quantities of product (analytical) |
| • Thermocouples all around the cell           | • Reduced choice of cells                |

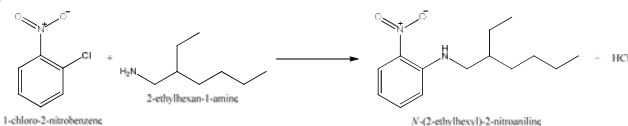
- André R: *Microcalorimétrie de mélange et de réaction – Appareils, cellules et applications*, Setaram, 2013
- U.S. Chemical Safety and Hazard Investigation Board, *Morton International Explosion – Investigation Report*, 2000
- Johnson R, Rudy S, Unwin S: *Essential Practices for Managing Chemical Reactivity Hazards*, 2003

### Indium fusion: temperature & enthalpy

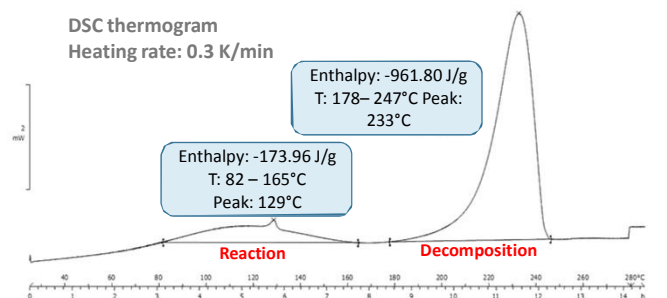
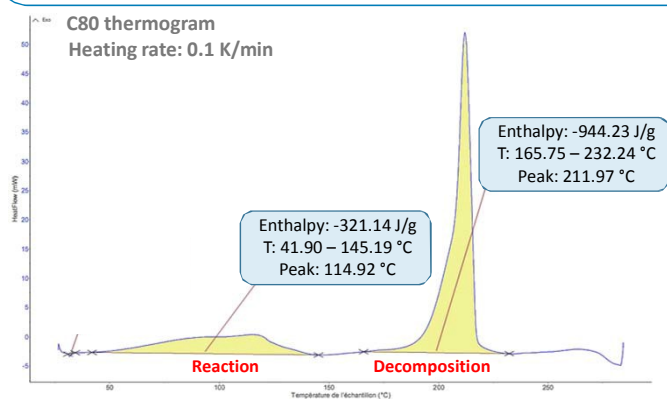


### Morton case: Industrial accident

Synthesis: Automate Yellow 96



| Reaction    |                                | Decomposition Temperature |                                 |
|-------------|--------------------------------|---------------------------|---------------------------------|
| Temperature | From 60-80°C to 160°C          | From 180°C to 240°C       |                                 |
| Enthalpy    | Approx. -330 J·g <sup>-1</sup> | Enthalpy                  | Approx. -1230 J·g <sup>-1</sup> |



### CONCLUSIONS

- Large domain of studies Solid, liquid and/or gas phases Synthesis, decompositions, phase changes, etc...
- Precision in measurement  
Cell completely surrounded by thermocouples
- Large choice and re-usable cells
- Software capabilities (Calisto made by AKTS)

