PROCESS SAFETY

THERMAL ANALYSIS, CALORIMETRY & THERMOKINETICS SOLUTIONS

• QUICK STABILITY SCREENING •
• THERMAL STABILITY WITH PRESSURE AND EVOLVED GAS DATA •
• PROCESS / SYNTHESIS REACTION UNDERSTANDING •
• ADIABATIC / ACCELERATING RATE CALORIMETRY •
• MODELLING AND PREDICTION WITH KINETICS ANALYSIS SOFTWARE •
YOUR CHALLENGES

In chemical, pharmaceutical and many more industrial settings process safety is of critical importance. Thermal risks must be assessed in normal and runaway process conditions to avoid costly downtime and damaging incidents to health, the environment and corporate reputations.

The Process Safety laboratory is fundamental to any chemical facility. Whether synthesising small or large amounts of material each process step and compound needs assessing for thermal stability.

To avoid thermal hazards the entire process from lab, to pilot, to plant, needs safe scale-up support with dedicated instruments. KEP Technologies understands your challenges and offers a choice of solutions that provide experimental control, instrument versatility and quality results.

COMMON PROCESS SAFETY STUDIES & SOLUTIONS

- Thermal Stability with Pressure and Evolved Gas
- Adiabatic Tests
- Quick Screening
- Modelling with software
- Process Understanding

In chemical, pharmaceutical and many more industrial settings process safety is of critical importance. Thermal risks must be assessed in normal and runaway process conditions to avoid costly downtime and damaging incidents to health, the environment and corporate reputations.

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CUSTOMER TESTIMONIAL

“If I was setting up a new process safety laboratory, I would start with a CALVET Calorimeter because even if I couldn’t solve each process safety problem, it can be used for both synthesis reactions (mixing cell) and decomposition reactions, by offering an appreciable advantage, namely pressure measurement. I would start with a CALVET even if it meant supplementing it later on.”

Professor Stoessel - TÜV SÜD Process Safety Basel, Switzerland

THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is radically step-changing its coverage of the process safety market by offering the widest and most versatile choice of solutions. Now you can consult with one company, KEP Technologies, to address your challenges across the broadest number of process safety studies on the market.

Each solution embodies our “Reimagine Material Characterization” value proposition by delivering strongly against the three core customer benefits of Experimental Control, Instrument Versatility and Quality Results.

We believe solutions that provide these benefits will deliver the highest value to our customers.

In addition to our core range we are able to provide customised solutions by harnessing the engineering and project management of our highly skilled organisation.

EXPERIMENTAL CONTROL

INSTRUMENT VERSATILITY

QUALITY RESULTS

CUSTOMIZED SOLUTIONS

Modular design allowing for upgraded and tailored functionality
Access to all previous non-proprietary custom requests
Open access to engineering development team
INSTRUMENT

SETLINE

EASY TO USE WITH ROBUST SENSOR TECHNOLOGY
ensuring quality, consistent and reliable data

AVAILABLE WITH HIGH PRESSURE CRUCIBLES

ACCESSIBLY PRICED INSTRUMENT & SENSOR
for easy cost effective replacement

COST OF OWNERSHIP LOWERED through simplified maintenance and a Replacement Parts Guarantee

TECHNICAL & APPLICATION SUPPORT
for fast expert help on any question

CALISTO 2.0 EXCLUSIVE SOFTWARE
for intuitive and easy data handling

SPECIFICATIONS

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range (°C)</td>
<td>-170 to 700</td>
</tr>
<tr>
<td>Programmable heating rate (°C/min)</td>
<td>0.01 to 100</td>
</tr>
<tr>
<td>Enthalpy accuracy / precision* (%)</td>
<td>+/- 0.8 / 2.5</td>
</tr>
<tr>
<td>Temperature accuracy / precision* (°C)</td>
<td>+/- 0.3 / 0.50</td>
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<tr>
<td>DSC measurement range (mW)</td>
<td>+/- 6 000</td>
</tr>
<tr>
<td>Atmosphere</td>
<td>Inert gas, air, High pressure crucibles up to 500 bar at 600 °C</td>
</tr>
<tr>
<td>Autosampler</td>
<td>SETLINE DSC+ version featuring a 59 positions autosampler</td>
</tr>
</tbody>
</table>

* Based on indium melting tests

For more information on specifications please consult the product information and brochures available on our website: www.setaramsolutions.com

APPLICATION

SCREENING DSC : ANALYSIS OF PROPERGOL

INTRODUCTION

The thermal decomposition of Propergol was studied in a tightly closed high pressure crucible and in an open crucible with milligram-scale samples.

EXPERIMENT

Both experiments were run at a scanning rate of 3°C/min, under inert gas conditions.

RESULTS AND CONCLUSION

It was observed that if the pressure rises during decomposition (closed crucibles), both total heat and reaction schemes are different. However, three peaks remain at similar positions and shape (cf. arrows), which was confirmed by built-in deconvolution module of Calisto data treatment software.

The flexibility of Setline DSC gives fast insight into the behavior of chemicals under varying gaseous conditions (ex: ambient pressure vs. high pressure, oxidizing vs. inert conditions, etc...).
**APPLICATION**

**DECOMPOSITION OF DTBP BY RAPID SCREENING CALORIMETRY**

**INTRODUCTION**

Di-Ter Butyl Peroxide (DTBP) is an unstable chemical used in the polymer industry to initiate polymerization reactions. As large quantities of such a product need to be stored at the plant, it is necessary to assess the risks with the thermal decomposition of this chemical. DTBP is also a typical compound used to assess the performance of Accelerating Rate Calorimeters and Rapid Screening Calorimeters.

**EXPERIMENT**

The temperature and pressure increase data obtained are very repeatable.

**RESULTS AND CONCLUSION**

5 g samples of the same 15 wt% (DTBP) solution in toluene were heated in a 8 mL titanium cell at 2 °C/min from 40 °C up to 300 °C.

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**INSTRUMENT**

**RSC-400 AS**

**DUAL SAMPLE TESTING** for greater throughput and for greater accuracy when one sample and one reference (inert solvent) are tested at the same time.

**RADIATIVE HEATING** and accurate temperature control (0.01°C) for more accurate decomposition temperature measurements.

**WITH 8 mL SAMPLE HOLDERS**, representative samples (in terms of volume and mass) can be tested.

**ACCESSIBLY PRICED** instrument and replacement parts.

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**SPECIFICATIONS**

<table>
<thead>
<tr>
<th>Specification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range (°C)</td>
<td>Room Temperature to 400</td>
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<tr>
<td>Temperature accuracy / control (°C)</td>
<td>0.01</td>
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<tr>
<td>Heating rate range (°C/min)</td>
<td>0.5 to 10</td>
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<tr>
<td>Modes</td>
<td>Temperature Scanning, Isothermal, Dual Scan</td>
</tr>
<tr>
<td>Pressure Range (bar)</td>
<td>0 to 200</td>
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<tr>
<td>Pressure Resolution (bar)</td>
<td>0.001</td>
</tr>
<tr>
<td>Pressure Accuracy (bar)</td>
<td>+/- 2</td>
</tr>
</tbody>
</table>

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**INSTRUMENT**

**CALVET Calorimeter**

**APPLICATIONS**

**ANALYSIS OF DIETHYL SULFATE**

**INTRODUCTION**

The thermal decomposition of diethyl sulfate was studied in a tightly closed high pressure cell equipped with a pressure measurement system.

**EXPERIMENT**

The experiment was run at a scanning rate of 0.5°C/min.

**RESULTS AND CONCLUSION**

It was observed that the biggest contribution to the pressure increase was due to a first decomposition at about 175°C. The second decomposition peak is probably consuming part of the gas produced at the first stage. More information can be extracted from this signal like pressure release rate, condensable / non condensable gas ratio.

**POLYMERIZATION STUDY**

**INTRODUCTION**

The polymerization reaction of Vinyl Pyrrolidone in presence of 4,4'-azobis-cyanovaleric acid was studied using a CALVET Calorimeter with membrane mixing cells.

**EXPERIMENT**

Vinyl Pyrrolidone and 4,4'-azobis-cyanovaleric acid were placed in the two chambers of the cell, separated by a thin membrane. The calorimeter was run under isothermal mode at 50°C. In-situ mixing was provided by piercing the membrane.

**RESULTS AND CONCLUSION**

It was observed that a first, sharp peak, probably linked with the initiation of the reaction, was followed by a slower kinetics and higher heat process. This second peak is linked with the polymerization of Vinyl Pyrrolidone.

Deconvolution of these peaks gives the heat of the initiation, and by difference, the heat of polymerization could be calculated.

**SPECIFICATIONS**

<table>
<thead>
<tr>
<th></th>
<th>CALVET</th>
<th>CALVET CRYO</th>
<th>CALVET HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range (°C)</td>
<td>Ambient to 300</td>
<td>-196 to 200</td>
<td>Ambient to 600</td>
</tr>
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<td>Temperature accuracy (°C)</td>
<td>+/-0.3*</td>
<td>+/-0.5**</td>
<td>+/-1*</td>
</tr>
<tr>
<td>Temperature precision (°C)</td>
<td>+/-0.15*</td>
<td>+/-0.25**</td>
<td>+/-0.5*</td>
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<tr>
<td>Programmable temperature scanning rate</td>
<td>0.001 to 2°C/min</td>
<td>0.01 to 1°C/min</td>
<td>0.01 to 2°C/min</td>
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<tr>
<td>Enthalpy accuracy</td>
<td>+/-0.4*</td>
<td>+/-0.2**</td>
<td>+/-1*</td>
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<tr>
<td>Calorimetric precision (%)</td>
<td>+/-0.5**</td>
<td>+/-1.5*</td>
<td></td>
</tr>
<tr>
<td>Cells (ml)</td>
<td>Up to 12.5 (standard cell)</td>
<td>Up to 12.5 (standard cell)</td>
<td>Up to 7</td>
</tr>
<tr>
<td>Pressure measured and controlled (bar [psi])</td>
<td>350 [5,075]; 600 [8,700]; 1000 [14,600]</td>
<td>100 [1,450]; 600 [8,700]; 1000 [14,600]</td>
<td>100 [1,450]; 300 [4,350]; 400 [5,800]</td>
</tr>
</tbody>
</table>

* Based on indium melting tests ** Based on naphthalene melting tests

For more information on specifications please consult the product information and brochures available on our website: www.setaramsolutions.com
### INSTRUMENT

**TAC-500 AS**

- **FAST TRACK RATE** for a good measurement accuracy even at lower phi factors
- **LOW ONSET TEMPERATURE** detection threshold
- **PRACTICAL BENCH TOP INSTRUMENT** with a compact design for lab space saving and easier maintenance
- **CONVENIENT TO USE** with good usage to maintenance time ratio
- **ACCESSIBLY PRICED** instrument and replacement parts

### SPECIFICATIONS

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range (°C)</td>
<td>Room temperature to 500</td>
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<tr>
<td>Temperature increase detection</td>
<td>0.005 to 0.02</td>
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<tr>
<td>Threshold (°C/min)</td>
<td></td>
</tr>
<tr>
<td>Temperature Precision (repeatability, °C)</td>
<td>+/- 0.05</td>
</tr>
<tr>
<td>Modes</td>
<td>Heat-Wait-Search, Isothermal, Temperature scanning</td>
</tr>
<tr>
<td>Options &amp; Cells</td>
<td>Magnetic stirring</td>
</tr>
<tr>
<td></td>
<td>Cells made of Titanium, SS316L or Hastelloy</td>
</tr>
<tr>
<td>Pressure Range (bar)</td>
<td>0 to 200</td>
</tr>
<tr>
<td>Pressure Resolution (bar)</td>
<td>0.001</td>
</tr>
<tr>
<td>Pressure Accuracy (bar)</td>
<td>+/- 2</td>
</tr>
</tbody>
</table>

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### APPLICATION

#### DECOMPOSITION OF DTBP USING ACCELERATED RATE CALORIMETRY

#### INTRODUCTION

Peroxides, including Di-Ter Butyl Peroxide (DTBP), are typically unstable chemicals that require careful safety studies before being involved in industrial processes.

#### EXPERIMENT

The following were heated in 8 mL titanium cells using the Heat-Wait-Search mode:
- 5 g samples of the same 15 wt% DTBP solution in toluene
- 1 g sample of a 40 wt% DTBP solution in toluene

The Heat-Wait Search parameters were:
- Start temperature: 97 °C
- Temperature steps: 5°C
- Soak Time: 30 min, wait time: 30 min, search time: 15 min
- Detection threshold: 0.02 °C/min
- End Temperature: 250 °C (400 °C with the 40 wt% DTBP solution)

#### RESULTS AND CONCLUSION

The analysis of experimental data allows for the determination of the onset temperature of decomposition, the adiabatic temperature rise (raw and phi-factor corrected), the temperatures at maximum temperature rise and pressure rise rates, and the pressure increase under adiabatic conditions.

This series of tests has shown the impact of concentration on the temperature and pressure rise of DTBP. A significant increase of concentration leads to drastically higher thermal and pressure risks.

Accelerating Rate Calorimetry provides the necessary data to evaluate these risks, and the precision (repeatability) of TAC-500 AS measurements has been proven.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Tonset (°C)</th>
<th>ΔTad, raw (°C)</th>
<th>ΔTad, corrected (°C)</th>
<th>ΔT at max T rate (°C)</th>
<th>ΔT at max P rate (°C)</th>
<th>ΔTad (Mpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DTBP 15% -1</td>
<td>121.56</td>
<td>56.17</td>
<td>104.50</td>
<td>169.16</td>
<td>164.73</td>
<td>1.90</td>
</tr>
<tr>
<td>DTBP 15% -2</td>
<td>121.70</td>
<td>54.46</td>
<td>101.32</td>
<td>166.34</td>
<td>172.07</td>
<td>1.84</td>
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<tr>
<td>DTBP 15% -3</td>
<td>121.64</td>
<td>54.89</td>
<td>101.90</td>
<td>164.30</td>
<td>168.35</td>
<td>1.83</td>
</tr>
<tr>
<td>DTBP 40%</td>
<td>116.79</td>
<td>138.71</td>
<td>257.44</td>
<td>210.55</td>
<td>187.46</td>
<td>9.70</td>
</tr>
</tbody>
</table>
The decomposition of 3-methyl-4-nitrophenol was studied at different heating rates using DSC and CALVET (A).

The experiments at different heating rates were treated with AKTS software (B).

The variation of the runaway time under true adiabatic mode (Phi Factor = 1) can be calculated for any process temperature (C). The critical value TMRad = 24 hours is obtained at 153°C in that case. Dashed lines depict the confidence interval of the calculation.

An adiabatic experiment with a Phi factor = 3.2 was performed for the final validation of the simulation, and compared to calculated adiabatic data (D).

SADT can be determined applying «Finite Element Analysis» (D).