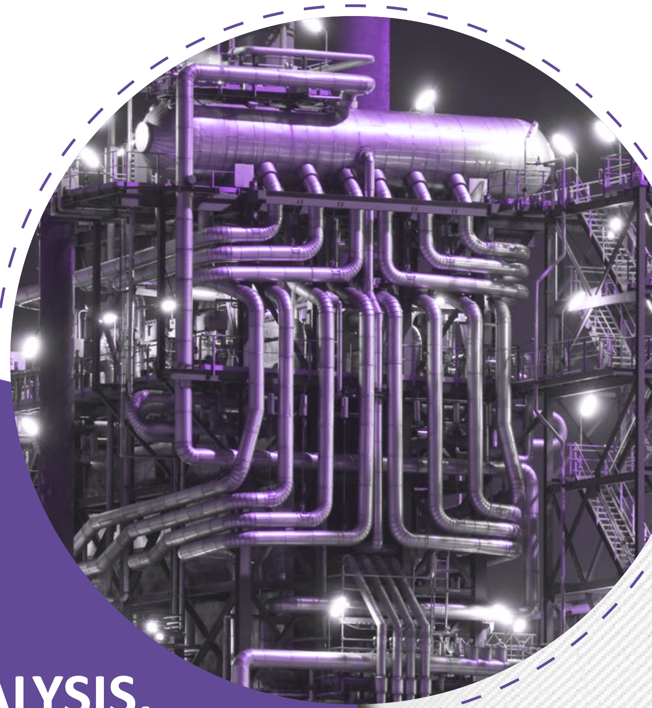
PROCESS SAFETY



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 •

setaram
KEP TECHNOLOGIES

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.

COMMON PROCESS SAFETY STUDIES & SOLUTIONS

This brochure presents some of our solutions in this field and we encourage you to contact us for more information.

Quick Screening

Using small samples a fast indication of thermal instability can be gained by running all process materials through a DSC Thermal Analyzer. The DSC measures the temperature and heat of decomposition, with the main study focus being when and how much heat is given off. As sample sizes are small and pressure data isn't an output of DSC screening, if potential process safety issues are indicated, such as exotherms, further study is required.

Thermal Stability with Pressure and Evolved Gas

For suspect materials from the DSC stage and due to the central role of gas releasing reactions in plant process hazards, larger samples of about 5-10g are heated to destruction in cells equipped with a pressure sensor. The focus of the study is to understand volume of gas evolved, when and how fast it's evolved in order to understand it's decomposition profile.

Process Understanding

Once decomposition risks and boundaries are established, it's necessary to understand the desired chemical reaction. Reaction Calorimetry determines how much heat is given out under normal operating conditions. The focus here is on heats of reaction,heat flows and gas evolution, with study results being the heat of reaction and heat release rate.
The cooling capacity of the reactor should also be checked to be sufficient to control the reaction temperature profile from the prior step and so avoid runaway situations.

Adiabatic Tests

Adiabatic conditions are a worst case scenario that assumes no heat loss from the process, but are quite realistic for large plant vessels that lose heat slowly. Here an adiabatic reaction calorimeter (A.R.C.) tests samples at minor to no heat loss conditions, and measures the largest temperature rise achieved to establish the worst case scenario if decomposition occurs. Depending on process specifics, a low phi adiabatic study can also be done to more closely mimic large plant conditions and determine vent sizing.

Modelling with software

The best software serve as a complement to all of the above studies by modelling scenarios to reduce the number of actual experiments, vastly speeding-up process safety testing.

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

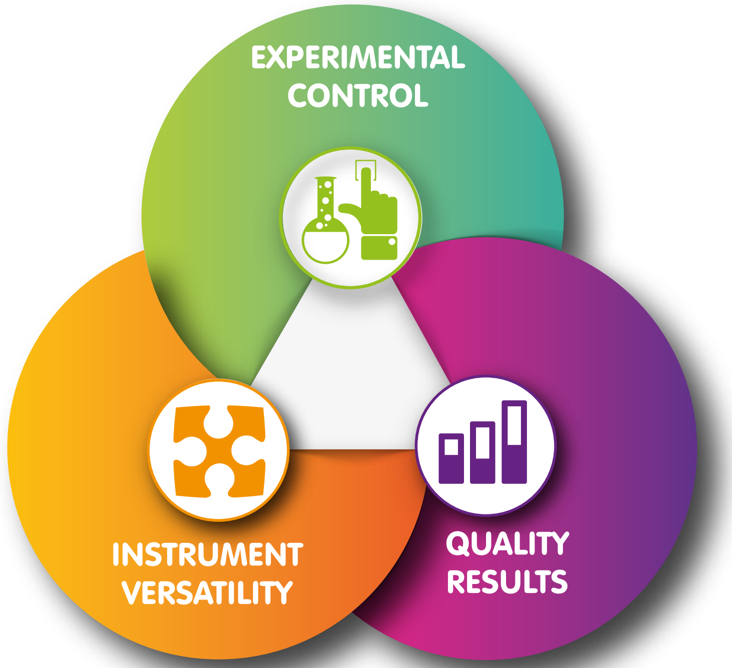
THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is radically step-changing it's 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 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 customer benefits, we are able to provide **customized solutions** by harnessing the engineering and project management of our highly skilled organization.



CUSTOMIZED SOLUTIONS

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

QUICK SCREENING

INSTRUMENT

Setline

- **EASY TO USE WITH ROBUST SENSOR TECHNOLOGY**
  ensuring quality, consistent and reliable data
- **AVAILABLE WITH HIGH PRESSURE CRUCIBLES**
  up to 500 bar at 600°C
- **REASONABLY PRICED INSTRUMENT & SENSOR**
  for easy, cost effective replacement
- **LOWER COST OF OWNERSHIP**
  through simplified maintenance and a Replacement Parts Guarantee
- **TECHNICAL & APPLICATION SUPPORT**
  for fast expert help with any questions
- **CALISTO 2.0 EXCLUSIVE SOFTWARE**
  for intuitive and easy data handling

SPECIFICATIONS

Temperature range (°C)	-170 to 700
Programmable heating rate (°C/min)	0.01 to 100
Enthalpy accuracy / precision* (%)	+/- 0.8 / 2.5
Temperature accuracy / precision* (°C)	+/- 0.30 / 0.50
DSC measurement range (mW)	+/- 6 000
Atmosphere	Inert gas, air, High-pressure crucibles up to 500 bar at 600°C
Autosampler	SETLINE DSC+ version featuring a 59 position autosampler

** 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

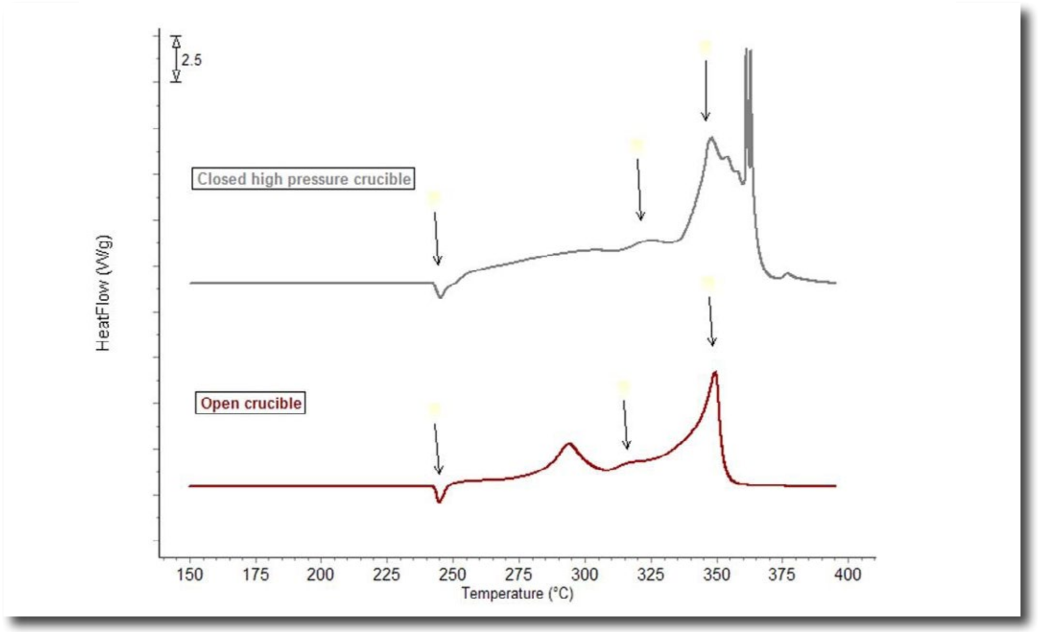
The thermal decomposition of Propergol was studied in a tightly closed high pressure crucible and in an open crucible with milligram-scale samples.

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...).



THERMAL STABILITY WITH PRESSURE AND EVOLVED GAS

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

SPECIFICATIONS

Temperature range (°C)	Room Temperature to 400
Temperature accuracy / control (°C)	0.01
Heating rate range (°C/min)	0.5 to 10
Modes	Temperature Scanning, Isothermal, Dual Scan
Pressure Range (bar)	0 to 200
Pressure Resolution (bar)	0.001
Pressure Accuracy (bar)	+/- 2

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

APPLICATION

DECOMPOSITION OF DTBP BY RAPID SCREENING CALORIMETRY

INTRODUCTION

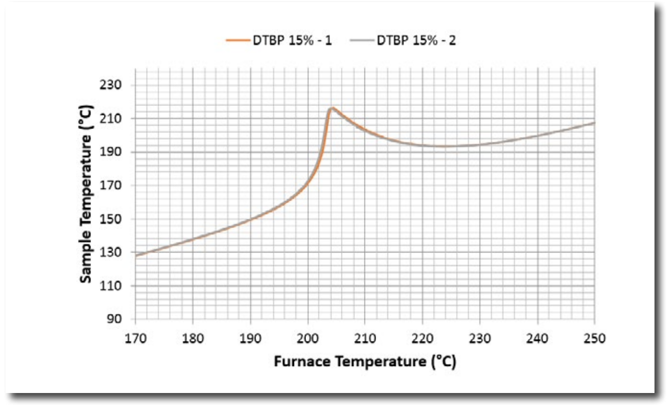
Di-Ter Butyl Peroxide (DTBP) is an unstable chemical used in the polymer industry to initiate polymerization reactions. As large quantities of such a product need to be stored at the plant, it is necessary to assess the risks with the thermal decomposition of this chemical. DTBP is also a typical compound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

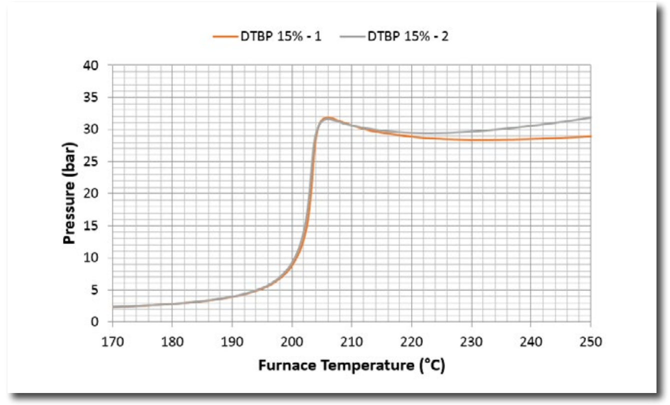
EXPERIMENT

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.





REIMAGINE MATERIAL CHARACTERIZATION

QUICK SCREENING
THERMAL STABILITY WITH PRESSURE AND EVOLVED GAS
PROCESS UNDERSTANDING

INSTRUMENT

CALVET

ISOTHERMAL OR TEMPERATURE SCANNING MODES
for increased flexibility

HEAT MEASUREMENT ACCURACY
with Calvet 3D sensor capturing 93-95% of all heat forms. The highest level on the market

CONVENIENT, INTERCHANGEABLE CELLS
to perform even the most demanding experiments using one instrument

WIDE TEMPERATURE RANGE
with low temperature version CALVET CRYO and high temperature version CALVET HT

SPECIFICATIONS

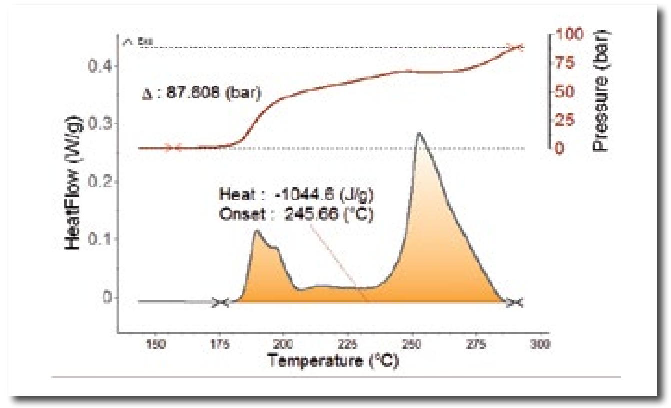
	CALVET	CALVET CRYO	CALVET HT
Temperature range (°C)	Ambient to 300	-196 to 200	Ambient to 600
Temperature accuracy (°C)	+/-0.3 *	+/-0.5 **	+/-1*
Temperature precision (°C)	+/-0.15*	+/-0.25**	+/-0.5*
Programmable temperature scanning rate	0.001 to 2°C/min	0.01 to 1°C/min	0.01 to 2°C/min
Enthalpy accuracy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

** 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

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.

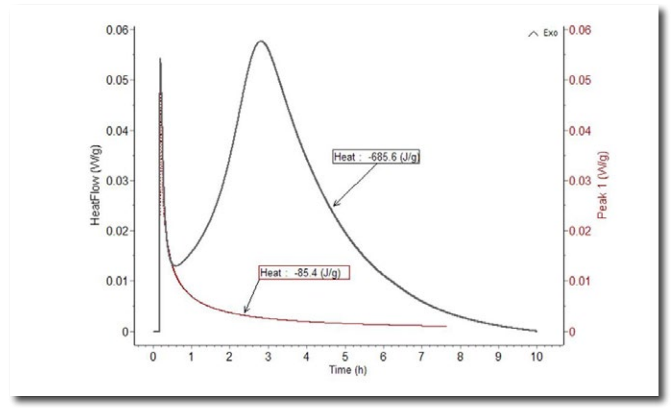
EXPERIMENT

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

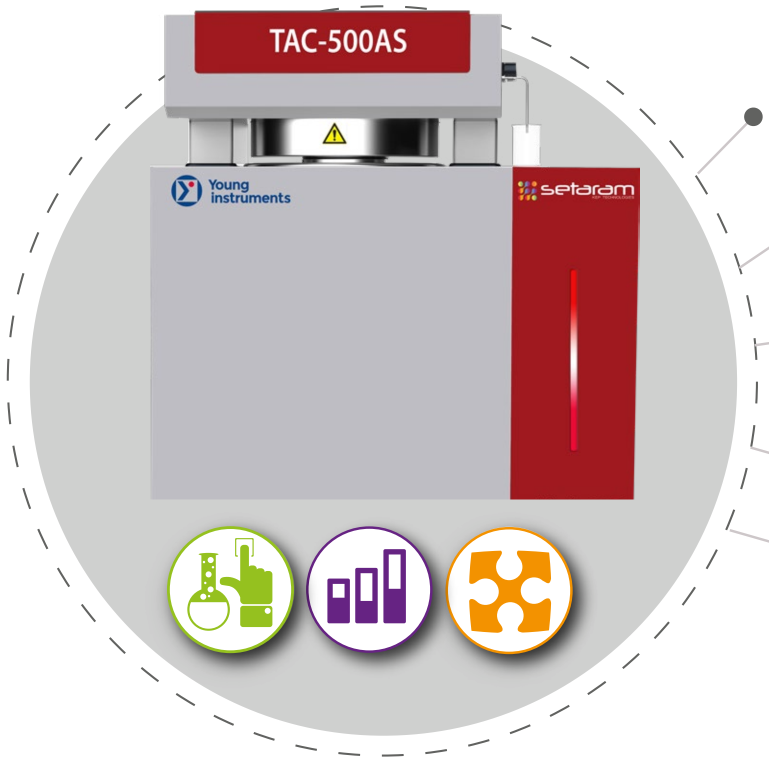
Vinyl Pyrrolidone and 4, 4'-azobis-cyanovaleric acid were placed in the two chambers of the cell, separated by a thin membrane. The calorimeter was run under isothermal mode at 50 °C. In-situ mixing was provided by piercing the membrane.

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.

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

Temperature range (°C)	Room Temperature to 500
Temperature increase detection Threshold (°C/min)	0.005 to 0.02
Temperature Precision (repeatability, °C)	+/- 0.05
Modes	Heat-Wait-Search, Isothermal, Temperature scanning
Pressure Range (bar)	0 to 200
Pressure Resolution (bar)	0.001
Pressure Accuracy (bar)	+/- 2

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

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
• One 5 g sample of a 40 w% DTBP solution in toluene

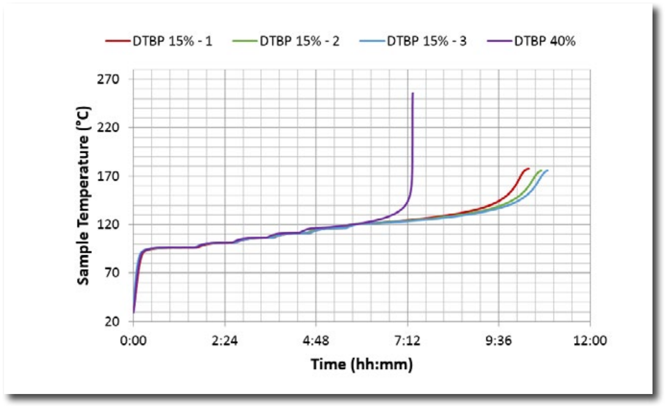
The Heat-Wait Search parameters were:

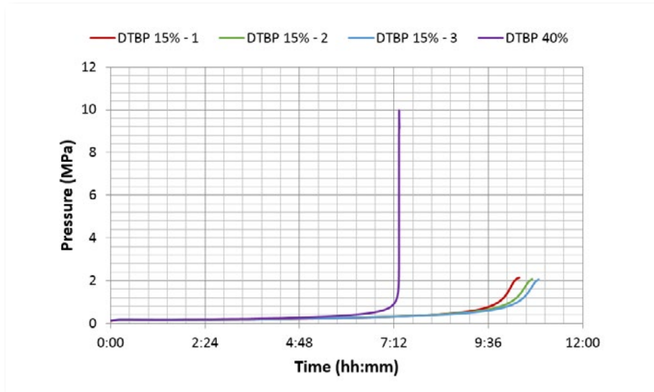
• Start temperature: 97 °C
• Temperature steps: 5°C
• Soak Time: 30 min, wait time: 30 min, search time: 15 min
• Detection threshold: 0.02 °C/min
• End Temperature: 250 °C (400 °C with the 40 wt% DTBP solution)

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.





	T_{onset} (°C)	$\Delta T_{ad, raw}$ (°C)	$\Delta T_{ad, corrected}$ (°C)	ΔT at max T rate (°C)	ΔT at max P rate (°C)	ΔT_{ad} (Mpa)
DTBP 15% -1	121.56	56.17	104.50	169.16	164.73	1.90
DTBP 15% -2	121.70	54.46	101.32	166.34	172.07	1.84
DTBP 15% -3	121.64	54.89	101.90	164.30	168.35	1.83
DTBP 40%	116.79	138.71	257.44	210.55	187.46	9.70

MODELLING WITH SOFTWARE

INSTRUMENT

AKTS Thermal Safety Software

KEP TECHNOLOGIES ALSO PROVIDES AKTS SOFTWARE
for precise modelling of runaway situations

WITH AKTS CALORIMETRY DATA SETS
provides the Self-Accelerated Decomposition Temperature (SADT), Time to Maximum Rate (TMR) and the full package of thermal hazards data can all be precisely predicted

ADIABATIC CALORIMETER
can be used as single experiment verification instead of for a series of experiments at the start of process evaluation as such reducing countless time and resources

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

APPLICATION

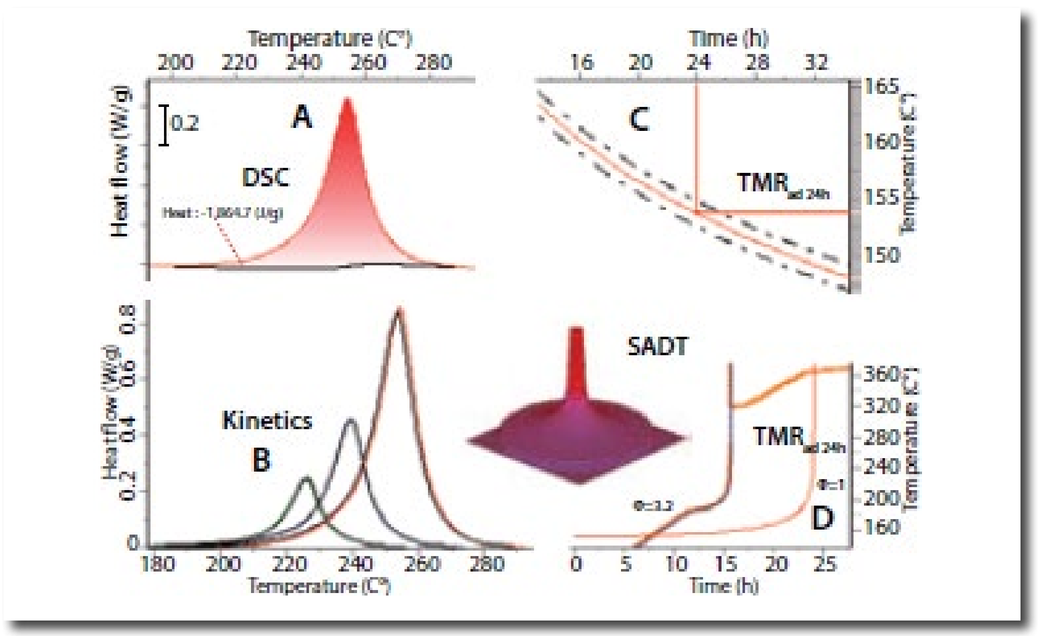
DECOMPOSITION OF 3-METHYL-4-NITROPHENOL USING DSC AND CALVET

INTRODUCTION

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).



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