



Thermal Safety Analysis

FlexyTSC with

RADEX V5 / V6 and SEDEX

The new SYSTAG FlexyTSC Safety Calorimeter provides accurate information about the thermal behaviour of substances combined with the easiest operating conditions. Whether investigating a large number of samples or one particular substance under a variety of conditions, FlexyTSC can be adapted to suit any requirements.



Application

Measuring Methods

The assessment of process safety and classification into appropriate risk categories is based on an evaluation by reaction calorimetry and soundly based thermal safety analysis.

FlexyTSC meets exactly these needs. It provides optimal support for qualitative and quantitative process safety investigations. FlexyTSC is extremely user friendly, and offers the necessary flexibility to meet the most varied requirements.

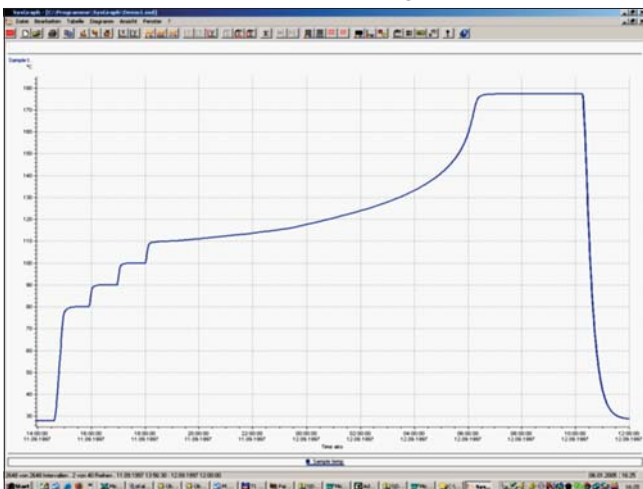
The FlexyTSC supports all standard methods of measuring. A number of options are available to configure the FlexyTSC, resulting in a flexible operation.

Independent from the measuring cell in use, the following **evaluation methods** can be utilised:

- ◆ Scanning
- ◆ Iso-peribolic steps
- ◆ Iso-peribolic long-term evaluations
- ◆ Adiabatic operation (optional)
- ◆ IsoARC method (heat-wait-search), optional

Up to 6 measuring cells of the same type or different type can be connected to the FlexyTSC control units. Combinations of 2, 4 or 6 cells are supported. Additionally, individual cells can be operated using different sample sizes and a variety of sample vessels.

Illustration 1 IsoARC Run



Measuring Conditions

Measuring Conditions

A further major advantage of the FlexyTSC is the capability to utilise all available measuring methods universally and multi-functionally.

Valuable time is saved as samples can be individually prepared and evaluations are started by mouse click.

Single samples can be evaluated under different conditions if more information of the thermal behaviour is required. Results can be subsequently displayed and compared in a single diagram.

Different conditions

- ◆ Open glass vessel
- ◆ Glass vessel, inert atmosphere
- ◆ Glass vessel with catalyst
- ◆ Glass vessel, measurement of gas evolution
- ◆ Autoclave with or without pressure measurement
- ◆ Autoclave with glass liner

The operator is provided with a large variety of useful information on the process or the substance under investigation for subsequent characterisation.

Illustration 2 RADEX Glass open / Inert gas evolution / Autoclave



Measuring Cells

RADEX V5

The RADEX V5 measuring cell permits cost-effective and safe evaluations within a range from ambient temperature to +400°C. Low volumes are used (typically 2,5ml; ranging from 1.5 to 3ml, depending on vessel). One major advantage of RADEX is the affordable cost of the vessels. It allows occasional discarding if cleaning is impossible.

The detection threshold for onset temperatures is lower than in a DSC.

The RADEX cell is used for evaluations of a typical volume of about 2.5ml. Non-homogeneous substances can therefore be reliably evaluated.

A large selection of different sample vessels and accessories is offered:

- ◆ Glass vessels open/closed
- ◆ Unit for gassing
- ◆ Gas volume measurement
- ◆ Steel autoclave up to 200 bars
- ◆ Pressure measurement
- ◆ Glass liner for autoclaves

RADEX V6

The RADEX V6 measuring cell permits cost-effective and safe evaluations within a range from -50°C up to +190°C. The well-insulated RADEX V6 prevents freezing of the vessel and erroneous measurements.

The detection threshold for onset temperatures is lower than in a DSC.

The RADEX cell V6 is used for evaluations of a typical volume of about 2.5ml in a pressure vessel. Non-homogeneous substances can therefore be reliably evaluated.

SEDEX

SEDEX is the most versatile measuring cell. It is equipped with built-in illumination for progress monitoring and a magnetic stirrer. By using an oven with circulating hot air, solid substances can be evaluated for stability in a wire cage (transport stability etc).

The large size of the oven allows the installation of customer-specific vessels, even with dosing facilities. Optional thermostat controlled cooling coils provide cooling of the oven interior. This option changes the temperature range from +35°C...+400°C to -10°C...+150°C.

Illustration 3 RADEX cell V5 (left) and V6 (low temperature)



Illustration 4 SEDEX



Experimental Control

Method Selection

Intuitive execution of experiments is supported by selecting the most suitable measuring method (Scanning, Iso-peribolic, Adiabatic), with a single mouse click. Uncomplicated operation, starting values and heating speeds can be stored as default parameters.

Data Recording

The continuous data recording function permanently stores every input such as sample weight, sample description, vessel type etc. This greatly reduces the chance that sampling results are mixed up. The format of the report configurable to suit individual requirements.

Calibration

Each cell and vessel type can be calibrated. Methods for SCANNING and ISOPERIBOLIC are provided.

The calibration with inert substances permits the best possible correlation between sensors.

Appropriate reference substances are used to calibrate vessels for quantitative evaluation (mW/g) of measurements.

Illustration 5: FlexyTSC Report File

LABOR JOURNAL		SYSTEM	
Lab-Nr.		Projekt	
Technique		Exp. Nr.	
Chem. Name			
Abkürzung			
Art of Exp.			
Experiment:			
Type	Analysis		
Analysis	IsoARC_Versuch_01		
Rezept	IsoARC		
Linearisierungstabelle	Oxas_Vessel_IsoPeribol_041130_OVEN1		
Defekt-Nr.	Oven 1		
Analysedatum	10. Dezember 2004 08:53:43		
Probenbeschreibung			
-			
Erwäge	1.51 g		
CP der Vorlage	1.139K		
Aspekte vor der Messung			
-			
Rückwäge	1.3 g		
Aspekte nach der Messung			
-			
Defekt-Typ	RADEX Oxas Vessel 16mm open		
Zufuhr	None		
Atmosphäre	Air		
Defekt	No		
Defekt	No		
Defektgeschwindigkeit	14 min		
Operator	Fs		

Operational Control

On-line Display

The FlexyTSC On-Line Display directly shows the output power of the reaction in progress. The display thus provides valuable information about the potential of a substance even during initial measurements - without the need of further analysis. This is helpful, especially in the case that a sample of a continuous production process has to be evaluated.

Illustration 6: Input dialog for IsoARC method (heat-wait-search) also suitable for multi-step decomposition (blue graph)

Illustration 7: Main display with user interface

Results

Heat-flow

„ThermoGraph“ evaluation software determines the decomposition output and the resulting heat. If several measurements of the same substance are carried out under different conditions, ThermoGraph generates a single graph. Comparisons can be made for individual ovens or a number of measurements. Results can be selectively displayed and calculated in absolute or standardised mode (e.g. J/g).

All results and diagrams can be directly printed or easily transferred via clipboard into other applications. Meaningful analyses are generated with a few mouse clicks.

Adiabatic Evaluations

A mouse-click is sufficient to generate the following results for a substance that was evaluated under adiabatic conditions:

- ◆ Actual adiabatic temperature increase
- ◆ Arrheniusplot
- ◆ Activation energy
- ◆ Self Heating Rate (SHR)
- ◆ Time-to-Maximum Rate (TMR)

Illustration 8: Onset comparison

Sample	Onset Temperatur [°C]		
	ARC	RADEX	DSC/DTA
t-Butylperoxy benzoate	86	90	122 (A)
Benzoylperoxide	86	98	105 (B)
4-Chloro 2-nitrobenzoic acid	241	253	240 (A)
di-t-Butylperoxide	115	124	125 (A)
2-Nitrophenol	261	258 (A)	
Hydroxylamine sulphate	131	159	185 (A)
Acodicarbonamide	168	196 (A)	
		(A) DSC	(B) DTA

Onset Temperature

By using the standard graph „Temperature difference vs. Measuring cell temperature“, the onset temperature is easily determined.

Process Safety

The recorded values and results supplied by the FlexyTSC are prerequisites for a reliable characterisation of a substance or substance blends. It accurately determines the suitability for a production process. Furthermore, FlexyTSC supports the safety specialist with the identification of external influences on safety due to deviations in the sample quality.

Illustration 9: Time to Maximum Rate Graph; identifies time to run-away as a function of temperature.

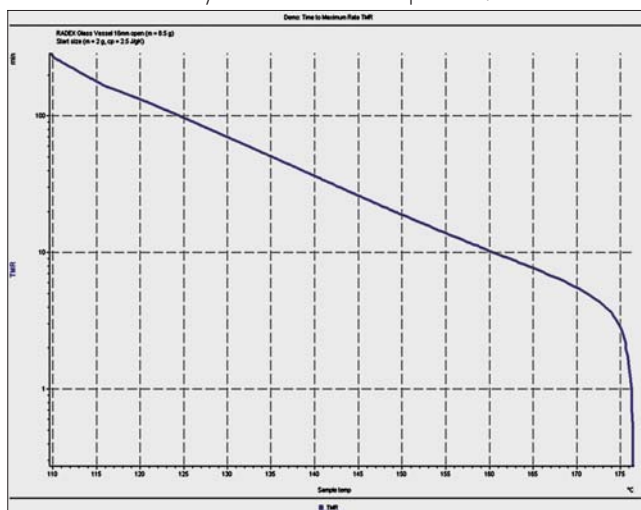
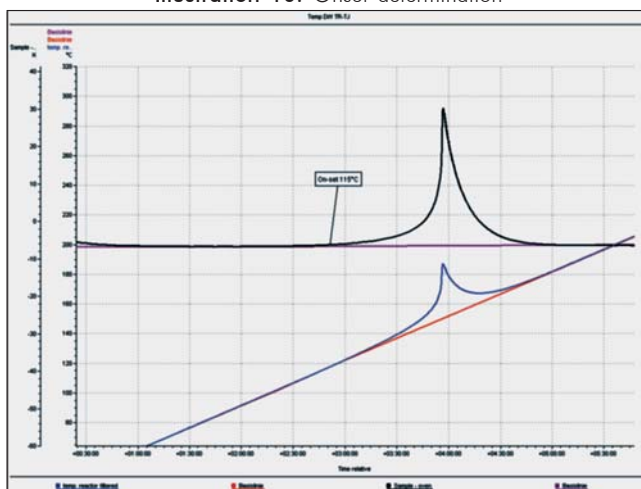


Illustration 10: Onset determination



Accessories

Gas flow measurement

The FM4 gas flow meter displays the volume of evolving gas. The FM4 provides a standardised signal for data-recording purposes. The FM4 measuring method is suitable for managing the smallest volumes of discontinuous gas flow.

The gas flow measuring instrument is designed to accommodate flow rates of up to 4 l/h for the FlexyTSC application and up to 60 l/h for reaction calorimeters.

No physical characteristics of the gas mixture are required to perform the measurements, since the sensor operates using a strictly volume-based technique.

An accuracy of between 0.5 % and 1 % is achieved depending on the measurement range. The FM4 is designed to be used in pressure-free environments.

Illustration 11: FM4 gas flow measuring instrument



Assembly Tool Kit

These tools assist with the proper assembly of RADEX pressure vessels. The collet holder, used for securely fixing the lower section of the pressure vessel, and the torque wrench, used for generating the correct tightening torque, are especially important. The illustration below shows the complete set-up (pressure vessel, capillary and pressure sensor) for the pressure measurement application.

The correct handling of the couplings ensures a long service life. For this purpose, assembly instructions are provided.

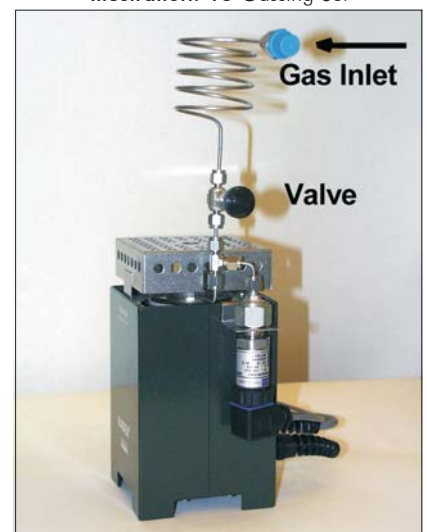
Illustration 12: Tool kit for assembly of RADEX pressure vessels



Gassing Set

In case you need to test your sample under gas pressure (N₂, O₂, He, etc.) we are able to supply the appropriate fittings. An example for such an application is illustrated below.

Illustration: 13 Gassing Set



Technical Data

Measuring Cell RADEX®V5	Temperature range	RT ... 400°C
	Temperature measurement	Pt-100 probes for internal and external temperature
	Sensitivity	0,05°C or 2 mW of sample
	Choice of vessels	- Glass, gassings inserts available - V4A Steel for evaluations under pressure, with bursting foil, pressure measurement
	Net volume	2.5 ml (1.5 to 3 ml)
	Construction	V4A steel jacket with cylindrical, heated metal block
	Cooling	Cooling fan for use at the end of an experiment
	Dimensions	250 x150 x150 mm (height x width x depth)
	Weight	approx. 3.5 kg
	Standard vessel	Glass vessel Optional: pressure vessel for up to 200 bars (with or without glass liner); closed glass vessel

Measuring Cell RADEX®V6	Temperature range	-50 ... +190°C, min and max temperatures depend on thermostat/parameters
	Temperature measurement	Pt-100 probes for internal and external temperature
	Sensitivity	0,05°C or 2 mW of sample
	Vessel	V4A Steel, 200 bar, for evaluations under pressure, with bursting foil
	Net volume	2.5 ml (1.5 to 3 ml)
	Construction	V4A steel jacket with cylindrical, heated metal block
	Cooling	External thermostat
	Dimensions	340 x 200 x 340 mm (height x width x depth)
Weight	approx. 12.8 kg	

Measuring Cell SEDEX®	Temperature range	RT ... 400°C, with cooling -10°C...+150°C
	Temperature measurement	Pt-100 probes for reactor and jacket temperature
	Sensitivity	0,05°C or 0,5 mW/g of sample
	Choice of vessels	- Glass, Gas flow measurement optional - V4A steel, for evaluations under pressure, with bursting disc, with pressure measurement - Wire cage for storage and transport evaluations - Adiabatic insert for controlling very slow increases
	Net volume	20 ml (ranging from 2 to 100 ml or more)
	Construction	Air circulation oven with pressure relief and inertisation facilities
	Cooling	Cooling unit for experiments to approx. -10°C
	Stirrer	Magnetically operated
	Dimensions	530 x 420 x 500 mm (height x width x depth)
	Weight	approx. 54 kg
	Standard vessel	Glass, open Optional: pressure vessels to 150 bars

Configurations / Options	FlexyTSC
For use with RADEX V5 RADEX V6 (low temp) SEDEX	● ● ●
Calibration Scanning Isoperibolic	● ●
Methods Scanning Isoperibolic Adiabatic IsoARC method	● ● ○ ○
Online Display Temperatures Output Pressure * Gas volume *	● ● ● ●
Evaluation Output Heat TMR SHR Arrhenius Activation Energy	● ● ○ ○ ○ ○ ○
Various Max. No. Cells per PC Temp.-Resolution Delta-T Resolution +/-	6 0.01K 0.01K

- Standard
○ Optional

* mutually exclusive, can be configured

Notes
