

FlexyTSC

Thermal Safety Analysis with RADEX V5 und 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.

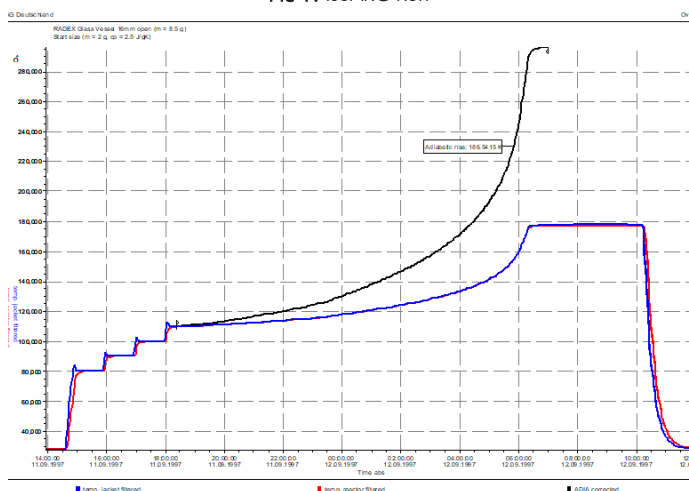
The FlexyTSC supports all standard methods of measuring. A number of options are available to configure the FlexyTSC, resulting in a flexible operation.

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.

Pic 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.

Pic 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 measuring cell V5 is typically used to examine a sample volume of approx. 2.5 ccm, which also allows the examination of inhomogeneous samples.

Sample preparation

Thanks to the relatively large sample volume and the large opening of the sample vessels, sample preparation is no longer an essentially important step. Even rough-grained samples can be processed quickly and easily without special physical processing such as grinding or milling.

Steel as a catalyst

For all steel vessels (SS316/HC22) for RADEX V5 as well as for SEDEX, glass liners are available to avoid catalytic influence on the sample under investigation. Besides preventing the catalytic effect, the inliner also protects the steel vessel against strongly oxidizing substances. Depending on the requirements, analysis can be performed with or without a glass inliner.

Pic 3: RADEX cell V5



Different test cells

A large selection of different sample vessels and accessories is offered for RADEX as well as for SEDEX:

- Glass vessels open/closed
- Unit for gassing
- Gas volume measurement
- SS316/HC22 autoclave up to 200 bars
- Pressure measurement
- Glass liner for autoclaves
- Wire basket (only for SEDEX)

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.

Pic 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.

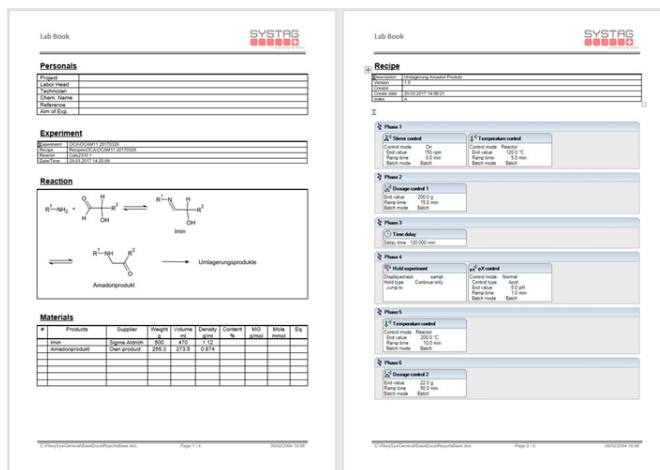
Calibration

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.

Pic 5: FlexyTSC Report File

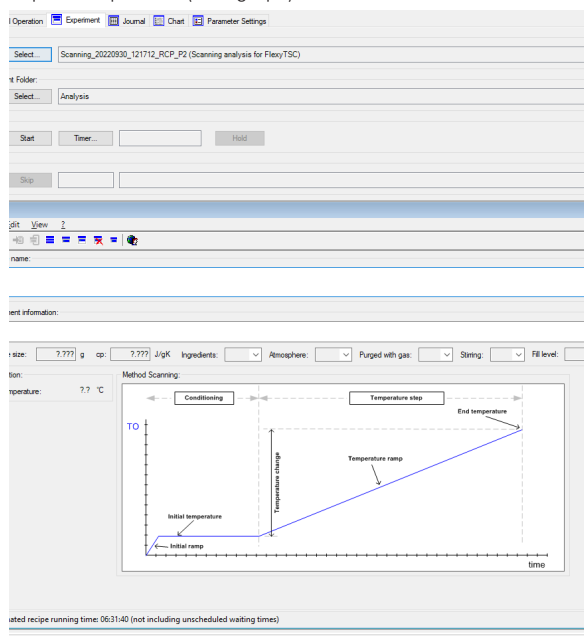


Operational Control

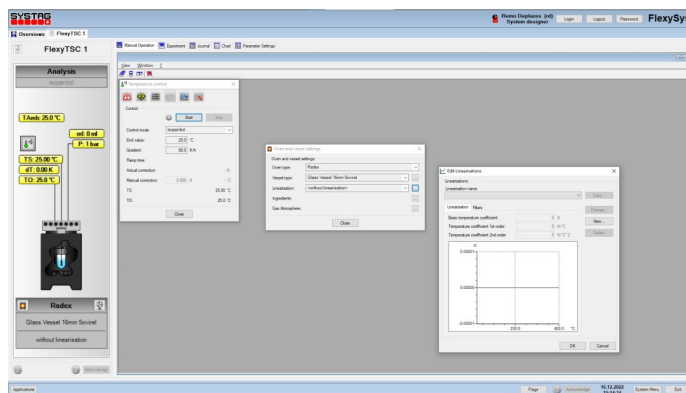
On-line Display

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.

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



Pic 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
- Arrhenius plot
- Activation energy
- Self Heating Rate (SHR)
- Time-to-Maximum Rate (TMR)

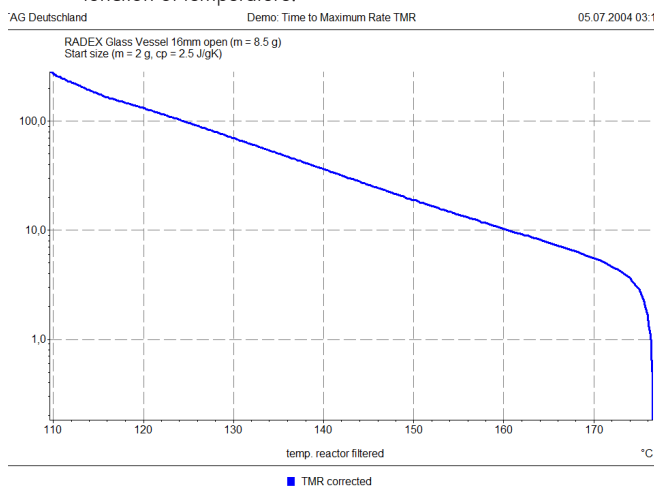
Onset Temperature

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

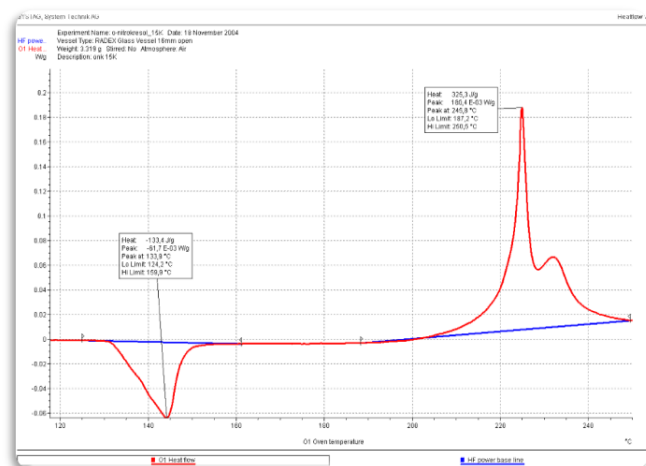
Process Safety

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.

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



Pic 10: On-set determination



Pic 8: Onset comparison

Probe	On-set Temperature [°C]		
	ARC	RADEX	DSC/DTA
t-Butyl-Peroxybenzoat	86	90	122 (A)
Benzoylperoxid	86	98	105 (B)
4-Chlor-2-Nitrobenzoesäure	241	253	240 (A)
di-t-Butyl-Peroxid (DTBP)	115	124	125 (A)
2-Nitrophenol		261	258 (A)
Hydroxylaminsulfat	131	159	185 (A)
Acodicarbonamid		168	196 (A)

(A)DSC (B) DTA

Accessories

Gas flow measurement

Optionally, a commercially available gas clock can be connected to the FlexyTSC by means of an analog interface. Depending on the signal used, the gas flow or the integrator is recorded. This value is of course also available in the on-line / off-line diagram.

Specific gas atmosphere

In order to examine samples under different gas atmospheres, RADEX offers among other things the possibility of a gassing set. With this optional accessory a sample can be analyzed e.g. under inert conditions (N₂) or under air-oxygen, or any other gas. This provides the chemist with additional information about the behavior of a substance under different conditions, which can sometimes be very important for classifying the thermal stability of a substance.

Gassing Set for autoclave

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.

Pic 11: Gassing-Set glass

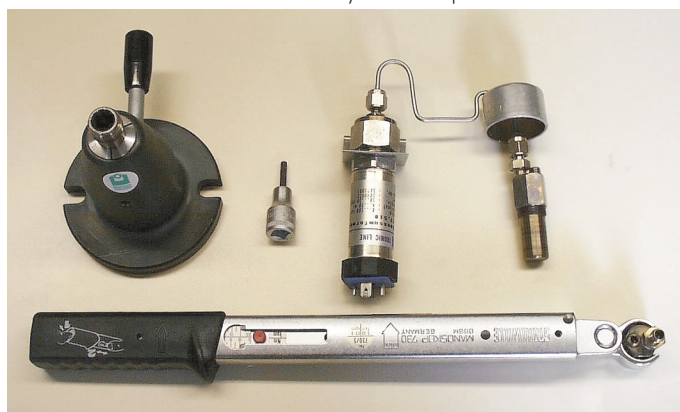


Assembly Tool Kit

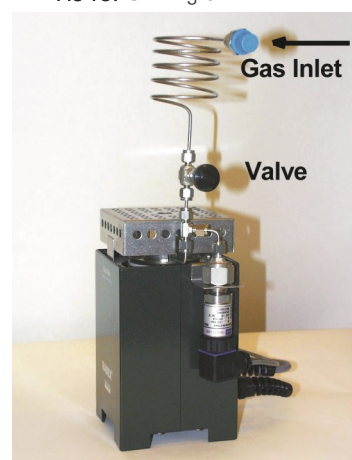
These tools assist with the proper assembly of RADEX pressure vessels. The collet holder, used for securely fixing the lower section of the pressure vessel, and the torque wrench, used for generating the correct tightening torque, are especially important. The illustration below shows the complete set-up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

Pic 12: Tool kit for assembly of RADEX pressure vessels



Pic 13: Gassing-Set autoclave



Technical Data

RADEX V5	Temperature range	RT... +400°C
	Temperature measurement	Pt-100 probes for internal and external temperature
	Sensitivity	0,05°C or 2 mW/g sample
	Choice of vessels	- Glass, gassings inserts available - V4A Steel for evaluations under pressure, with bursting foil, pressure measurement
	Net volume	2.5 ccm (1.5 to 3 ccm)
	Construction	V4A steel jacket with cylindrical, heated metal block
	Cooling	Cooling fan for use at the end of an experiment
	Dimensions	250 x 150 x 150 mm (height x width x depth)
	Weight	approx. 3.5kg
	Standard vessel	Glass vessel open Optional: pressure vessel for up to 200 bars (with or without glass liner); closed glass vessel
SEDEX	Temperature range	RT ... 400°C, with cooling -10°C... +150°C
	Temperature measurement	Pt-100 probes for internal and external temperature
	Sensitivity	0,05°C or 0.5mW/g 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. 54kg
	Standard vessel	Glass vessel open Optional: pressure vessel for up to 200 bars (with or without glass liner)

Configuration/ Options	FlexyTSC
For use with RADEX V5 SEDEX	● ●
Calibration Scanning Isoperibolic	● ●
Methods Scanning Isoperibol Adiabatic IsoARC method	● ● ○ ○
Online Display Temperatures Power Pressure* Gas volume*	● ● ● ●
Evaluation Power Heat TMR SHR Arrhenius Activation Energy	● ● ○ ○ ○ ○
Various Max. no of Cells per PC Temperature resolution Delta-T resolution +/-	6 0.01K 0.01K
● standard ○ optional	
* mutually exclusive, can be configured	

Notes
