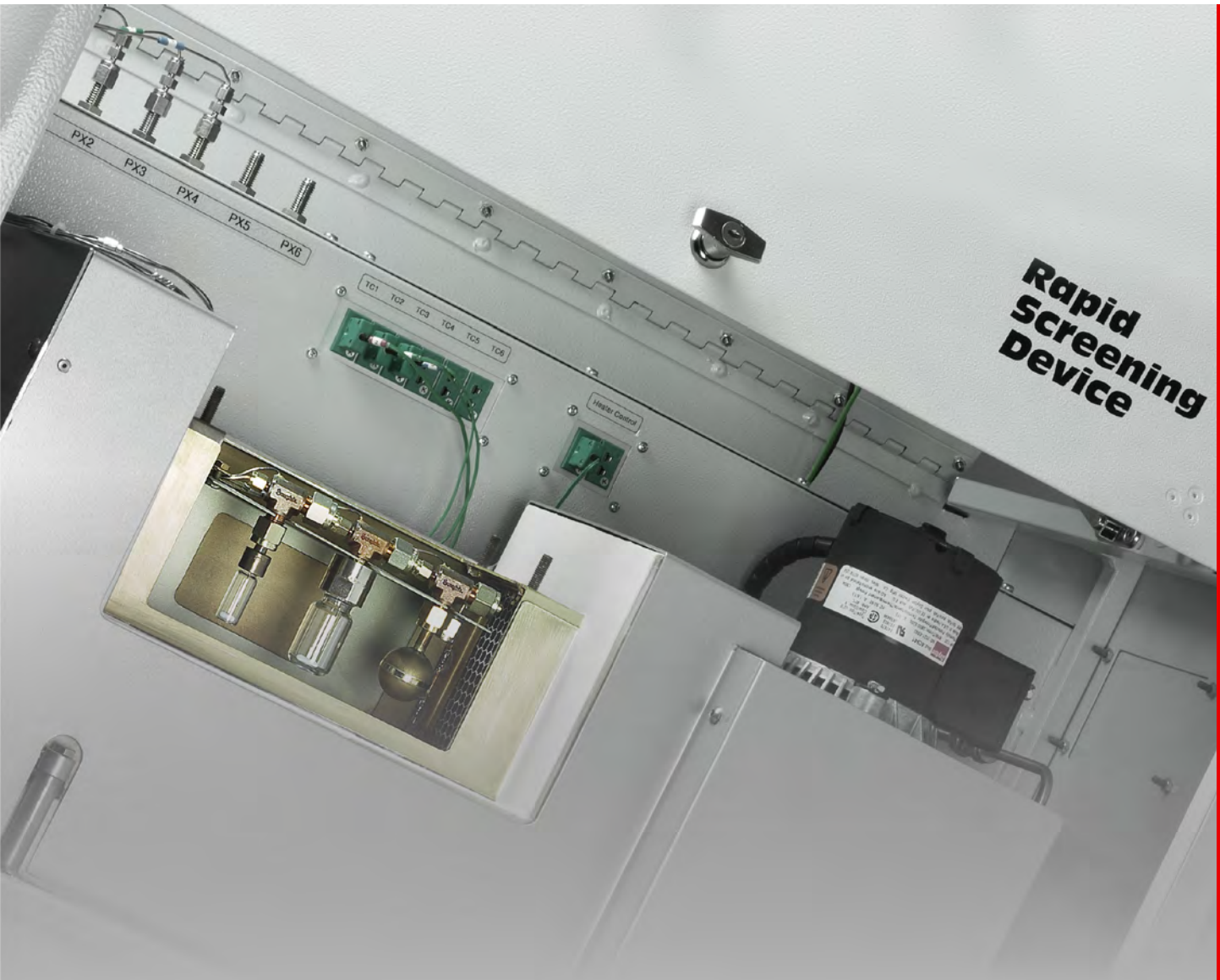
The THT Rapid Screening Device
Safety Calorimeter RSD



- 6 samples / references simultaneously
- Scanning Calorimetry
- Isothermal Calorimetry
- Dosing, Stirring, Cryogenic
- Temperature & Pressure
- mg to 100g sample size

- all in one instrument

RSD RAPID SCREENING DEVICE

thermal hazard technology

Introduction to the THT RSD

Novel • Rapid • Sensitive • Versatile • Inexpensive

A unique calorimeter with OPTIONS!

OPTIONS

- Cryogenic operation to -100°C
- Stirring
- Dosing
- Gas Release / Collection

The THT Rapid Screening Device (RSD™) is the most versatile hazards safety calorimeter.

This brochure shows how using the RSD will help chemists and engineers quantify exothermic (and endothermic) reactions from materials of low hazard to high explosives!

Compared to DSC, the RSD takes much larger samples and pressure measurement is standard.

Compared to Dewar, oven and home built calorimeters, the RSD is quantitative, safe in use and industry recognised.

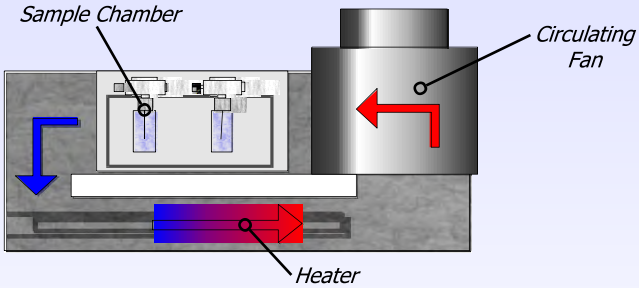
Compared to other screening safety calorimeters, the RSD takes up to 6 samples simultaneously, has design features to provide comparative and differential data of highest quality and uniquely incorporates multiple safety features.

UNIQUELY UP TO 6 SAMPLES SIMULTANEOUSLY

The RSD has a thermally controlled robust calorimeter sample chamber. This will allow up to 6 samples with 6 thermocouples and 6 pressure transducers. Sample holders may be tube holders (mg), glass or metal ARC™ bombs (1g-10g), or simply a larger vessel (10-100g)

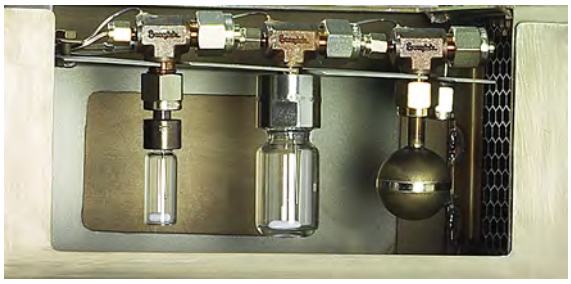
HOW IT WORKS

The heated calorimeter chamber is closed with a circular pathway to allow air (or inert gas) to circulate repeatedly during the test. The gas passes through the sample chamber and over the main heater. Circulation is achieved by a high temperature fan and a control thermocouple regulates power to the heater to implement the test protocol.



WHAT TO TEST

Typically a sample (in solvent) plus reference (solvent alone or air). Maybe 3 or 4 samples (differing batches, concentration, suppliers) against 1 or more references. Liquids, solid, mixtures, gas/liquid, pastes, any sample type!



Hardware & Software
Using the RSD

TEST PROTOCOLS:
SIMPLE, RAPID AND AS EASY AS 1, 2, 3...

1. (ONE)
Choose from the range of holders best suited to your sample. An ARC bomb (Stainless Steel, Titanium, Hastelloy, Glass), a simple tube, a glass 2ml or 4ml vial – a re-useable cylindrical holder, a gas flow holder – or simply put the sample in any suitable container. Weigh the sample into the holder and connect to the 'RSD head'.

Repeat this with further samples, a reference material, an empty holder and put these into the holding tray. Connect the thermocouples and gas lines, close and screw down the calorimeter lid. Shut the outer cover.

2. (TWO)
Using the industry standard National Instruments LabVIEW software set up protocol can be quickly completed (typically 2 minutes). Enter sample information (for each sample), then the test conditions, start and end temperatures, cut out temperature, pressure.

3. (THREE)
Click Start and relax; maybe check the test is going to plan or 'on-the-fly' change conditions as needed. The RSD has multiple safety interlocks. One or two hours later the test may be finished automatically and the system cooled or the test can be terminated manually. Analyse the data on this PC or transfer to your memory stick.

ANALYSIS: NI LABVIEW BASED FOR EASE OF USE
SIMPLE, RAPID AND AS EASY AS 1, 2, 3...

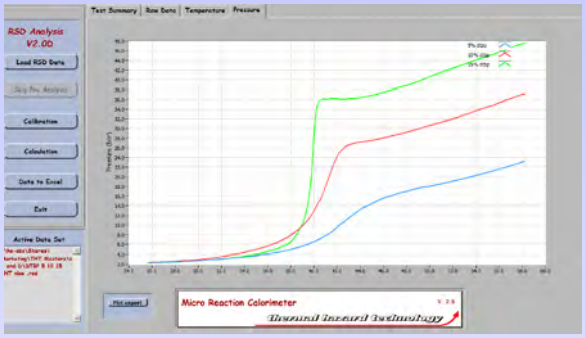
Open data on any PC with the RSD

1. (ONE)
Pre-analysis: remove data not needed for analysis and Save.

- Cut
- Filter
- Size

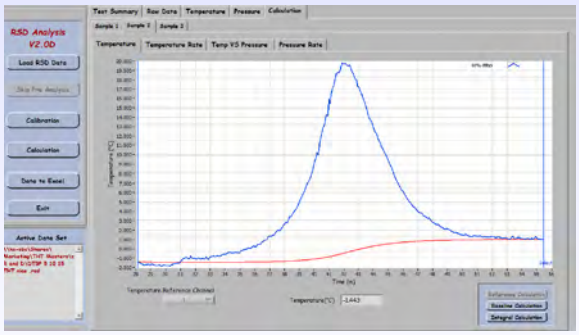
Qualitative analysis: present the sample data in tabular and graphical form.

- Temperature against time
- Pressure against time
- Temperature rate against temperature

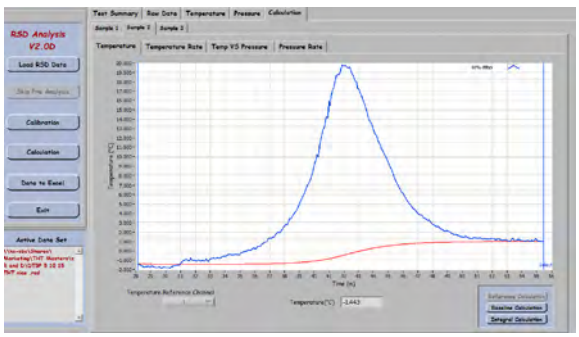


2. (TWO)
Differential analysis
Select the data set for analysis and the reference. The reference may be air, an inert solvent, or a sample from a different batch.

Generate graphs where the reference data is subtracted from the sample data – just as is done in DSC



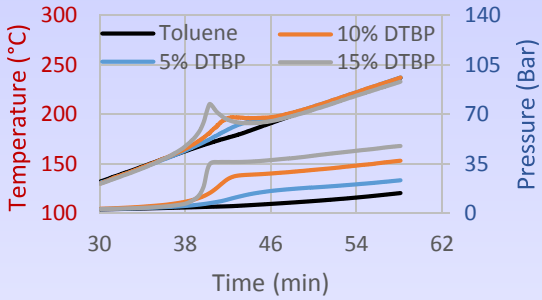
3. (THREE)
Quantitative analysis
Finally where appropriate, use the inbuilt thermodynamic and kinetic routines to determine heat of reaction, activation energy if the data allows this (please do not over-analyse complex data). Some data is not suited to quantitative analysis.



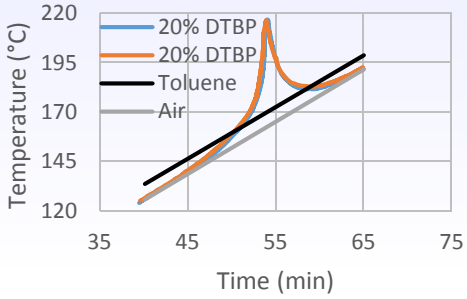
Performance illustrated with DTBP

UNRIVALLED PERFORMANCE COMPARED TO ANY OTHER SAFETY SCREENING CALORIMETER

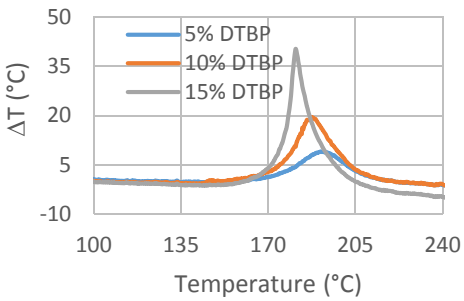
Di-tertiary Butyl Peroxide (DTBP) is an ideal sample to evaluate performance of a safety calorimeter. Exothermic decomposition of DTBP leads to explosion. Dilution, normally with toluene, provides a range of samples with varying Heat of Decomposition. Scientific literature reports show spread in thermokinetic parameters, values are dependent upon experimental protocol; however decomposition of DTBP is often taken as first order with a Heat of Reaction near 1200 J/g.



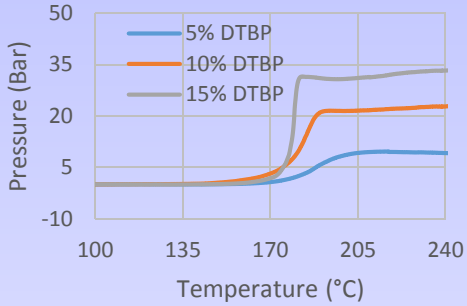
Raw data from a test with 4 samples 0%, 5%, 10% and 15% DTBP is shown above; data from 2 samples of 20% DTBP with toluene and air (as recorded inside the calorimeter chamber) are shown below; this illustrates reproducibility, shows no 'cross-talk' with the toluene and the chamber air temperature.
In the first stage of data analysis, results can be plotted against temperature and most usefully 'differential data'; ie the DTBP data minus the toluene data; to show heat from DTBP alone.



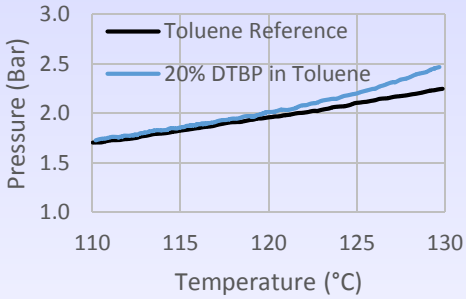
This is uniquely possible with the RSD and RSD RAP software and is illustrated as ΔT (DTBP-Toluene) Vs Temperature of toluene with 5%, 10% and 15% DTBP.



Pressure data as ΔP (DTBP-Toluene) Vs Temperature of toluene for 5%. 10%, 15% DTBP.

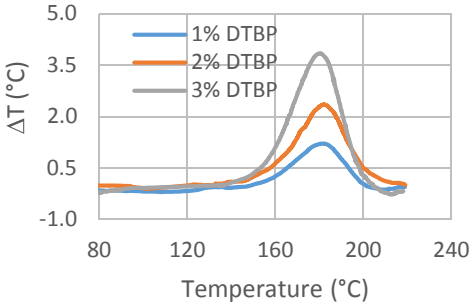


Ramp heating tests typically cause onset of reaction to be recorded at higher temperature than step heated (ARC-type) tests. Closer inspection of RSD data shows this with the thermal data more so than the pressure data.



DTBP data at low concentration is equally interesting. Classic safety calorimeters are sensitive, but due to test conditions the RSD can detect a smaller heat release than the DSC, ARC or similar systems. In the RSD the thermocouple is in the sample and the temperature ramp forces heat release over a short time.

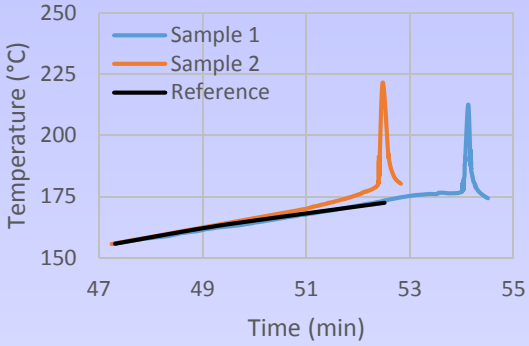
Below RSD results for one test with 1%, 2% and 3% DTBP in toluene. No heat could be detected in other calorimeters; 1% would equal a heat release from a sample whose Heat of Reaction is 12 J/g. The RSD will detect heat release to close on 1 J/g or 5J. Such data illustrates the ability of the RSD to quantify hazards or heat release associated with 'waste streams'.



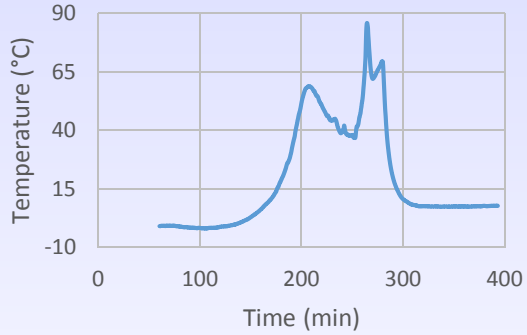
Applications Data

Quantitative, Flexibile and Versatile - not just DTBP and simple samples!

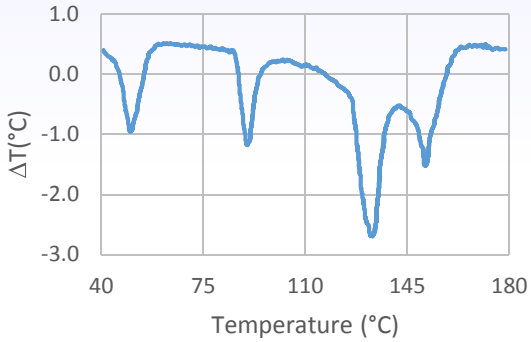
The flexible RSD can be used for varied applications. The previous page detailed not just a simple exothermic material but also low energy (waste stream) application.



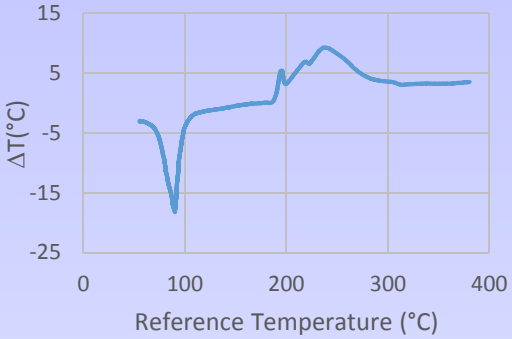
Data above shows an application to high energy explosives. Two 'similar' explosives run in one test; Sample 1 is a fresh sample, Sample 2 has been aged to simulate 6 months storage at high ambient temperature. The aged sample is less stable.



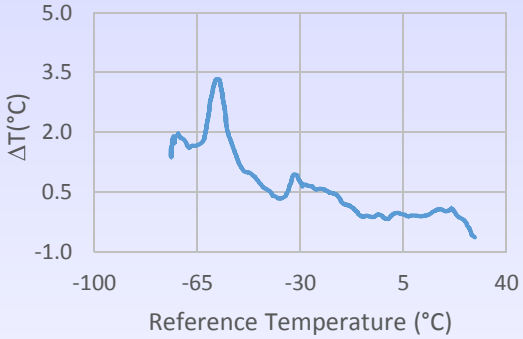
Oxidation example; illustrates heat of oxidation of a powdered coal sample in a flow of oxygen at 60ml/min



Ammonium Nitrate; illustrates RSD testing of samples undergoing endothermic processes. Ammonium nitrate has two phase transitions prior to melting and decomposition.



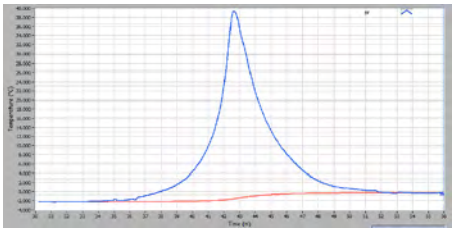
Complex organic chemical; illustrates melting below 100°C followed by multi-stage exothermic decomposition above 180°C.

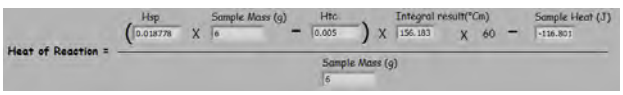


The RSD at cryogenic temperature. The 'Swern reaction' mixture, shows an exothermic reaction heated from -100°C

THERMOKINETIC DATA ANALYSIS

RSD RAP software has full routines for thermo kinetic data analysis. Determination of Heat of Reaction, Activation Energy, Order of Reaction. There are various baseline constructions and ability to make data correction based upon sample holder used. Nevertheless reliable quantitative data will only be observed if the reaction is singular and obeys Arrhenius kinetics.





Options and Specification

OPTIONS

RSD capability is enhanced with low cost options. These extend the range of applications.

LIQUID DOSING

Liquid dosing at ambient or low pressure is achieved by manually operated valves. The added liquid is pre-equilibrated to the same temperatures as the sample. Volume dosing can be done up to 50ml.

GAS FLOW

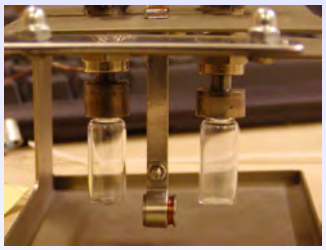
Gas flow during the test may be achieved at ambient or elevated pressure. Elevated pressure flow requires more sophisticated control, flow cells are available from THT. For example they allow flow of air (or oxygen) to study oxidation or combustion reactions.

CRYOGENIC

Extending the temperature range below ambient is possible and temperatures below -100°C can be achieved with control of liquid nitrogen / cold nitrogen gas flow. Such capability is unique to the RSD.

STIRRING

The RSD stirring assembly is positioned between 2 sample holders and therefore will stir both. Stirring can be achieved in both glass and metal sample holders. Stirring will homogenise 2 non-miscible liquid phase or disperse liquid/solid
2-phase samples. Stirring speed can be varied as required.



GAS COLLECTION

The gas collection option is manually operated. Allowing residual gas collection after (or during) a test. Gas collection from up to 6 samples is possible. The 10-25ml collection cylinders are 'quick release' to allow transfer to a gas analyser.

SPECIFICATION

Temperature Range	-100 to 500°C
Modes of Operation	Isothermal, step, scanning
Scan Rate	0 - 10°C/min
Detection Sensitivity	Less than 10 J/g
Pressure Range	Up to 160 bar (alternative ranges available)
Pressure Resolution	0.2 bar
Sample Size	>20mg
Sample Quantity	1 - 6 samples
Stirring	Up to 2000 rpm
Software	Dedicated Windows based control and data analysis software through link to PC
PC Requirements	Windows USB Port Monitor Resolution 1280 x 1024
Footprint	70 (W) x 43 (H) x 40 (D) cm
Power Supply	Single phase, 200-240V, 13A

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