The RSD – Operation and Application – more details

To supplement the RSD information, this document gives some details of how the unit works and what and how one can test. More info will be available in specific documents, TANs and Manuals – all available from THT – but this is a summary of some salient points

Design guidelines!

- Criteria considered in the design:
  - Encompass All Sample types
  - Multiple samples in 1 test
  - Variety sample holders (low cost)
  - Measure Temperature to 400°C
  - Measure Pressure to 180bar (2500psi)
  - Various atmospheres...
  - Insert or reactive gas, high pressure reactions
  - Measurement under gas flow (calibration)
  - Difference measurement of T and P
  - Scanning and isothermal (0-100min)

- But also to allow...
  - Sample mixing, stirring (2-phase samples)
  - Sample addition, dosing
  - Cryogenic operation, from -100°C

Features to allow:
- Low purchase price
- Ease of use
- Low running cost
- Simple maintenance
- Quantitative Operation
- Ease of Data Analysis

But the product must be:
- Flexible
- Versatile
- Innovative
- Operator friendly
- Operator safe

Product...

How it works...
- The RSD is a Ramped Passive-mode Calorimeter (RPmC), which depends on the principles of heat flow and transient heat accumulation.
- The key to this technique is a very stable heat transfer coefficient. The heat transfer capacity can thus be modelled successfully during the entire reaction period.
- The RSD measures temperatures and pressures, and as such is capable of providing a real-time basis for determining the partial pressures resulting from condensable vapours and non-condensable gases.
Note the sample container, its connector block (head) is fully in the hot zone – to eliminate issues with heat loss. A number of ‘low cost safety calorimeters have been devised, THT with the RSD has aimed to eliminate the ‘negative points’ associated with each of these (heat loss is a typical one!)

Sample types – any type of sample holder in the chamber!

Therefore sample size may be mg to kg – explosive to low energy output…

Test types – thermal ramping or isothermal with simple software for Operations

Typical test with several samples – OFTEN WILL INCLUDE A REFERENCE – or maybe two references – one an inert solvent, the other a thermocouple in air. Typically maybe 2-4 samples may be run in one test together with the reference.
Data Analysis – simple, intuitive, easy to use (again Labview-based)

Raw data will look as below

The lower two graphs show temperature and pressure – four samples. A peroxide (DTBP) in toluene, 5%, 10% and 15% - plus pure toluene (the reference). Ramped at 4°C/min, note the 'calorimeter air temperature' running parallel to the sample temperatures but a few degrees higher at any time.

This is the raw data – the analysis package allows subtraction of the toluene (or any reference) to be subtracted from the peroxide sample temperature. This allows $\Delta T$ and $\Delta p$ results

This leads in to a possible thermokinetic data analysis.
Finally the software automatically produces a full report

This four page synopsis – information from various THT RSD documents aims to show somewhat more on how the RSD works, what samples can be used, how tests may be carried out and how, what data is obtained and how it is analysed and reported.

Please request full details if more would be of interest – eg the Manuals or PowerPoint presentations,

Add on to this the ‘Options’

Cryogenic (operation from -100°C)
Mixing
Dosing
Gas collection

The THT aim is to have a versatile, flexible low cost safety calorimeter with the widest application.

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