Rapid Screening Device - RSD[™] Technical Application Note 10 20% DTBP — a Standard Sample



Introduction

As its name implies the RSD, has been designed to be able to rapidly get data on exothermic materials. This is just one application, but a central one.

The RSD can operate isothermally or at heating rates to 10°C/min—and so for screening tests a rate near 5°C/min may be appropriate. It is designed to look at materials that may be of low or high energy release and for simple screening of unknown materials metal holders such as tubebombs or ARC-bombs are perhaps best used. The RSD can be operated with up to 6 samples simultaneously—or 5 and a reference and so screening could be done with a material with various impurities etc.

20% DTBP is a sample used extensively as a standard with the Accelerating Rate Calorimeter and other safety calorimeters—and so this sample run in the RSD shows not only the use of the RSD with a 'normal' sample but gives data that can be compared to other instruments.

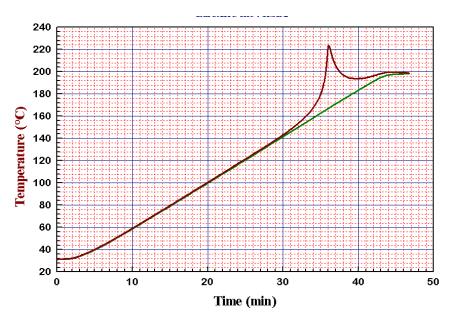
Experimental

The tests have been carried out with 6 gm of 20% DTBP in toluene contained in titanium ARC-bombs. The heating rate was set at 4° C/min and the system ramped till 200C

Tests have been carried out with two identical samples and with a third ARC-bomb containing toluene as a reference.

Results

Sample data is shown here. The ramp is linear until the exotherm commences and with the increasing temperature ramp, the reaction proceeds and causes large temperature excursion within the sample.



Typical result from 20% DTBP



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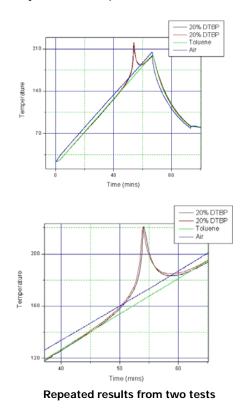


Discussion and Conclusions

The graph shown overleaf simply shows the thermal data to be expected from this standard sample. It is plotted alongside a reference.

20% DTBP is reasonably exothermic sample and with this heating rate a large temperature excursion is observed with the sample. Onset can be detected in the temperature range 130-140°C.

A feature of the RSD is that up to 6 samples may be tested simultaneously. In the graph below results from two repeated experiments are given, to indicate the reproducibility of the technique.

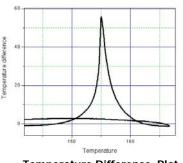


The onset of reaction when the RSD is run at 4°C/min is shown in the graph below. It should be noted that the onset is seen near 135° C. This is significantly higher than the onset seen by adiabatic calorimetry, but temperatures of onset 10-20°C lower are recorded when the heating rate is reduced to 1°C/min or below (TAN 13).

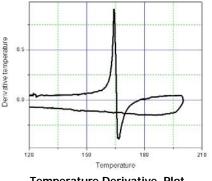
Also shown here is the calorimeter or air temperature.

A further measurement is that of pressure. Pressure can be a more sensitive detector of a reaction. This is shown in TAN 13

A standard method of graphical presentation with the RSD is temperature difference or Delta T (sample minus reference). This of course is only possible when a reference sample has been run. Such a plot is shown below



Temperature Difference Plot



Temperature Derivative Plot

If a reference is not included in the test it is possible to plot the derivative of the sample temperature. This is shown above. Both plots give better visualisation of the sample temperature and onset than the time plots.

If a sample is run in solution the difference plot appears preferable. If there is no reference the difference plot may be constructed against the plot of air temperature.

Aternatively for a sample without a reference the derivative plot may be used.

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