Rapid Screening Device - RSD<sup>™</sup> Technical Application Note 12 1% - 3% DTBP — a Standard Sample



### Introduction

Data from 5% - 20% DTBP in toluene is presented in TAN 10 and TAN 11.

DTBP in toluene is a good example to indicate the sensitivity and performance of the RSD. The Heat of Reaction of DTBP is approximately 175 kJ/mole or 1200J/g. Therefore 6 g of a 1% solution would decompose to give approximately 72 Joules and this figure would be doubled for a 2% solution etc.

In comparison with an Accelerating Rate Calorimeter the RSD operates rather differently. The sample temperature is measured within the sample and the RSD ramps in temperature—a feature that would cause the heat to be released in a more condensed time period. The RSD however does not simulate an adiabatic worst case scenario

### Experimental

The tests detailed here have been carried out in one experiment. Three titanium ARC-bombs contained 6 gm of 1%, 2% and 3% DTBP in toluene and a fourth similar sample container had 6g of toluene.

## Results

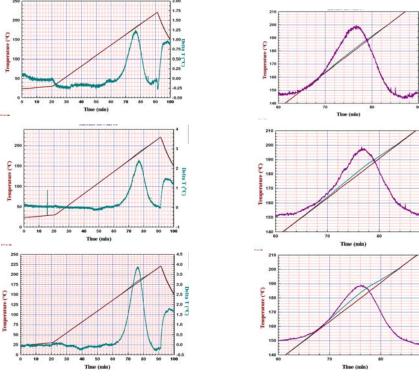
The raw data is temperature and pressure and temperature data is shown here. The sample data is compared with the reference data and automatically subtracted to give differential temperature data. The results are shown graphically and clearly with all three samples the heat of decomposition is shown. Whilst this is not clear from the raw temperature data, the differential data makes the results very clear.

# **Discussion and Conclusions**

Heat release from DTBP solutions of less than 5% are not easily seen by other safety calorimeters, but are readily seen in the RSD. The reasons for this are the internal temperature measurement and the temperature ramp

The data shown here are also an excellent indication of the performance of the RSD. The stability of the ramp is important, and this is shown. The speed of transition form idle to ramp is also shown.

Further tests have shown that DTBP solutions of 0.5% give



Results from 1% (top) 2% (middle) and 3% (bottom)

thermal hazard technology



measurable heat outputs. The results imply that under these experimental conditions-conditions that are perhaps 'standard for screening' the RSD will record heat outputs down to the 20 Joule level.

It is also possible to see the decomposition by the pressure rise-though data is not presented here.

Comparison of the graphs may be done as shown opposite.

#### The quantitative nature of the RSD is well illustrated here.

In TANs that show results from higher percentages of DTBP (eq 20%) it is clear that the sample temperature is raised by a large amount-10's of degrees. When this happens the ramp temperature and environment temperature of course will significantly lag the sample temperature. Heat will be lost from the sample. Under such circumstances quantitative data is unlikely to be available.

However here the temperature rise is small and a minimum of heat is lost. Therefore the data is likely to be quantitative.

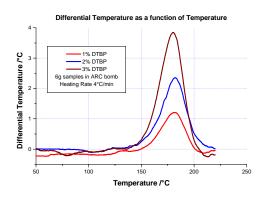
Measuring the peak areas has shown a linear relationship between the recorded heat output and the sample DTBP content.

As such and probably over a DTBP range from below 0.5% to above 5%, the heat output peak could be used to measure the DTBP content. Alternatively the peak size from similar systems could then be converted into heat output, ie Joules.

The differential plot illustrated on the same graph as the two temperature plots shows just how useful the differential data is. The onset is visually seen 20-30°C earlier when presented as differential data!

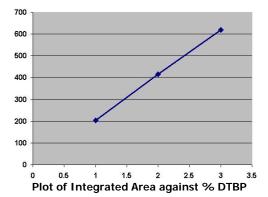
A particular application of this would be in the field of solvent recovery and distillation. Often solvents are used in industry and they are contaminated with a small percentage of reactive chemical. After recycling they are returned and re-used. The RSD offers a simple and

inexpensive determine the hazards way to from contaminated solvents, to determine the percentage of reactive chemical in the solvent before and after distillationand can of course determine the hazard associated with the still bottom residue a product that would increase in volume and hazard with time!



Comparative Plot of the Three Samples

% DTBP	Raw Integrated Area
1	204
2	416
3	620



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