Rapid Screening Device - RSD[™] Technical Application Note 11 5% - 20% DTBP — a Standard Sample



Introduction

Data from 20% DTBP in toluene is presented in TAN 10.

DTBP in toluene is a good example to generally investigate the overall performance of the RSD. The Heat of Reaction of DTBP is approximately 175 kJ/mole or 1200J/g. Therefore 6 g of a 5%, 10%, 145% and 20% solution would decompose to give approximately 350 Joules, 700 Joules, 1050 Joules 1400 Joules respectively.

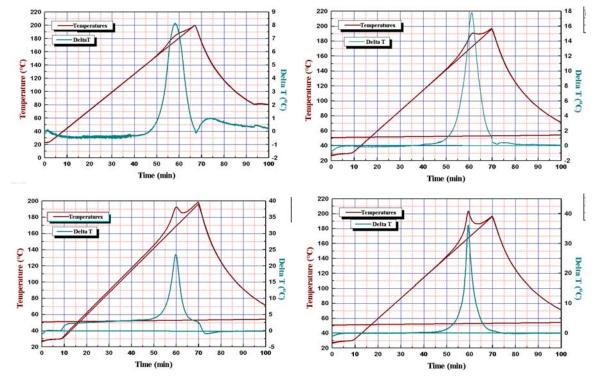
The ability of the RSD to test several samples simultaneously makes these good samples to test in a demonstration situation. This we have done on several occasions and at the same time run a fifth sample, pure toluene as a reference

Experimental

The tests detailed here have been carried out in one experiment. Four titanium ARC-bombs contained 6 gm of 5%, 10%, 15% and 20% DTBP in toluene and a fifth similar sample container had 6g of toluene. The samples were heated at 4° C/min.

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. The differential temperature is plotted to give good visual indication of the result and ease interpretation.



Raw Data results from the RSD, 5% upper left 10% upper right, 15% lower left 20% lower





Discussion and Conclusions

The four data plots shown overleaf give a good indication of temperature data recorded from the RSD and its performance with a reactive sample.

The temperature rise in the sample is clearly seen in all four samples. However plotting this temperature on the same graph as the reference sample shows the temperature excursion very clearly. From this it can be seen that the exotherm would be observed in this way for materials with even lower heat output.

But the plot showing temperature difference is more revealing. Clearly this plot shows the exotherm most distinctly—and is the best plot to display the onset of exothermic reaction. Looking just at the temperature plot may suggest an onset near 140° C, but from the differential plot, the onset is seen 15-20°C lower, near 120° C.

It should be noted that there are two ways of looking at such a result. What is shown here is differential with respect to a reference. It would also be possible to plot the derivative curve, I.e. the dT/dt of the sample.

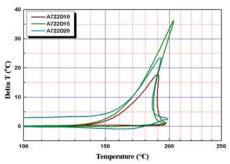
The temperature rise of 8°C, 18°C, 23°C, 36°C approximately is in uniform variation with the percentage of DTBP and the heat output. This is not to suggest that there is quantitative operation of the RSD with samples of this heat output—but as a 'rule of thumb' the relationship between temperature rise and heat output can be useful.

The plot of temperature rise in sample compared to temperature of reference is shown opposite. This clearly shows heat loss the more exothermic samples as even with the temperature gradient there is a temperature reduction after the peak of heat output.

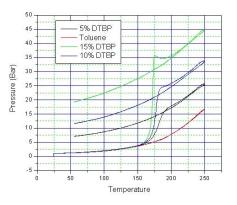
Pressure data from such tests is shown opposite. The red curve is the data from the reference sample and it is clear that all deviate from this at a similar temperature with the pressure rise approximately linear with DTBP content.

Further tests carried out under carefully controlled conditions have shown that the pressure data can be used quantitatively to assess percentage of constituent in a mixture.

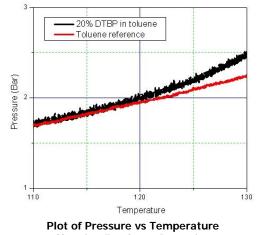
The test illustrated here is at rather rapid ramp rate and thus is not ideal to gain and interpret quality pressure data. Better data would be obtained from slower ramping—or during an isothermal age. TAN 13 discusses pressure data in some more detail. Ramp rates of 1°C/min will give for example much more accurate onset temperatures, as shown in the 3rd figure.



Plot of Temperature Rise in Sample as a function of Reference Temperature



Pressure vs Temperature for Different Concentrations of DTBP



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