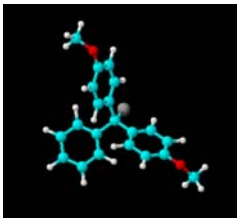
Rapid Screening Device - RSD™
Technical Application Note 103
Sample Data: 4-4 Dimethoxytrityle Chloride

Introduction

Dimethoxytrityl chloride is a light pink solid. Originally this reagent was developed as a selective protective group for 5'-OH nucleosides and nucleotides during hydrolysis. It has a melting point at 120°C. The Structure is shown below, but this does not indicate likelihood for significant energy release upon decomposition, but this does not mean that no pressure hazard will result upon decomposition.



The RSD is a rapid and reliable method to screen any chemical to see thermal and pressure effects and thus will quickly screen a new reagent for all decomposition hazards

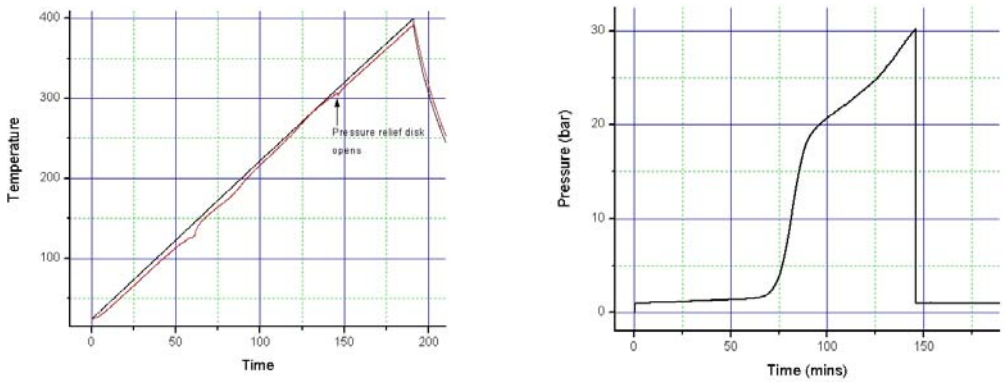
Experimental

A sample of 2.45 grams was placed in a metal pressure tight sample container and no reference was used. The local air temperature outside the sample/container was used as the control temperature and this with ambient pressure was used as 'reference' in data analysis. A heating rate of 2°C/min was used. This is somewhat lower than used for screening but is a heating rate used in other apparatus, notably the Carius Tube. The ramp was set to 400°C with a safety pressure limit of 30 bar (controlled with a burst disc).

Results

The test proceeded according to the program but near 300°C the pressure exceeded 30 bar and the burst disk ruptured. The test however proceeded to 400°C.

The results showed two endotherms, the first a major endotherm clearly due to melting of the sample at 120°C. There was a second endotherm recorded in the temperature range 170-210°C. There was significant pressure associated with this exotherm. Between 210°C and 270°C no thermal reaction is observed but there is still some pressure rise (this may be pressure rise from the non-condensable gas—or vapour pressure rise from the melted sample / residue. Above 270°C a small gradual exothermic reaction starts. This has an associated pressure rise. The pressure rise soon causes the burst disc to rupture (300°C) however the temperature ramp continues with no sign of a major exothermic event.



Temperature and Pressure data plotted against Time

thermal hazard technology

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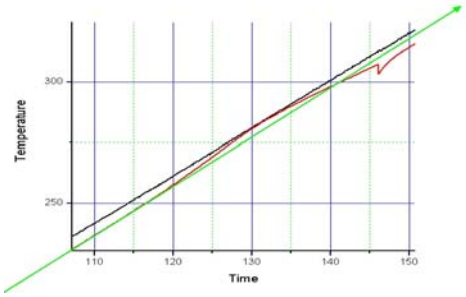
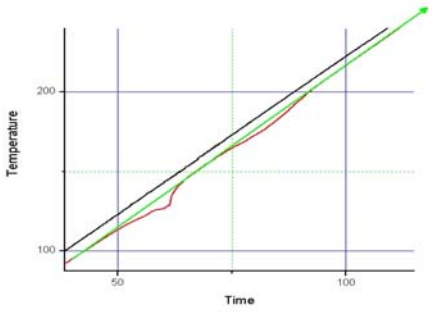
Discussion and Conclusions

The graphs shown overleaf illustrate raw data from the RSD the pressure rise is clear, the temperature events are less clearly visible. Below the two graphs are zoom plots of the temperature against time data.



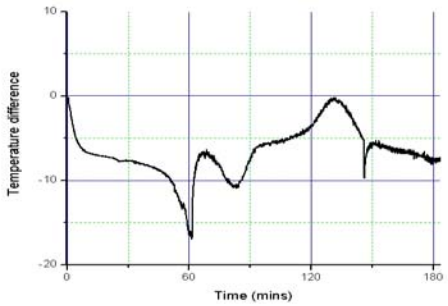


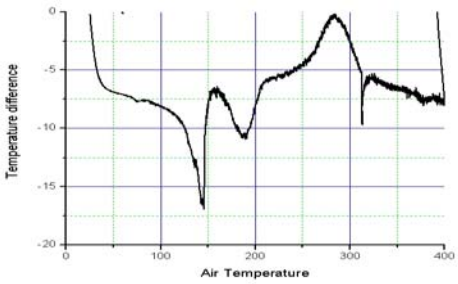
The upper plot shows the small exotherm, that is disrupted when the burst disc is ruptured and the lower plot shows the melting at 120 and the second minor endotherm.

There was no reference with this test—but the two plots below shows temperature difference between sample and the air control temperature that may be used as a reference.

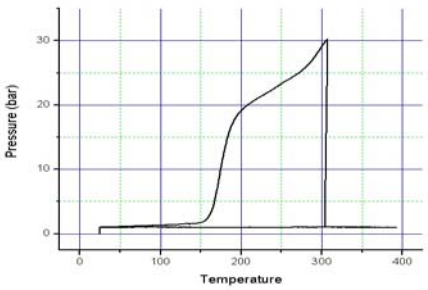
Without a reference and with no contribution from any solvent or excipient material the appropriate method to plot pressure is against temperature. In addition the pressure rate may give useful information, the next two plots show this information.

Because no reference was used the 'baseline' temperature difference is not zero but close to –6°C. The graphs now clearly show the two endotherms and the start of the exotherm, the graphs being plotted against time and 'reference' temperature.





The pressure data may be plotted against temperature (of the air reference), this is shown below.



Finally it is possible to plot temperature rate against time or reference temperature. This is not shown here, the peaks are shallow and difference rather than derivative temperature is the appropriate form of data presentation. (Pressure rate data is also an option not shown here)

In conclusion, the results from this sample indicate pressure hazard but little thermal hazard, they show the ability of the RSD to detect small endotherms and exotherms.