Rapid Screening Device - RSD™
Technical Application Note 18
Glass & Reusable Containers and Linings

Introduction

The ability of the RSD to work with a range of sample containers was central to the design philosophy of the RSD.

Glass is often the material of choice as the possibility of catalysis of a reaction is very low. Metals such as Stainless Steel are most likely to catalyse a reaction and in any calorimeter the surface area to sample volume is much greater than in a real situation.

ARC-bombs made from Titanium or Hastelloy-C may be appropriate (and will of course withstand very high pressure) but especially where many tests are envisaged the cost of such sample holders becomes significant.

A range of glass vessels may be used in the RSD. Indeed the design of the sample cavity, with the sample holder, is such that vessels of varying size and shape may be included. It is possible to use simple open test tube and these may be appropriate if there is no gas generated and lost from the sample or if simulation tests are appropriate in such an open configuration.

However for general work with glass sample holders the RSD was designed to incorporated HPLC vials. These can be attached to the connector in the normal way to give a pressure seal and can be run typically to 10 bar (150 psi). Note that in the RSD software the pressure units may be defined (bar or psi).

The sample holder connector contains the integrated thermocouple and a burst disc holder. The burst disc itself is a simple aluminium disc—of varying thickness and hence burst pressure. The glass vials have a burst pressure near 20 bar 300 psi—and thus a burst disc with that relieves pressure near 15 bar 225 psi would typically be chosen.

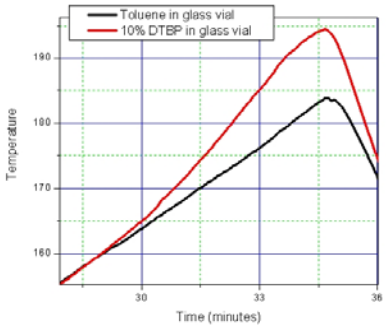
Nevertheless bursting of the glass vials might occur. If this does happen then the test is concluded and perhaps 1 bursting vial will rupture one or more others. But the glass shatters to small pieces and is easily removed from the sample chamber.

In addition there are thermocouple liners available and reusable cells where various sizes of glass sample holder can be used.

Experimental

This experiment was performed using two glass vials, one containing 1ml of DTBP in toluene solution at a concentration of 10%, the other 1 ml of toluene to be used as a reference. The thermocouples had glass lining. A ramp rate of 5ºC/ minute was chosen with a pressure stop at 12 bar.

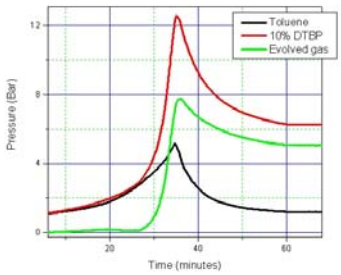
Results



The onset was seen, the reaction proceeded and the test stopped by the software when the maximum pressure set is reached at 12 bar. By this time the sample had reached a temperature 10°C higher than the reference. The glass thermocouple lining will have an effect on heat transfer and thus it would be usual to choose slower heat rate for such tests.

Because both the sample and reference have simultaneous independent pressure measurement, a differential pressure measurement is possible as shown above.

When the pressure in the sample reached 12 bar, the system switched on the cooling air to stop the test.



An examination of the pressure indicates an onset of reaction at about 150ºC.

thermal hazard technology

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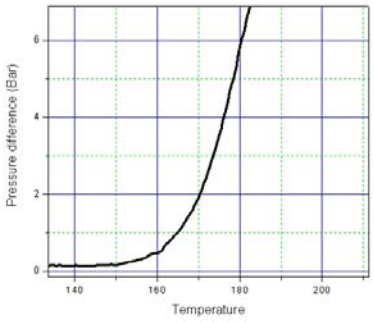


Discussion and Conclusions

The ability to use standard HPLC glass vials to perform multiple simultaneous screening tests in the RSD provides considerable benefits. The low cost of these sample containers means that the running costs of the instrument are minimised and multiple samples can be run for a fraction of the cost of a single bespoke higher pressure test cell.

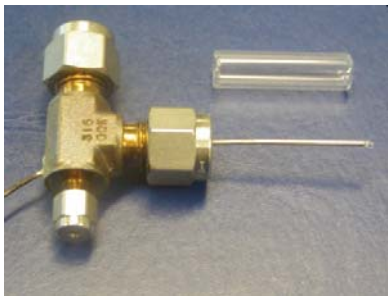
The glass thermocouple lining can also protect the thermocouple if for example polymerisations reactions were being investigated and a solid residue obtained.

The use of standard HPLC vials also brings other benefits. Internal low volume glass inserts can be placed inside the vial and used to allow testing of reactive agents in much smaller quantities. Typically volumes of 250 µl can be tested.

The all-glass system has been extended—for sample holders that are metal and reusable. This is shown in the photographs below. Here the glass vial is replaced by a screw top metal outer holder—with a variable size glass insert. In the RSD as temperature is measured 'internally' there is not the phi issue and thus this versatility is possible. Such reusable cells with throw-away metal or glass inserts (and with if necessary the thermocouple lining) can be the standard basis for use of the RSD if many tests are to be performed daily.

HPLC Vials in use

Thermocouple liner and insert



Reusable metal cell with glass insert and thermocouple liner

Reusable cell with range of inserts