Rapid Screening Device - RSD™
Technical Application Note 16
Cryogenic Operation of the RSD — Liquid-Solid Phase Transitions in Solvents; the Natural Warming and Forced Cooling Rates

The Cryogenic option for the RSD was implemented to describe the cooling and warming rates under normal use. During this test 5 solvents were used and the sixth channel used for an air reference.

Figure 1 illustrates the complete data graphically

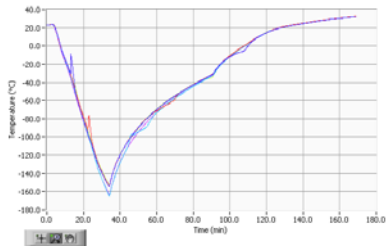

Figure 1

There was an adjustment at -40°C on the warm up but otherwise the data shows

Cooling to -100°C in 20 minutes and to -150°C in 30 minutes. Warming to -100°C at 5°C/min, then to -80°C at 3°C/min, to -60°C at 2°C/min and to -40°C at 1°C/ min. Further warming is at a rate below 1°C/min.

The melting of solvents is shown on the warm up and some solvents freezing at super-cooled temperatures is shown in the cool down. 1 ml of solvent was used in a glass vial, pressure was not recorded. The results are shown graphically, all graphs are screen copies from RSD-RAP the RSD data analysis program discussed here.

Air

Cooling and warming profiles are shown below

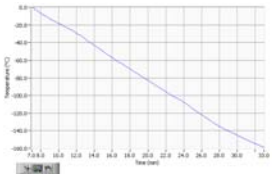

Figure 2

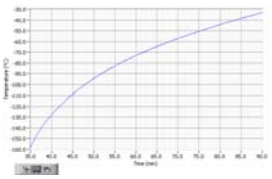

Figure 3

DMF

Cooling and warming profiles and the difference between this solvent and air are shown below

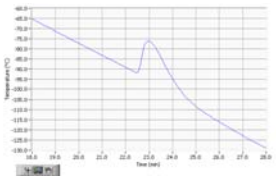

Figure 4

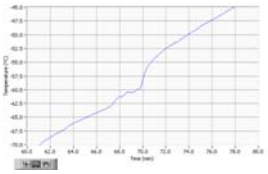

Figure 5

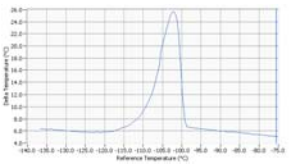

Figure 6

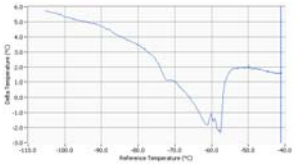

Figure 7

DMF shows super cooling and upon warming a multistage melting. No discussion here will be given as to the effects seen. It should be noted that these are simple experiments and not designed to determine freezing or melting effect. The test will show what to expect with the RSD if similar cooling and warming tests are to be carried out.

Aniline

Cooling and warming profiles and the difference between this solvent and air are shown below.

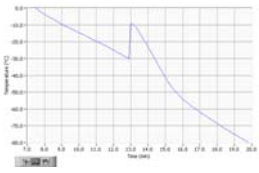

Figure 8

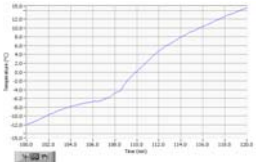

Figure 9

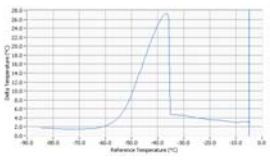

Figure 10

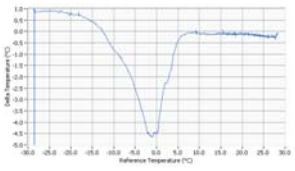

Figure 11

thermal hazard technology

Rapid Screening Device - RSD™
Technical Application Note 16
Cryogenic Operation of the RSD — Liquid-Solid Phase Transitions in Solvents; the Natural Warming and Forced Cooling Rates

Aniline shows super cooling and a rather simple melting profile.

Isopropanol

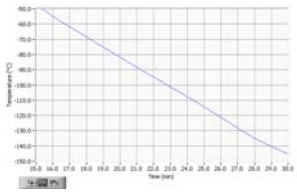

Figure 12

Figure 13

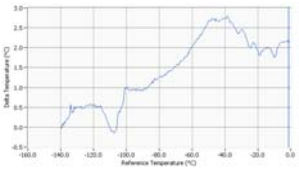

Figure 14

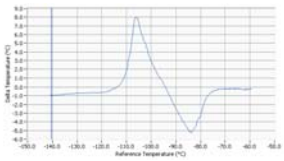

Figure 15

There was little observed upon cooling, the difference data indicated freezing over a wide range. A small endotherm was observed upon further cooling. Upon warming a clear exotherm was observed and this led into melting of the sample.

Acetic Anhydride

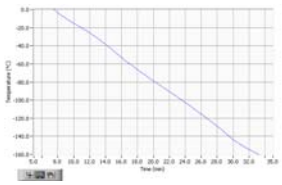

Figure 16

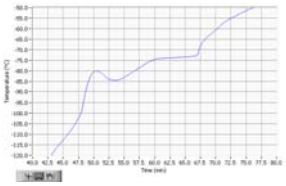

Figure 17

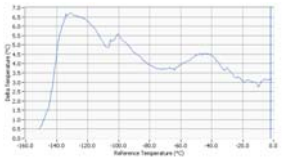

Figure 18

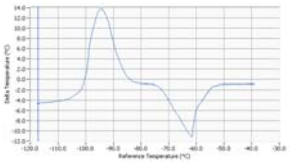

Figure 19

Again no clear freezing peak was observed. Again on warming an exotherm was observed prior to melting.

Thiophene

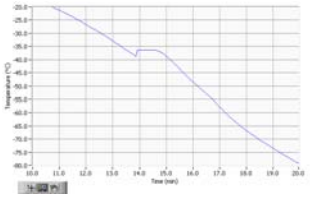

Figure 20

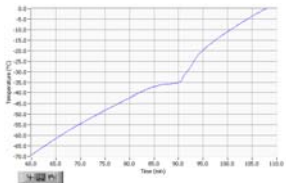

Figure 21

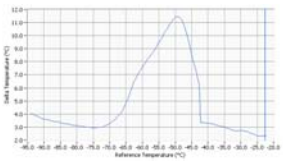

Figure 22

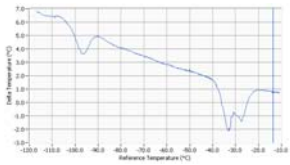

Figure 23

A clear freezing was observed with thiophene – with evidence of super cooling. There was an endotherm upon warming in the solid state and melting showed a double peak.