THERMAL APPLICATIONS NOTE

Purge Gas Recommendations for use in Modulated DSC®

Introduction

A flowing inert purge gas should always be used when operating a DSC instrument, either in MDSC or standard DSC mode. The purge gas contributes to the experiment in several ways:

- The flowing gas helps to remove moisture or oxygen which may accumulate and damage the cell over time.
- The purge gas provides for a smooth thermal "blanket", which eliminates localized hot-spots which can lead to artifactual heat flow.
- The purge gas provides for more efficient heat transfer between the constantan disc and the sample pan, resulting in more sensitivity and faster response time.
- The purge gas helps to cool the cell, so that faster cooling rates and wider modulation parameters may be achieved.

Therefore, in order to optimize the MDSC experiment, and generate accurate and reproducible results, a flowing inert purge gas should always be employed.

Types of Purge Gas

Historically, nitrogen has been used as the gas of choice in thermal analysis experiments. Nitrogen is inert, inexpensive and readily available. In the DSC cell, it provides for excellent sensitivity, as its low thermal conductivity does not interfere with the heat measurement.

Helium is recommended as a useful purge gas for MDSC experiments. The thermal conductivity of helium is about seven times that of nitrogen. Because of this, helium provides for very fast response times and excellent cooling capabilities within the DSC cell. When performing an MDSC experiment, this added cooling capability permits the use of larger modulation amplitudes and faster modulation periods. However, the use of helium has several drawbacks. Since helium is highly thermally conductive, it tends to sweep heat away from the sample reducing sensitivity. In addition, the thermal conductivity of helium remits the same effect with He upper temperature limit to ca. 400°C.

In some rare cases, argon has been used as a purge gas for MDSC. As argon is more dense than air, the flow of the argon through the cell is diminished. The purge rate which is indicated on the flowmeter is not necessarily the rate through the purge gas opening in the DSC cell. For these reasons, the use of argon as a purge gas should be carefully considered.

Purge Gas Flow Rates

The purge gas flow rate used in a MDSC experiment should be carefully set and regulated. Inconsistencies in flow rates during an experiment can cause inaccuracies in measured heat flow, especially when using helium. Figure 2 illustrates this effect. This figure shows the calibrated cell constant (indium, $\Delta H = 28.4$ J/g) as a function of flow rate using both nitrogen and helium. Note that with a nitrogen purge, the calibrated cell constant is virtually independent of purge gas flow rate, in between 10 ml/min. and 100 ml/min. However, when helium is used, there is a considerable dependence of the cell constant on the purge gas flow rate, especially at low rates. Thus when using helium, an accurate and precise flowmeter should be used, so that the flow rate does not fluctuate during the experiment, or in between experimental runs. Further, it is good practice to leave the purge gas flowing even when the instrument is not in use, to minimize any potential discrepancies in flow rate from run to run.

The recommended flow rate when using nitrogen as the purge gas is 50 ml/min. This purge gas flow rate provides for efficiency in volatile evacuation, as well as good heat transfer properties. When using helium, the recommended flow rate is 25 ml/min. This flow rate is chosen to minimize the heat dissipation effects, while still providing for volatile evacuation

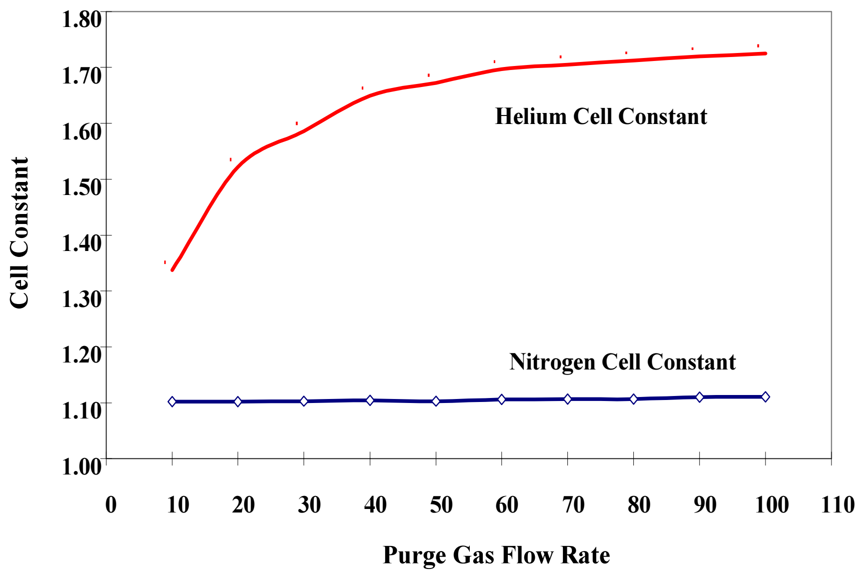

Figure 1. Cell Constant as a Function of Purge Gas Flow Rate

and heat transfer. As shown in Figure 1, minor deviations from a helium flow rate of 25 ml/min., may cause inaccuracies in measured heat flow. For this reason, it is again recommended that a reliable flowmeter be used, to insure consistent metering of purge gas flow.

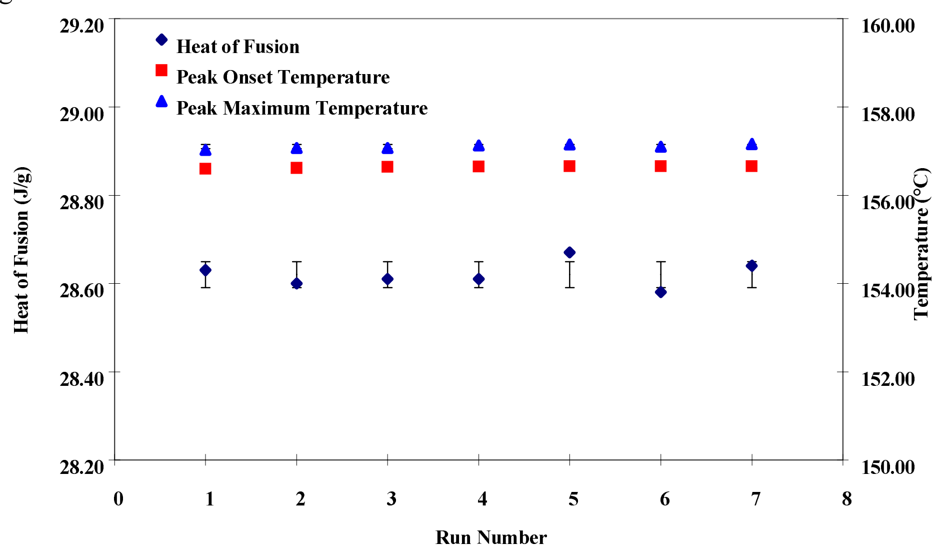

Figure 2. Reproducibility of the Indium Melt

Figure 2 shows the reproducibility of the analysis of an indium melt, when the helium purge gas is properly maintained at 25 ml/min, using the Rotameter flowmeter available from TA Instruments (part # 270134-001). The error bars in this plot represent ±1 standard deviation over seven experimental runs. Note that the measurement of peak onset temperature and peak maximum temperature. The measured heat of fusion is also very consistent.

When performing DSC and MDSC experiments, in addition to flowing a purge gas through the DSC cell (port marked "Purge"), it is also recommended that purge gas be introduced into the "Vacuum" port of the instrument at 50 ml/min. This gas will flow around the outside of the furnace, and prevent moisture condensation at low temperatures. The Refrigerated Cooling System (RCS) provides this additional purge as part of its normal operation, so when an RCS is used, the vacuum port should be capped or blocked with a rubber stopper.

Proper Installation of a Flowmeter

One of the common mistakes made when installing a precision flowmeter is supplying too low of a delivery pressure to the flowmeter. If the delivery pressure is too low, the flow rate is not adjusted by the flow meter effectively, and flow rate deviations can occur with changes in room temperature, gas tank pressure, or flowmeter position. Therefore, a minimum delivery pressure of 5 psi should be applied to the flowmeter, so that it can regulate the purge gas effectively. Generally, a delivery pressure of 5-10 psi works well (excessive delivery pressures could cause safety concerns).

Summary and Recommendations

- A flowing inert purge gas should always be used when performing a DSC or MDSC experiment.
- Nitrogen is the preferred purge gas, although helium may be use if faster cooling rates or wider modulation parameters are desired.
- Using a reliable flowmeter, flow rates of 50 ml/min (nitrogen) or 25 ml/min (helium) should be introduced into the "Purge" port, and 50 ml/min (nitrogen or helium) should be introduced into the "Vacuum" port.
- Make sure the flowmeter has a delivery pressure of at least 5 psi, to insure stable flow.

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