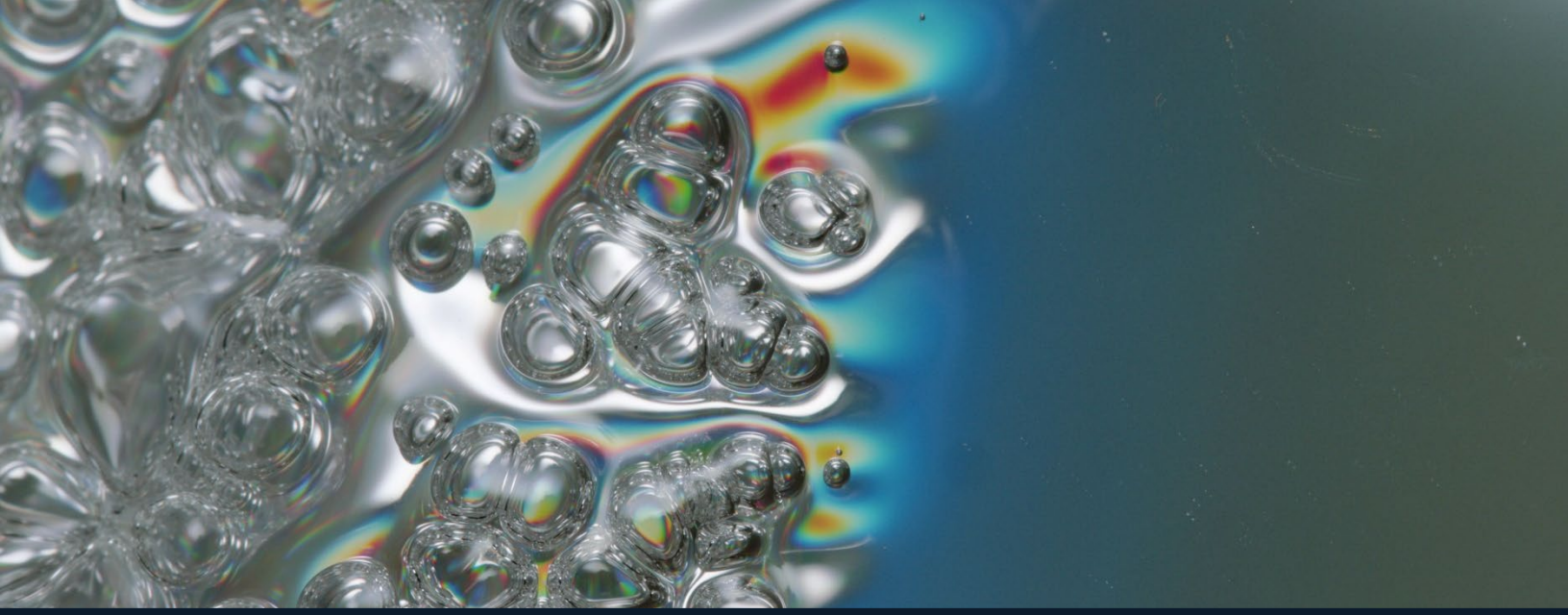

Covalent Academy Episode 22 Q&A

Optimizing Polymers and Adhesive Materials with Thermogravimetric Analysis and Differential Scanning Calorimetry

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01 Q: What is the advantage of the vertical balance design on your TGAs compared to the horizontal balance design of your STAs?

A. Both balances are great designs and are designed for certain purposes. The vertical balance design enables really flat baselines and has a slightly better performance than the horizontal balance on our SDT 650. The horizontal balance on the SDT 650 also is designed that way because it serves the purpose of also doing DSC measurements.

02 Q: How does the TGA modulate with cooling?

A. The sinusoidal temperature program used in MTGA is based on an approach first described in 1968 by Flynn (TA App Note 237). Since there is no active cooling on the TGA, it can only cool by direct radiation.

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03 Q: Can DSC/TGA used as QC tool to study lot to lot consistency organic based products?

A. Yes, it can. DSC and TGA are commonly used for this purpose. Polymers and plastics are organic based products.

04 Q: In regard to TGA, can the effect of humidity be observed during a sample's decomposition while applying air?

A. I'm not exactly sure what the question is asking with regards to air and effect of humidity but maybe the following information may help. Certain TGAs allow delivery of steam into the TGA furnace (high pressure TGA systems from TA for example) while other systems like the Q5000SA allow one to control humidity and see the mass gain/loss in the sample.

05 Q: What role does the flange temp play in a DSC measurement?

A. This is the temperature of the cooling flange of the cell. The flange temperature is related to the RCS coolers connected to the DSCs and change as the DSC is heated and cooled. The flange temperature of the RCS at a certain temperature of the DSC cell can help indicate the health of the cooler system.

06 Q: Why do I sometimes observe weight gain in the TGA instead of weight loss?

A. Yes, this involved a change in the way certain elements of the window were formulated. The new formulations did not show this effect.

07 Q: Are there ASTM standards for modulated DSC measurement of heat capacity?

A. ASTM E2716

08

Q: Can I use a TGA or DSC to measure boiling point? And what precautions are needed?

A. Typically a DSC will be used to measure the boiling point of a sample. Usually boiling point measurements would use a pan with a pinhole lid since if the pan is completely sealed, you will shift the boiling point as the pressure increases. You do have to be careful with not contaminating the DSC cell however.

09

Q: What is the repeatability I can expect from my TGA, and what affects it?

A. The repeatability of the TGA depends on a number of factors including the health of the TGA, test conditions, the ability to ensure similar sample preparation, and most importantly the homogeneity of the sample. We commonly find that analysts fail to realize that some variable that comes from the measurement is due to the sample and not just the instrument. For something like calcium oxalate that we show in our training slides, we get accuracy of weight loss steps at 1.6% from the theoretically expected weight losses, with a precision of 0.2% or lower. We also find that controlling the sample mass is an easy way to improve repeatability.

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Q: What is the repeatability I can expect from my DSC, and what affects it?

A. Similar to the TGA, repeatability depends on a number of factors including the health of the DSC, test conditions, the ability to ensure similar sample preparation, and most importantly the homogeneity of the sample. We commonly find that analysts fail to realize that some variable that comes from the measurement is due to the sample and not just the instrument. The temperature repeatability for something like Indium is +/- 0.025C whereas the enthalpy repeatability is +/- 0.25%. Again controlling the sample mass greatly aids in getting the best repeatability.

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Q: When would I use what type of pan (I see many are offered)?

A. Different pans are used for compatibility with certain materials (e.g. gold vs. aluminum vs. graphite), or for experiments needing certain temperatures (aluminum vs. gold vs. graphite) or pressures (aluminum hermetic vs. high volume stainless steel vs. gold hermetic vs. high pressure capsules). Sometimes different lids are needed for certain types of experiments – a hermetic lid may prevent a curing reaction from completion as the water released is trapped so one may use a pin-hole lid or standard lid

12 Q: I see a lot of measurements at 10C per minute ramp. When would I go slower or faster?

A. Based on the Heatflow equation, the faster you go, the higher the heat flow signal - you would do this to try and maybe see a small feature. On the other hand, going slower helps improve resolution but at a cost of heatflow signal.

13 Q: Can I use DSC or TGA to identify an unknown type of plastic?

A. Analytical tools are used to analyze different types of materials and sometimes one tool can help fully identify a material, and sometimes you need more than one tool. If the unknown plastic is not a mixture, one maybe able to use DSC to identify the Tg, melt peak temperature and other features to identify the plastic, and the TGA decomposition curve may reflect the chemistry of that plastic but one would have to use other methods to cross-validate the results.

14 Q: How do I calibrate a DSC?

A. Calibration of a DSC is explained in greater detail in TA Instrument's Theory and Applications course available via our website. In summary, the DSC is calibrated for baseline (this can involve one or more runs based on the technology in the DSC), calibrated for enthalpy (cell constant) and temperature using a metal like Indium, and maybe calibrated for heat capacity using a material like sapphire or a metal depending on the types of samples being run.

15 Q: How do I calibrate a TGA?

A. A TGA is calibrated using standard masses and for temperature where one method involves the use of magnetic materials and a magnet to observe the Curie point. Calibration of a TGA is explained in greater detail in TA Instrument's Theory and Applications course available via our website. Temperature can also be calibrated using melt points in our newer TGA systems since there is a DTA signal available.

