Q5000 IR Features to Ensure and Maintain Optimum TGA Performance

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ABSTRACT

Important facets of a top-flight analytical instrument include performance (e.g., highest accuracy, precision and sensitivity), operational convenience, and powerful, flexible software. When properly integrated, they combine to make the process of generating high quality data easier and less subject to human error.

TA Instruments engineering and design teams addressed these requirements in developing the Q5000 IR, a new thermogravimetric analyzer (TGA) that meets the most demanding research applications. They also designed for performance consistency by providing automated protocols, that that can be scheduled at off-work hours, to permit the user to maintain the analyzer in top operating condition. Some impediments to obtaining consistent, high quality data and how the Q5000 IR resolves them via the scheduled protocols are described.

INTRODUCTION

While a TGA may not be the primary analyzer for structure determination in most laboratories, it often plays an important role in pre-evaluating materials prior to analysis by DSC, rheology, or other material characterization techniques. By heating milligram quantities of sample under an easily defined protocol, the TGA can indicate and quantify the presence of volatiles (e.g., water, solvent or monomer) and the temperature over which volatilization or decomposition occurs. It also serves to quantitatively separate formulations, assess volatiles, inert fillers, plus pyrolyzable and oxidizable components. The task for the TGA operator is to obtain reliable percentages and onset temperatures using a prescribed temperature and purge gas program.

In many laboratories today, multiple operators use the TGA, since it can function in both direct analysis and in sample pre-assessment roles. It is also increasingly configured with an integrated autosampler to optimize

Figure 1. Q5000 IR TGA

laboratory sample analysis efficiency and productivity. While obtaining consistent, high-quality TGA data should not require the dedicated presence of an “expert”, a certain amount of operator knowledge is required to ensure that the analyzer is free of contamination, properly calibrated and in good operating condition. For example, to ensure that previous experiments have not left behind semi-volatile decomposition deposits, it makes sense to perform a quick, high temperature conditioning, empty pan run using an oxidative purge. If the TGA has not been used for some time, it is prudent to make a validation run with a known material and if needed perform a complete calibration. While these are all reasonable steps to ensure that the results obtained can be trusted, they are also time-consuming, and are often omitted by many less experienced operators due to the constraints of time.

In the TA Instruments’ Q5000 IR, a powerful new software feature lets the user build some of this good laboratory practice into automated protocols that permit in-advance scheduling of key instrument performance checks and maintenance procedures, such as validation, calibration, verification and diagnostic analyzer tests at convenient off-work time periods. Notification of test completion can be sent to the user e-mail or Microsoft Messenger. Some of these new user convenience features designed into the Q5000 IR, and the benefits of their use, are discussed in the following sections.

PURGE GAS CONTROL

One source of variation in TGA results arises from incomplete control of purge gas flow rate and composition in the furnace chamber during analysis. If not under computer control and monitoring, this critical aspect requires operator diligence to check various purge flows prior to analysis and ensure that they are as specified in the method. Even when this occurs, changes in source pressure or line leakage can lead to an error and result in inconsistent data that would be virtually impossible to confirm after the fact. Purge gas errors can be particularly severe when the method calls for switching between inert and oxidative atmospheres.

Purge gas related issues are virtually eliminated when the purge flows are determined by digital mass flow controllers (rather than by line pressure) and the flow rates are under computer control using settings specified by the selected TGA method. In a worst-case situation, where a gas supply runs out during an analysis, the anomaly would be detected by the software and reported to the operator. In fact, the TA Instrument software allows the operator to anticipate this possibility and elect to terminate all further automated analysis. With this type of system the actual purge conditions during the analysis can always be checked afterwards should the results appear anomalous.

CALIBRATION SEQUENCE

When using a TA Instruments’ TGA with an autosampler, the samples reside in the autosampler tray, and the analysis methods are queued in the run sequence. Specific methods can be developed, stored and recalled, as can whole sequences of methods. It has long been possible to run a series of standard experiments to condition the analyzer and perform a calibration or verification run as a precursor to taking trustworthy data.

What is new in the Q5000 IR is the ability to schedule various additional validation, calibration, verification, and diagnostic tests, either separately or in a sequence, and have notification of test completion and the results automatically e-mailed to interested parties. A typical scheduled test sequence might include diagnostic tests, temperature calibration, mass loss validation and weight calibration. Figure 2 shows a typical Curie Point temperature calibration method setup. The real value to the busy customer is not only to have these necessary, but time-consuming tests automatically performed, but also to have them scheduled to be performed during non-working time, such as overnight or at weekends. A more detailed account of this novel capability will be supplied in a later application brief.

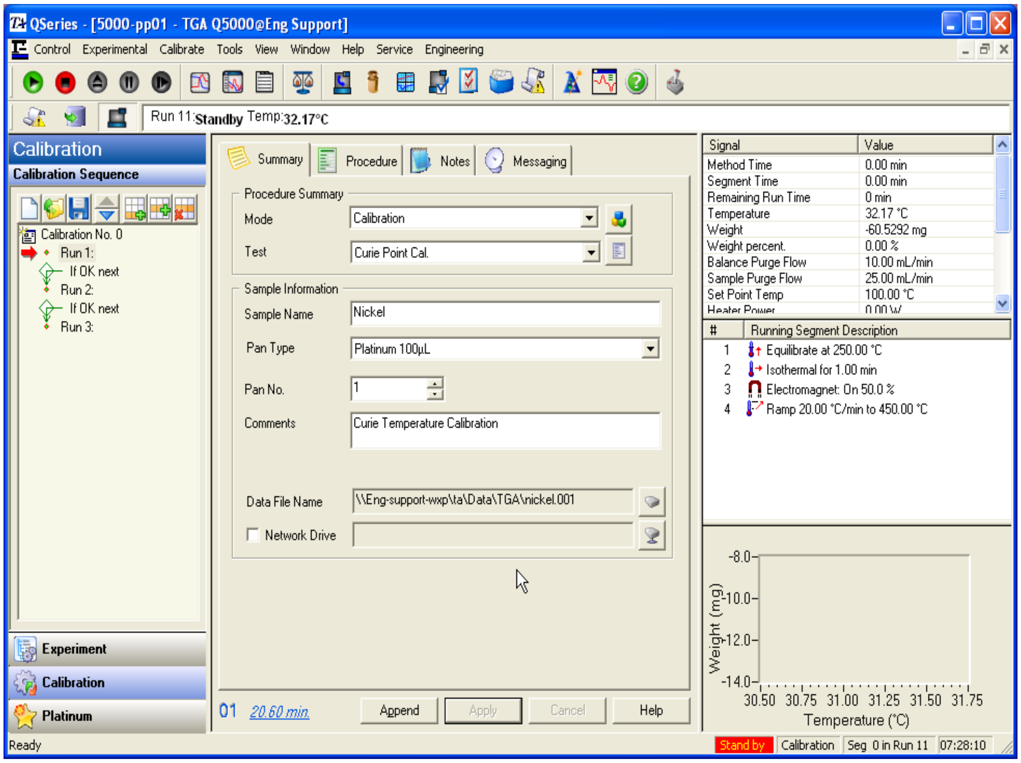

Figure 2. Input to set up a Calibration Sequence

TEMPERATURE CALIBRATION

The "gold standard" for TGA temperature calibration and verification is the use of Curie point magnetic standards. These materials (e.g., alloys of cobalt and nickel) each reversibly lose their ferromagnetic attraction at a reproducible, characteristic temperature (the Curie point), which is determined solely by the chemical (alloy) content. A working group under the auspices of the International Congress for Thermal Analysis and Calorimetry (ICTAC) selected a series of alloys having Curie points approximately every 200 ºC between 152 and 1,116 ºC (1), and the Curie points of these reference materials were determined in an interlaboratory test. These materials are now available exclusively from TA Instruments. In earlier TGA instrumentation it was necessary to manually place a magnet above or below the furnace to provide the magnetic field that results in the apparent weight change when going through the Curie transition. The Q5000 IR now provides a programmable electromagnetic field in the furnace chamber, which makes Curie point temperature measurement one of several automatic methods in a calibration sequence. The data taken in a Curie point determination (which can include several Curie transition points) can subsequently be used to update the temperature calibration.

Figure 3 shows a temperature calibration check using five magnetic transition standards in a single heating scan. Any or all of the calibration results can always be readily validated either after the calibration or at a later date by re-running the appropriate reference materials.

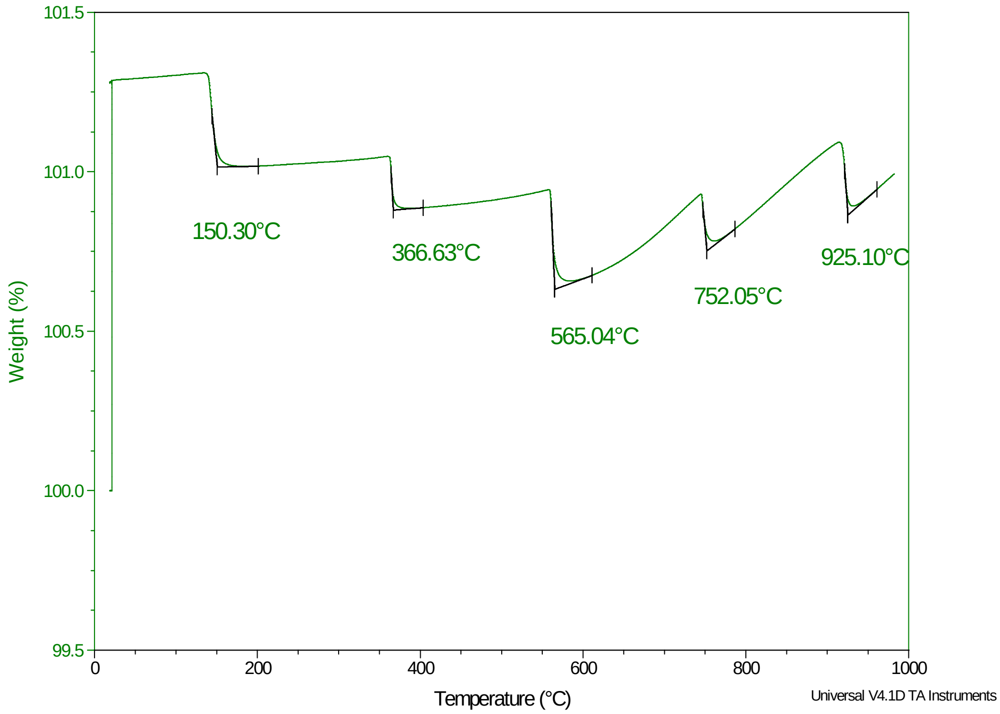

Figure 3. Multiple Point Curie Determination

AUTOWEIGHT CALIBRATION

As part of a calibration sequence the weight scale of the TGA microbalance can be calibrated. This step is accomplished in roughly 2.5 minutes using a tared empty pan and one containing a 100 mg weight. As with the above tests the operator prepares the appropriate samples in the autosampler tray, and the TGA steps are carried out automatically as part of a scheduled calibration sequence. At the operator's option, the successful completion of each test can be communicated via e-mail or Microsoft Messenger to interested parties. Figure 4 shows a screen from the routine to schedule a calibration sequence.

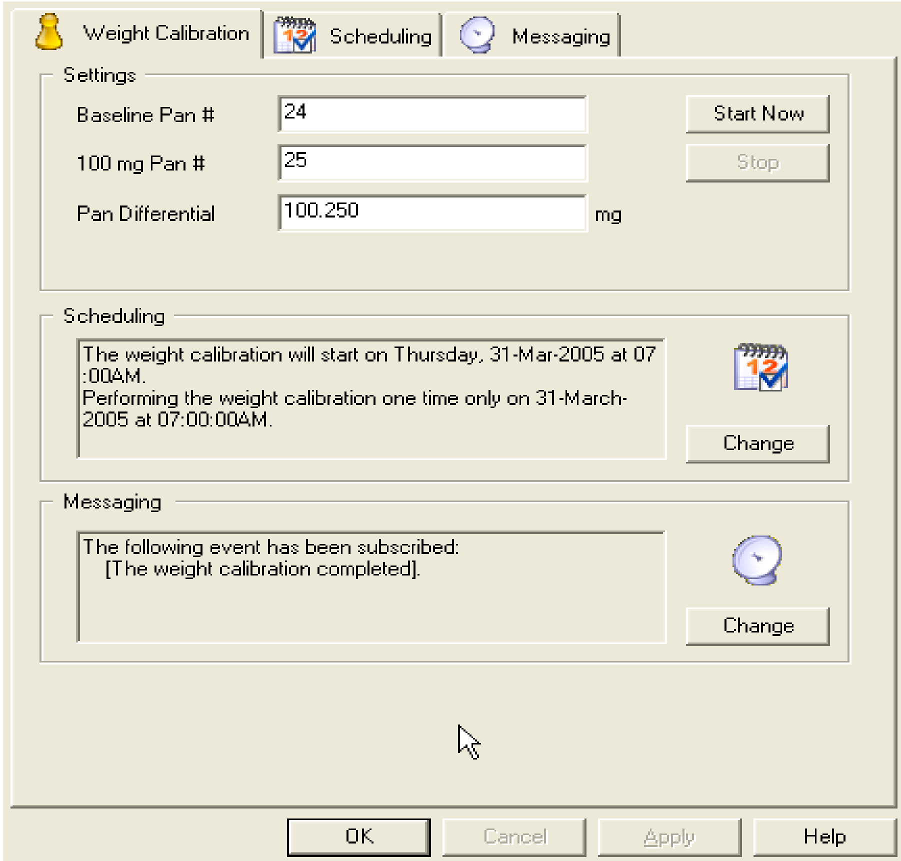

Figure 4. AutoWeight Calibration

MASS LOSS VALIDATION

Validation, as applied here, is a process designed to demonstrate that an instrument works as intended. Validation tests yield figures-of-merit data for repeatability, linearity, detection limit, quantitation limit and bias when running standard tests on a series of well-characterized materials. Such a standardized test for mass loss validation has been under development by ASTM International (2). The test materials consist of blends of two materials that boil at quite different, elevated temperatures. These pre-blended reference materials and the test method are available from TA Instruments. They can be used to validate that a TGA system is capable of generating accurate data, or they can be used on a regular basis to generate figure-of-merit data as a means of tracking instrument performance. Figure 5 shows a superimposition of three TGA runs each for a different reference mixture. The materials consist of three different concentrations of 2-ethoxy ethylacetate in a high boiling alcohol that show no decomposition profile overlap. The test protocol calls for determining the percent weight loss at 160 ºC, which lies between the boiling points of the two components of the mixture.

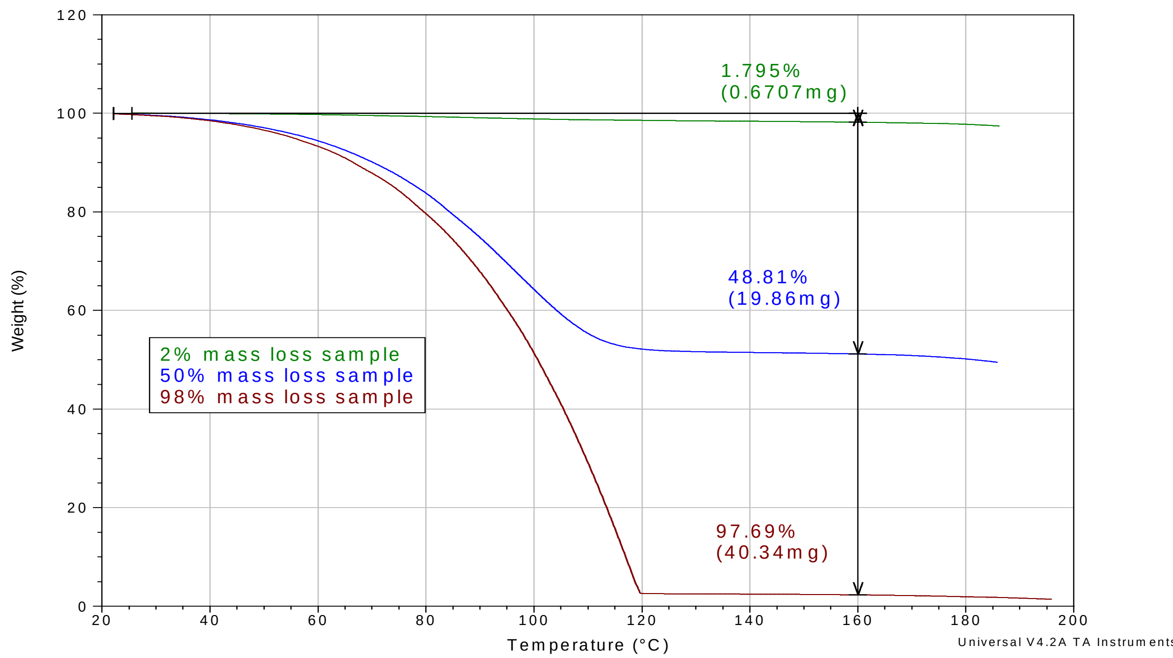

Figure 5. TGA data for validation runs on high, low and midrange mass loss reference mixtures

AUTODIAGNOSTICS

The AutoDiagnostics routine tests various analyzer electronic and sensor operations, including autosampler functionality, furnace impedance, board electronics, balance operation and mass flow controller balance. A complete diagnostic report identifying any problems can be automatically sent to interested parties by e-mail (See Figure 6).

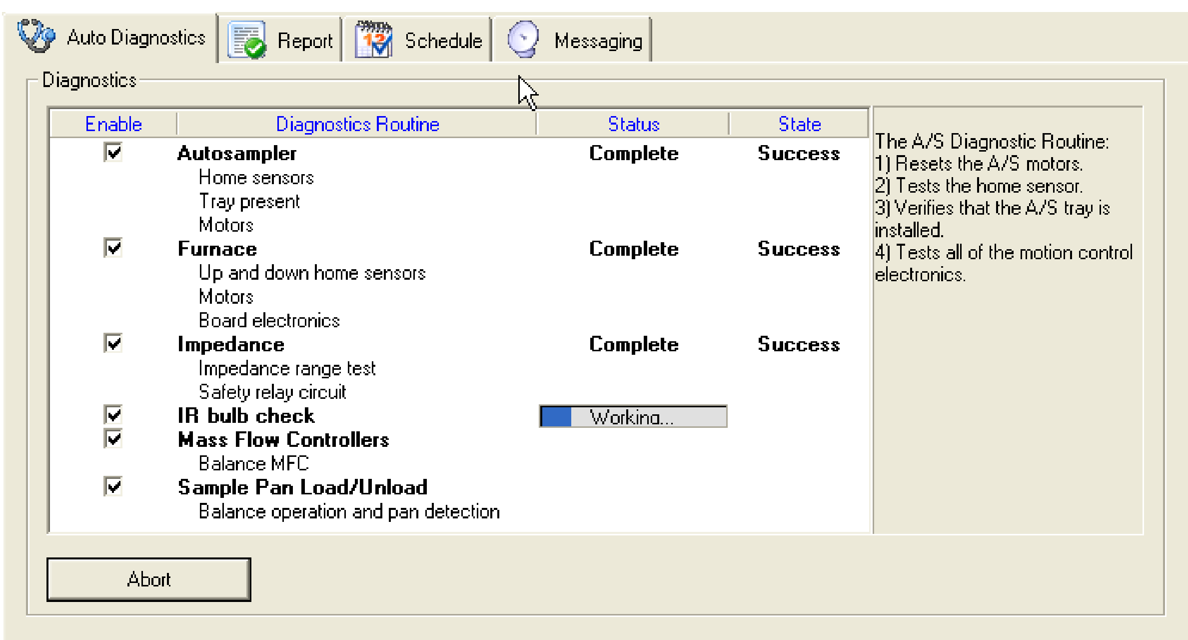

Figure 6. AutoDiagnostics Status Report

Another feature of the Q5000 IR software, checks the TA Instruments website for updates to its software, and facilitates downloading the latest version.

These advanced capabilities, that keep the Q5000 IR optimized for immediate quantitative sample analysis, are yet another example of TA Instruments' philosophy of seeking customer input in analyzer design.

CONCLUSION

Part of ensuring the success of a thermal analysis laboratory is setting up procedures for good laboratory practice, including regular instrument maintenance, calibration, verification and data tracking. The use of the Q5000 IR with its integrated 25-position autosampler and advanced Advantage software makes it convenient to carry out these procedures. Scheduling them to be performed at low, or non-usage, working times improves operator and laboratory efficiency, keeps the analyzer constantly conditioned to perform when required, and by provides a continuous electronic record of instrument operation.

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KEY WORDS

Temperature, calibration, Curie Point, TGA, Q5000 IR, Verification, Mass-Loss, Validation, Diagnostics, Purge Gas,

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