The Q5000 Automatic Sample Processor

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ABSTRACT

This paper describes the design, operation, performance and user convenience features of the Q5000 automatic sample processor.

INTRODUCTION

The capability for automatic sample processing, analysis and data reduction in Thermogravimetric Analysis (TGA) experiments is rapidly becoming a need in busy materials characterization laboratories. It improves productivity, minimizes operator error, and frees staff for other tasks. While most samples are analyzed in open pans (e.g., platinum), a trend exists for encapsulation of environmentally sensitive materials to preserve their composition in the autosampler tray until they are analyzed. Examples include materials containing volatiles, hygroscopic samples, and those that are oxidation or light sensitive.

DESIGN

The above concerns have been met in the design of the autosampler (Figure 1) integrated here into the TA Instruments' Q5000 IR, an advanced TGA that features excellent baseline flatness, outstanding sensitivity and unmatched heating rate range. These performance features have been described elsewhere (1,2).

Figure 1. Q5000 IR Thermogravimetric Analyzer

The autosampler contains three separate software controlled motors (Figure 2) designed to ensure a) smooth turntable rotational motion b) efficient horizontal translational motion between the park and load positions; c) precise vertical motion required to punch the sealed pans; and d) to load and unload the samples from the furnace hang down wire without disturbing the balance. The Q5000 IR autosampler turntable can accommodate up to 25 samples, which can be processed for analysis in a numerically sequential or random fashion. Position and operational calibration can be performed either from the system controller or from the Q5000 IR full VGA display screen. The 25-sample turntable can be replaced by a 10-sample version if operation with 180 microliter semispherical, metal-coated, quartz capsules is required for sorption studies. The 10-position autosampler is a standard feature on the Q5000 SA Sorption Analyzer.

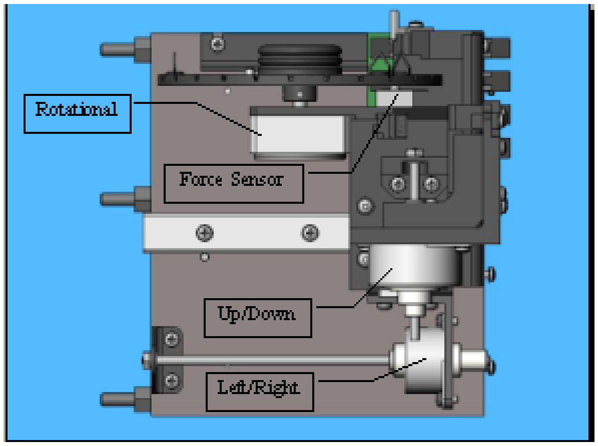


Figure 2. Autosampler Motor System

SAMPLE PANS

Samples can be analyzed in 50 or 100 µL platinum pans with Platinum bails, 100 µL platinum pans with Inconel ® 600 bails, 100 or 250 µL ceramic (alumina) pans with ceramic bails, 80 µL open or 20 µL sealed aluminum pans, and 180 µL semispherical metal coated quartz capsules as shown in Figure 3.

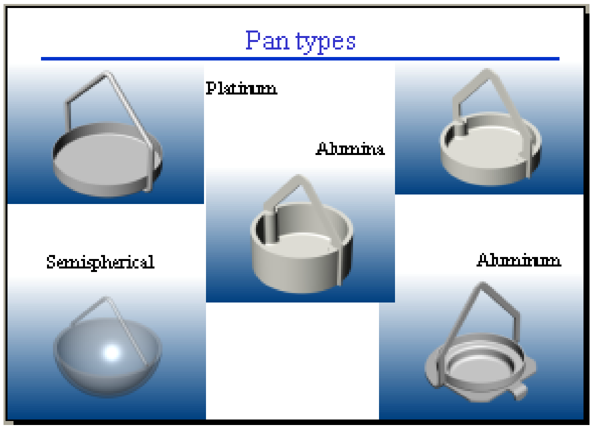


Figure 3. Q5000 Pan Types

PRESERVING SAMPLE INTEGRITY

The issue of compositional integrity of materials while awaiting analysis in the autosampler tray has been addressed in the Q5000 IR in to ways. The first is by the instrument's ability to pre-weigh samples awaiting analysis and then compare the value with the weight recorded at the actual time of analysis. This permits detection of any weight change while in the autosampler tray. The second, and more comprehensive, solution to the issue of environmentally sensitive samples is to encapsulate them in pans (e.g., aluminum), which are automatically opened just prior to analysis. With the Q5000 IR, samples are easily encapsulated in a two-stage process using the new design, aluminum pans and lids and a conventional DSC press with a dedicated lower die-set and crimping tool (Figure 4).

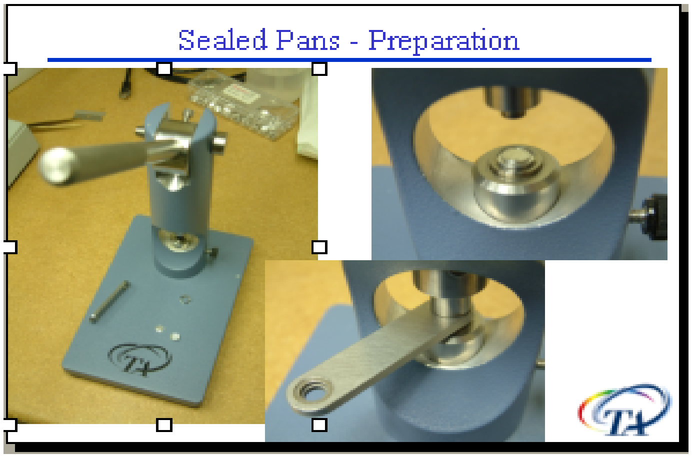
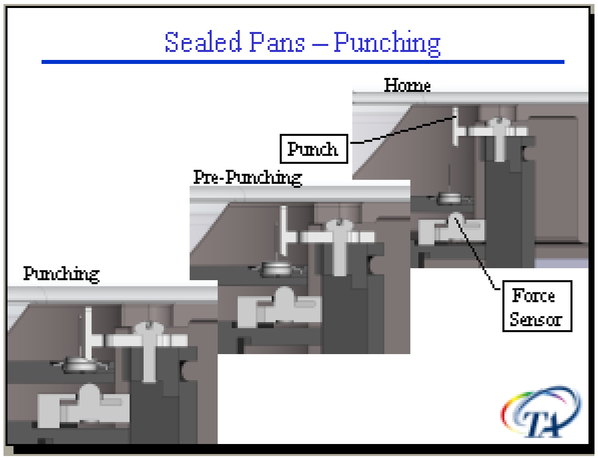


Figure 4. Sealed Pan Preparation

The use of the aluminum TGA pan permits flexibility for use either as an open or sealed pan depending on the desired use. In either version, the pan is supported by a reusable stainless steel carrier (bail). The aluminum pans are commonly used as disposable TGA pans for analysis of materials up to 600 ºC. The sealed version permits the automatic analysis of samples that traditionally needed to be analyzed manually.

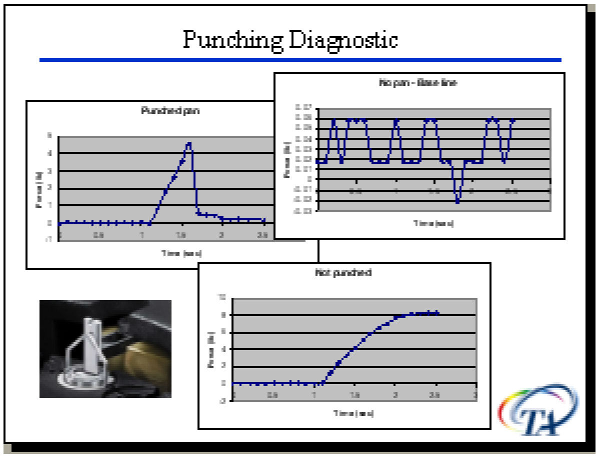
A powerful feature of the Q5000 autosampler is its ability to efficiently and precisely open sealed aluminum pans without contaminating later samples in the autosampler queue. In low-level analysis of volatiles, it is often necessary to maintain samples in a sealed environment until immediately prior to their being consigned to the furnace in order to avoid gain or loss of volatile materials. The autosampler design resolves both of these conditions with a unique force controlled sealed pan "punching" mechanism in which the stainless steel "punch" is always on the upper surface of the lid and thus cannot make contact with sample. This is a distinct improvement over other designs that use an "awl" style piercing device. The resulting opening is sufficiently large to minimize any small differences in the volume available for volatilization that may result from minor differences in lid deflection.

Figure 5 shows the pan punching mechanism. The stainless steel punch is secured and calibrated using a fixture. The sealed pans are set in selected positions in the tray, the analysis sequence programmed and the experiment is started. The turntable is smoothly rotated to the chosen location, raised quickly to the pre-punch position and then slowly until the sealed pan impinges on the fixed punch, which uniformly depresses the lid. An integral sensor monitors the force applied to the lid. With the seal broken, the turntable is lowered and moved horizontally to deliver the punched pan to the furnace loading position. Another motor then raises the pan to couple the bail to the hang down wire with essentially zero strain on the balance. The furnace is then automatically closed and the analysis started. Upon completion and cooling to a set temperature, the pan is returned to the tray and the process continues with the next sample. As a safety measure, if the punch fails to open the sealed pan, it will try again, and if still unsuccessful, the pan will not be placed in the furnace. The success of the punch mechanism can be verified electronically as shown in Figure 6. A successful pan opening is shown in the top left image, an unsuccessful one in the lower image, while the third image (on a higher sensitivity scale), shows random noise in the mechanism. On the same scale the noise would be undetected.



Sealed Pans – Punching

Figure 5. Pan Punching Mechanism



Punching Diagnostic

Figure 6. Punch Diagnostics

EXPERIMENTAL

An alternative way to view the operation of the punching mechanism is through the use of actual samples in pans designed to 1) subject the sample to different degrees of exposure to the purge gas and 2) to allow decomposition products to exit the pan under different conditions. To demonstrate this effect, Calcium Oxalate Monohydrate samples (3-8 mg) were loaded into the TGA aluminum open and sealed pans and additionally into a DSC hermetic pan with a lid containing a 75 µL laser drilled hole. This pan fits nicely into the TGA stainless steel bail. The open pan would be expected to allow most exposure to the purge gas and also to show the fastest rate of loss of the decomposition products, followed in decreasing order by the "punched " sealed pan and finally the DSC "pinhole" pan with the restricted opening. The latter pan also simulated the condition that would occur with other TGA autosamplers, where the sealed pans are pierced using a sharp "awl" like device.

Each sample was automatically loaded into the Q5000 IR furnace and heated at 20 ºC/min to 600 ºC in a nitrogen atmosphere. These conditions allow only the loss of moisture followed by the decomposition of calcium oxalate to calcium carbonate. Figure 7 shows duplicate results for each pan type. The results were in accordance with the prediction. In all cases the expected stoichiometric amount of moisture was released but at different rates. The open pan lost moisture fastest, followed by the "punched " pan and finally the DSC pan with the restricted opening. This provides a mechanism for control of exposure to the purge gas that may be utilized for other samples depending on the results desired.

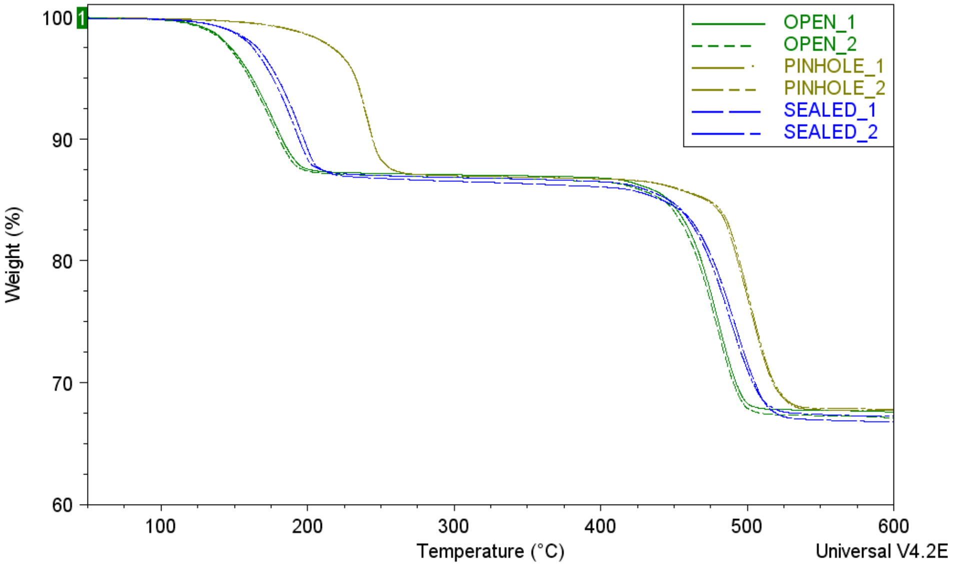


Figure 7. Calcium Oxalate Monohydrate Analyzed in Different Pans

Another experiment to confirm expectations was conducted using three equal mass samples of gypsum (Ca SO4.XH2O), a material that exists as a dihydrate / hemihydrate mixture. Figure 8 shows a previous analysis, using a Q500 TGA, of this material in an open pan and in a DSC hermetic pan with the pinhole lid described above. The data shows that in the open pan configuration, the water weight loss occurs faster but with no separation of the hydrate forms. In contrast, the significant constraint imposed by the pinhole opening provides sufficient internal pressure and time for good resolution of the dihydrate and hemihydrate forms.

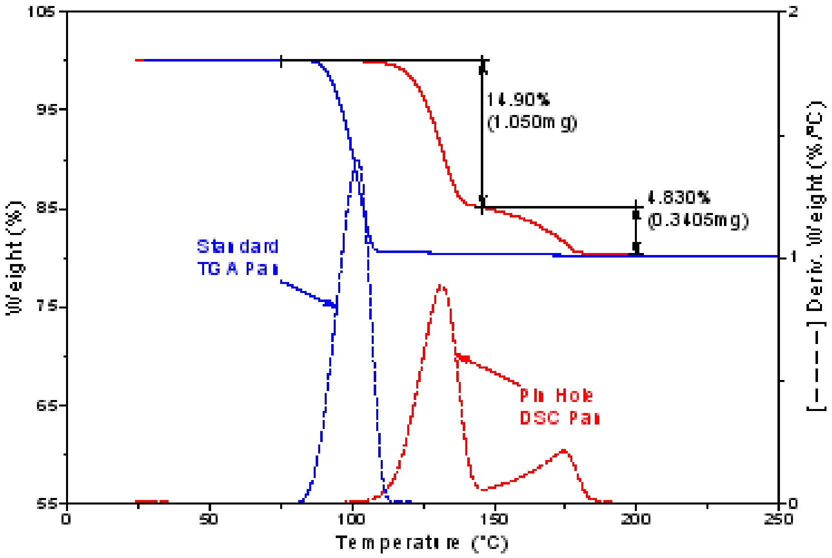


Figure 8. Gypsum in Open and Pinhole Pans

Figure 9 shows the result for a gypsum sample sealed in the aluminum pan and opened just prior to analysis. It reveals, as expected, characteristics of both plots in Figure 8. The single weight loss is indicative of the open pan (blue trace) in Figure 8. However, a time base plot showed a delay in the water loss akin to first loss in the pin-hole plot (red trace) in Figure 8. This effect may provide opportunities for separation of decomposition profiles of other closely related materials.

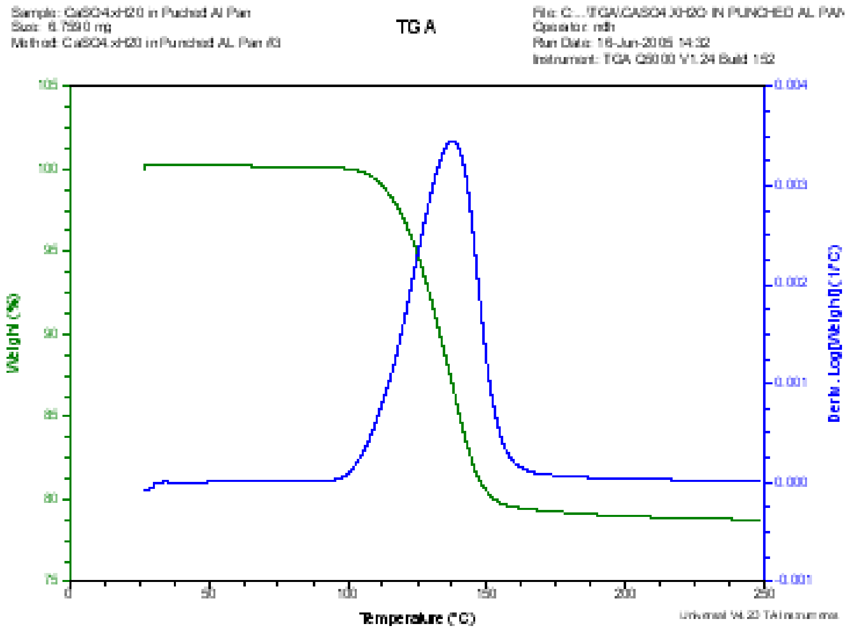


Figure 9. Gypsum in Punched Pan

The combined results show that TA Instruments has pan configurations to satisfy all common requirements that a customer may have for analyzing samples by TGA.

PLATINUM SOFTWARE

A highly valuable use of the Q5000 IR autosampler is with our latest Advantage software with Platinum features to schedule and initiate, at any time and date suitable to the user, a sequence of calibration, verification and diagnostic tests designed to ensure that the analyzer is constantly kept in maximum operating condition. These events could be mass, weight loss and temperature calibration (Curie Point) experiments as well as checks on key component positioning, sensor, furnace and mass flow control elements of the system. These experiments can be scheduled at quiescent times, such as weekends, when the instrument is not in regular operation. The Platinum software also has the ability to automatically notify the user when the experiment has been completed and if required with the results of the operation. This capability is found in no other TGA.

SUMMARY

These, and many other design features, permit the Q5000 autosampler to set new levels of performance and operational convenience in thermogravimetric analysis.

KEY WORDS

Q5000 IR TGA, Autosampler, Aluminum sealed pans, Punched pans

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2. Q5000 TGA Features to Ensure and Maintain Optimum Performance, TA Instruments Applications Brief (TA 327), 2005.

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