Aluminum Sealed / Punched Pan for use with Q5000IR and Q5000 SA Instruments

Nathan Hesse Ph.D. and Robert Hassel Ph.D
TA Instruments, 109 Lukens Drive, New Castle, DE 19720, USA

ABSTRACT
This document provides a summary of the use in thermogravimetric analysis (TGA) of aluminum sealed / punched pans to solve applications involving environmentally sensitive samples.

INTRODUCTION
The integrated autosampler on the TA Instruments Q5000 IR Thermogravimetric Analyzer (TGA) and the Q5000 SA (Sorption Analyzer), shown in Figure 1, was designed for use with a 25-position sample tray, which is included in the Q5000 IR and can optionally replace the 10-position tray, which is standard on the Q5000 SA. The advanced design of the autosampler includes three motors, which serve to smoothly load and unload the sample pans without disturbing the thermobalance from its null position. In addition to the platinum, aluminum and ceramic pans commonly used in TGA, the autosampler design also includes a new pan-punching feature (Figure 2), which allows for samples to be encapsulated for isolation from environmental conditions and then be punched open just prior to analysis.

Figure 1. Q5000 IR

Q5000 SA

Figure 2: Q5000 IR Autosampler Tray and Aluminum pan in “punch” position

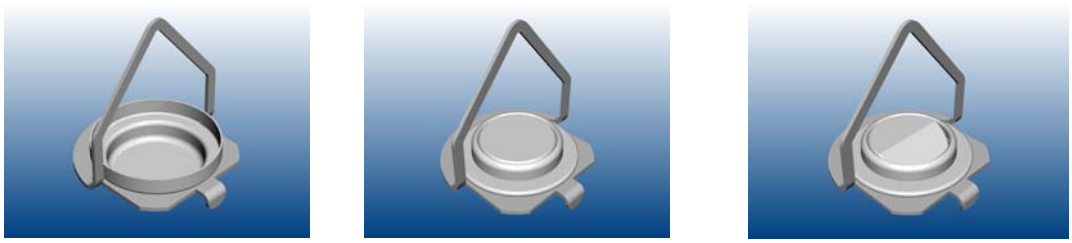
Q5000 PAN TYPES

A variety of sample pans are available for use with the Q5000 Series instruments. Platinum (50µL; 100µL), alumina (100µL; 250µL) open pans are commonly used with the Q5000 IR, while 180 µL semispherical metal coated quartz crucibles are standard with the Q5000 SA (Figure 3).

Figure 3 (left to right): Alumina, Platinum and Quartz Sample Pans

ALUMINUM TGA PANS

In TGA, aluminum pans are commonly used as disposable capsules for analysis of materials up to 600 ºC. The aluminum pan designed for the Q5000 IR and Q5000 SA offers a choice for use as an open or sealed pan depending on the desired application. The pan is supported by a separate reusable stainless steel carrier (or bail) as shown in Figure 4. The sealed aluminum pan allows automatic analysis of samples that traditionally needed to be analyzed manually.



Open Sealed Punched

Figure 4. Q5000 Aluminum Open and Sealed Pans in SS Bail

SEALED ALUMINUM PANS

When using sealed pans, the autosampler punches the pan just prior to loading the sample into the TGA furnace. Sealed pans are useful for the analysis of sensitive materials that may change their composition or properties while awaiting analysis under ambient conditions. Sealing the sample in a DSC press eliminates the possibility of gain or loss of moisture, loss of other volatiles, oxidation or exposure to light.

Q5000 IR PAN PUNCHING MECHANISM

Figure 2 shows that the punching mechanism collapses a large section of the pan lid so that the sample is adequately exposed to the purge gas during the analysis. Since the "punch" is always on the upper surface of the lid, it cannot make contact with the sample and so cannot contaminate a subsequent sample as is possible with "awl" like piercing devices used by others.

The mechanism of pan punching is shown in Figure 5, in which the sample tray first rotates at the lower position to prevent the bail from contacting the "punch". When the selected pan position is taken, the sample tray is quickly elevated to the pre-punch position. There after the tray slowly rises until it makes contact with the punch. An integral sensor monitors the force applied to the pan lid during the punching operation. The punched pan is then loaded into the furnace for analysis. If an improper force profile is recorded, the pan will not be permitted to be loaded into the furnace.

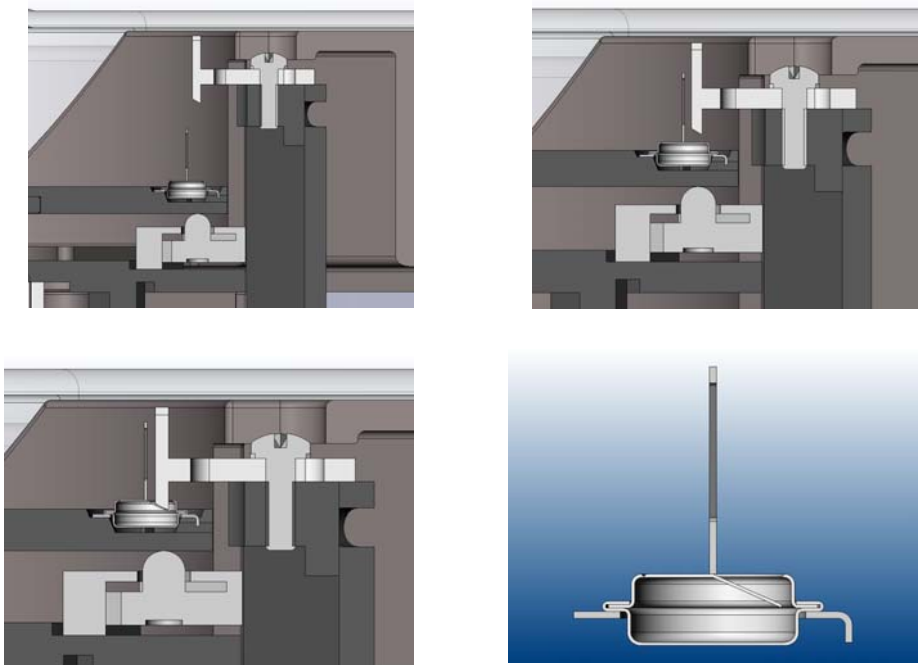


Figure 5. Pan Punching Mechanism

RESULTS AND DISCUSSION

To demonstrate the effect of use of pans that offered differing exposure of the sample to the purge gas, 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 DSC pan fits nicely into the stainless steel

bail. The pan types should allow significantly different sample exposure to the purge gas with the open pan offering most exposure, followed in decreasing order by the new "punched " pan and finally the pinhole restricted DSC pan.

Each sample was automatically loaded into the Q5000IR 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 6 shows duplicate results for each pan type. The results were in accordance with the prediction. In all cases the expected stoiciometric 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.

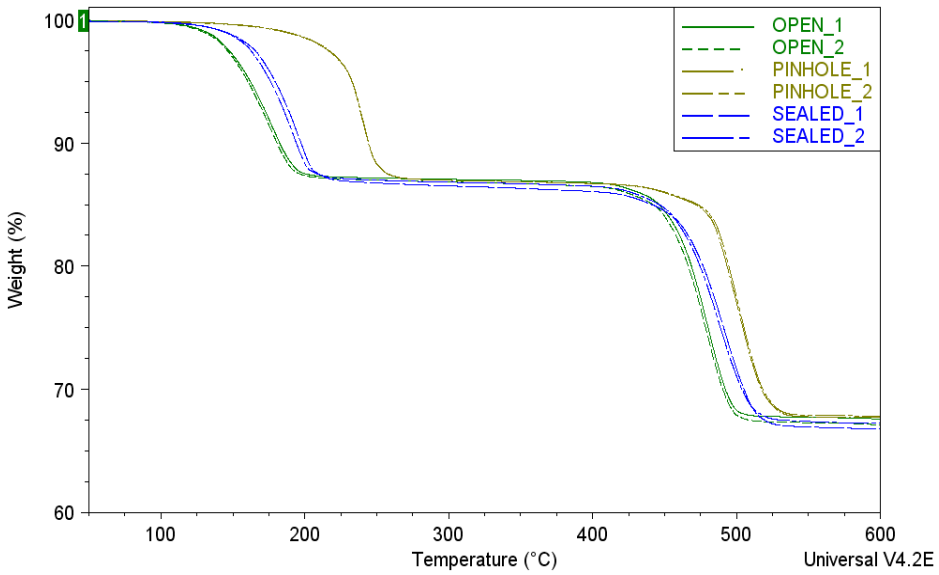


Figure 6. Calcium Oxalate Monohydrate – Multiple TGA pans

SORPTION ANALYSIS

The sealed aluminum pan approach can also be used for sorption analysis of materials, where long-term exposure of the sample to the prevailing laboratory conditions prior to analysis is undesirable. For example, drying of paints is a process that is strongly affected by the temperature and humidity of the environment where the paint is being used. The Q5000SA with the sealed / punched aluminum pans offers an excellent way to study and compare different paint formulations. Figures 7 and 8 show Q5000 SA results of a latex based paint (~ 20 µL) at various degrees of temperature and humidity. Additional tests for this formulation confirmed that temperature is a more significant factor on drying than is humidity.

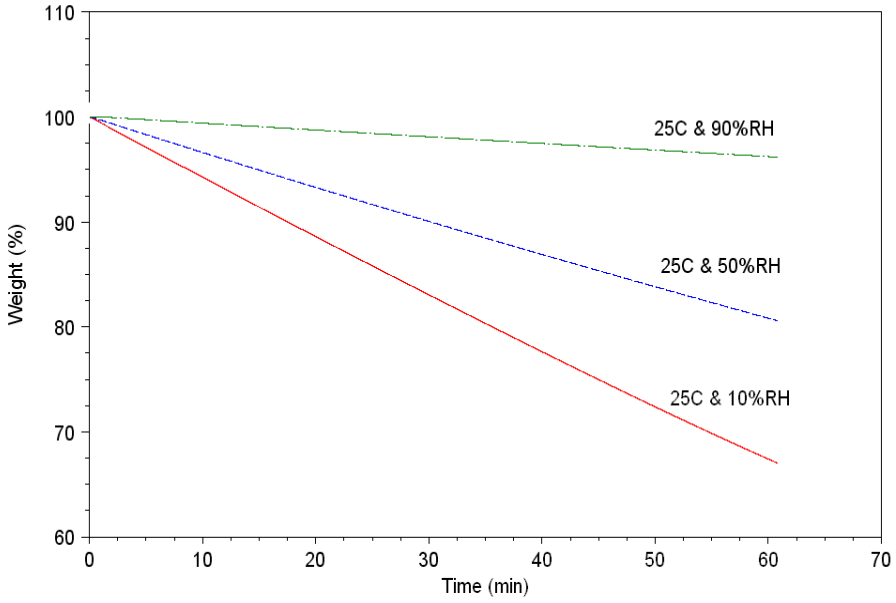


Figure 7. Drying of paint at 25 ºC and varying humidity in the Q5000 SA

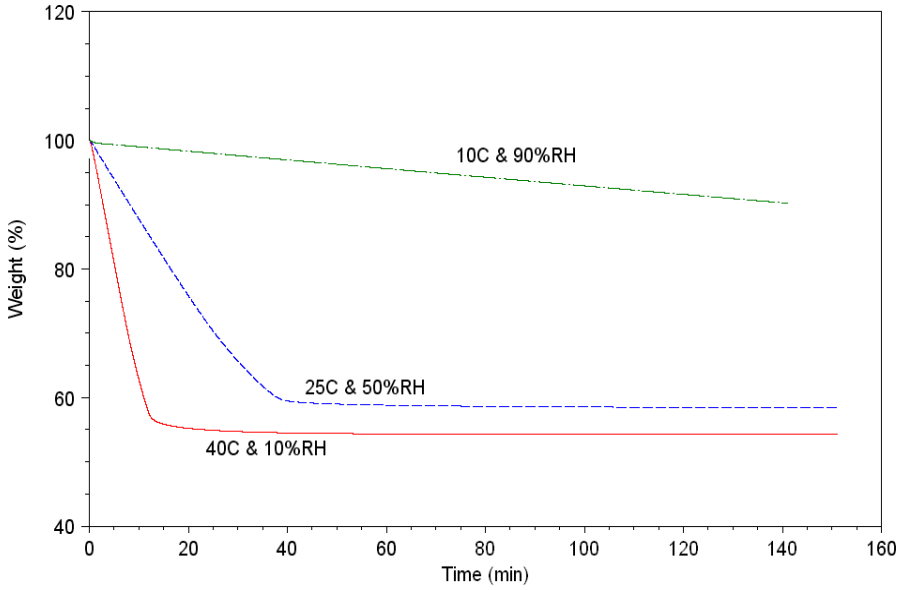


Figure 8. Drying of paint at various temperatures and humidities in the Q5000 SA

Polyvinylpyrrolidone (PVP) is a well-known material used for moisture sorption verification. It is hygroscopic and gains 42 % +/- 2 % of moisture when exposed to a relative humidity (RH) >/= 80 %. Figure 9 shows Q5000 SA results on PVP when held at 25 ºC and 80 %RH. The green plots represent PVP loaded in open pans, which were left in the autosampler for 8 and 24 hours prior to analysis. The red plot represents PVP

loaded into the sealed pan that remained in the autosampler for 24 hours prior to analysis. The weight gain from the PVP in the sealed pan was 41.25 % in the experiment, which was in good agreement with the expected 42 % value. In contrast, the samples loaded in the open pans gained about 6 % less weight due to adsorption of ambient moisture prior to analysis.

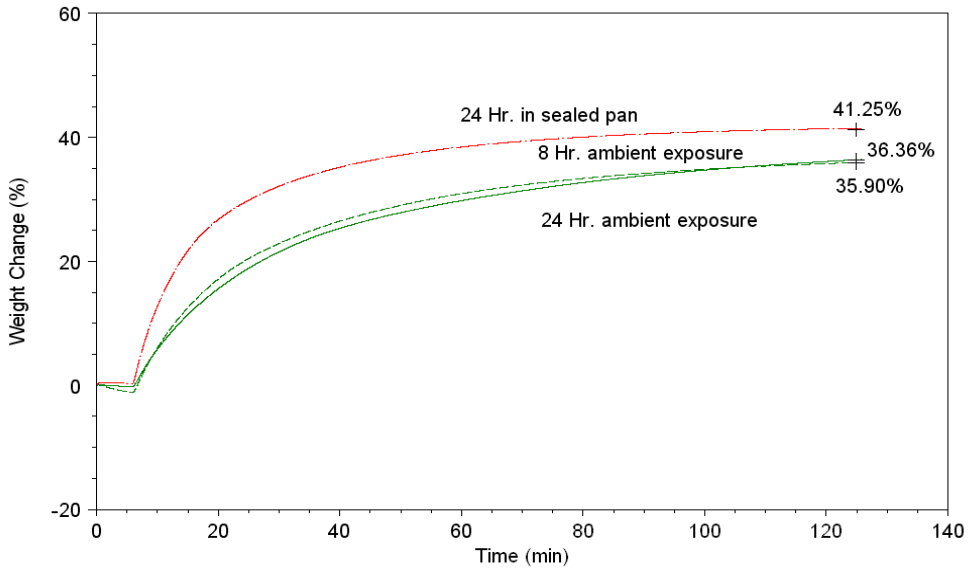


Figure 9. Dried PVP after Exposure to Ambient Conditions

CONCLUSION

A series of sample pans are available for use with the Q5000 IR TGA and the Q5000 SA Sorption Analyzer. The aluminum sealed / punched pans are shown to be very useful for the automatic analysis of materials that are sensitive to ambient conditions. The automatic punching mechanism integrated into the autosampler opens the pans just prior to analysis and avoids cross contamination of subsequent samples. This allows the automated collection of reproducible weight gain / loss results.

KEY WORDS

Q5000IR, Q5000SA, TGA, Moisture analysis, Sealed / punched aluminum pans.

TA INSTRUMENTS

United States, 109 Lukens Drive, New Castle, DE 19720 • Phone: 1-302-427-4000 • Fax: 1-302-427-4001
E-mail: info@tainstruments.com

Spain • Phone: 34-93-600-9300 • Fax: 34-93-325-9896 • E-mail: spain@tainstruments.com

United Kingdom • Phone: 44-1-293-658900 • Fax: 44-1-293-658901 • E-mail: uk@tainstruments.com

Belgium/Luxembourg • Phone: 32-2-706-0080 • Fax: 32-2-706-0081
E-mail: belgium@tainstruments.com

Netherlands • Phone: 31-76-508-7270 • Fax: 31-76-508-7280
E-mail: netherlands@tainstruments.com

Germany • Phone: 49-6196-400-600 • Fax: 49-6196-400-6666060 • E-mail:
germany@tainstruments.com

France • Phone: 33-1-304-89460 • Fax: 33-1-304-89451 • E-mail: france@tainstruments.com

Italy • Phone: 39-02-2742-11 • Fax: 39-02-2501-827 • E-mail: italia@tainstruments.com

Sweden/Norway • Phone: 46-8-555-11-521 • Fax: 46-8-555-11-520
E-mail: sweden@tainstruments.com

Japan • Phone: 813 5479 8418 • Fax: 813 5479 7488 • E-mail: **j-marketing@tainstruments.com**

Australia • Phone: 613 9553 0813 • Fax: 614-1374-9030 • E-mail: sshamis@tainstruments.com

To contact your local TA Instruments representative visit our website at www.tainst.com