MOST COMMON DSC PANS.

My summary based on information from Nick Hawkins' (ASM engineer) advice (he has already approved it) and from "the Selecting a sample pan TA theory" (attached to this word too).

A)- Standard pans plus standard (non-hermetic) lids (green dyes): maximize the quality of the heat flow measurement. The lid is usually used to flatten the sample against the bottom of the pan for improved thermal contact. In both configurations crimped pans provide the opportunity for good sample–atmosphere (purge gas) interaction.

However, these types of lids are not sealed to prevent the loss of volatiles present in or generated by the sample during the experiment. Therefore, these pans release the volatiles without falling from the cell platform, you can help the release of pressure making a pin hole.

Often, if these volatiles are allowed to evaporate the endothermic heat flow associated with the vaporization obscures the transitions of interest (specially with water).

In some cases, the sample itself can flow out of these pans at higher temperatures/pressure and contaminate the cell, which is more unlikely in hermetic pans with a pin hole.

USE them for:

Uneven samples and even/flat solids (like powders) study Tg (glass transitions, crystallizations, crosslinking…)

As long as: (conditions of use)

1) NO big changes in volume/pressure can happen (for example these pans are not for liquids, or samples going through melting or suffering big volatiles releases) since they can easily fall, they have small volume to cope with these changes.

Ideal: for dry samples unlikely to evolve significant amounts of volatiles. *These samples could also be studied in Tzero pans with hermetic lids, but:*

- *Standard pans maximize the quality of the heat flow measurement better results (especially reproducibility), better contact, especially with uneven samples (the lid is crimped against the sample).*

Therefore, they are better than standard Tzero pans (with normal lids)

- *Many hermetically sealed aluminium pans cannot withstand high internal pressure and will deform (sample leakage and busting can occur, resulting in contamination and artefacts in the signal) with increasing T. When these hermetic lids are used the best is to make a pin hole, ideally one big enough or two small ones.*

2) NO need for high amounts (for higher amounts use Tzero or special pans, which are bigger). Only fill the bottom of the pan.

3) Do not use them when you don't want that sample volatiles are released.

B) Hermetic pans, however, do provide a poorer thermal contact between the sample, pan and disk. This fact, plus the added mass of the hermetic pans and covers, leads to a slight loss of resolution compared to the crimped pans. The calorimetric accuracy is not affected, only the time constant of the system and the resolution of the measurement.

C) Tzero pans plus hermetic lids (without a pin hole):

Use them for experiments where volatiles can't be released (example: food and other water-containing samples analysis, studies of volatiles, liquids, gels, materials that subline and generate corrosive gases…). Maximum T: not much higher than 100C.

NICK HAWKINS' COMMENTS about the use of these pans for food analysis: *the T zero pans/hermetic lids should be fine for measuring food samples with water content. As you rightly said though, there will be a limit to which these can be used at elevated temperature. The pans will at some point burst as the internal pressure builds with temperature. When they rupture the pressure release is normally controlled and so there is minimal risk to damaging or contaminating the cell.For weak transitions and/or the need to heat to higher temperatures in a sealed environment the High pressure Stainless Steel pans are ideal.*

Remember that for uneven solids you get a better contact with standard pans with standard lids.

- Tzero pans plus hermetic lids (with a pin hole): higher volume than standard pans and they can hold the sample at higher pressures.

Use them for analysis where big changes in volume/pressure can happen (for example liquids, or samples going through melting or suffering big volatile releases) but when volatiles can be released without affecting results.

Remember that for uneven solids you get a better contact with standard pans with standard lids.

D) Special pans: Aluminum Alodined Pan, gold pans, copper pans, Tzero Low-Mass Aluminum.

SEE AT THE END MORE INFO ABOUT THESE PANS.

- For samples which contain large **amounts of evolved water**, it may be preferable to use the Tzero Hermetic Aluminum Alodined Pan.

Pedro: I have several food researchers and we have used Tzero pans, without having seen any apparent reaction.

NICK HAWKINS' COMMENTS: Alodined Aluminium pans are pans which have been coated to increase their resistance to corrosion. ***They are normally used if it is suspected that a sample may react with Aluminium*** *(Particularly materials that are Basic in nature).* ***I have not come across the issue of Aluminium reacting with water/steam at high temperature****. This is certainly not why the alodined pans were developed - they were developed to provide increased inertness for the containment of samples.*

- I have also seen **that high volume pans** are particularly useful for measuring weak transitions in aqueous-based biological and food samples

NICK HAWKINS' COMMENTS:

The stainless steel ***High volume Aluminium pans are very good for measuring weak transitions*** *(such as denaturation for example) in foods and Biological samples. I have used them a lot. However, I do not buy them from TA Instruments because they are so expensive. The ones I purchased were from Paul Clarke at PETA solutions.*

The larger pans allow much bigger samples to be measured - this is the only way to generate enough signal for weak transitions (such as denaturation) to actually detect them by DSC. Signal to noise ratio isn't compromised but resolution is (not hugely though) because of the increased mass. You can only run them using low heating rates (Maximum 5C/Minute).

I used them a lot to measure the denaturation of skin (human and animal), denaturation of native silk, denaturation of keratin in hair, starch gelation (low concentrations of starch) and they worked perfectly. These are all very weak transitions because of the low concentrations involved.

You do not need a sample press to seal them either. I worked out a way of sealing them using a small steel bar and they work perfectly.

Because there are no contact resistance values stored in the Q2000 DSC for these pans (or the TA equivalent pans in fact), you can only run them using the T4 mode of operation. This does not require any baseline calibrations to be repeated. You will however have to recalibrate for temperature and cell constant.

The pans weigh approximately 300 mg and they can be heated to >200C before they release pressure - this is fine for most foodstuffs and biological solutions. They have a volume of 100 ul so you can run approximately 3X the amount of sample that you can place in a T zero pan. For weak transitions it is the only way to detect them.

Unfortunately these pans are not re-useable so it will cost approximately £10 per run !!

Let me know if/when you intend to use them and I can come in *and show you the tricks for good results.*

SUMMARY:

Pan Type	Pan	Lid	Die Set	Software Pan Type	Application
Tzero Aluminum	Tzero Pan	Tzero Lid	Black	Tzero Aluminum	Basic DSC/MDSC applications ACORDING TO NICK (he did a comparison study between both types) BETTER STANDARD PANS, also cheaper. See text.
Tzero Hermetic Aluminum	Tzero Pan	Tzero Hermetic Lid	Blue	Tzero Hermetic Aluminum	DSC applications which require hermetic seals See text to know when apply a pin hole. *Much better (advised by Nick and based on my experience) than Standard Aluminium Hermetic pans which have very small volume*
Tzero Hermetic Aluminum Alodined	Tzero Alodined Pan	Tzero Hermetic Alodined Lid	Blue	Tzero Hermetic Aluminum Alodined	DSC applications which require hermetic seals and may evolve water
Tzero Low-Mass Aluminum	Tzero Low-Mass Pan	Tzero Lid	Black	Tzero Aluminum	High-sensitivity for low mass of sample

Special considerations when preparing the sample:

Taken from the book

Reactions with the pan

Samples that react with a pan can cause serious damage to an analyser since they may also react with the furnace beneath. Solder pastes and inorganic salts are typical of the type of samples where care must be taken. If in doubt check it out separately from the analyser, and then choose a pan type which is inert. Sometimes the effect of catalysis is of interest and copper pans may be used to provide a catalytic effect. Aluminium pans are normally made of very high-purity metal to prevent unwanted catalytic effects.

1.3.1.6 *Liquid samples*

Liquid samples must be placed in sealed pans of a type that can withstand any internal pressure build-up. Do not overfill the pan or contaminate the sealing surfaces, which will prevent sealing and cause leakage. When sealing a liquid, bring the dies together gently to avoid splashing the sample.

SAMPLE CONTACT.

Samples **need to be in good thermal contact** with the pan.

- Liquids and powders, when pressed down, give good thermal contact, other samples should be cut with a flat surface that can be placed against the base of the pan.

- Avoid grinding materials unless you are sure it will not change their properties.

- If possible, films should not be layered to prevent multiple effects from the same transition, though this may be the only way to get enough samples. If so, take care to make sure that the films are pressed well together.

- Low-density samples provide poor heat transfer so should be compressed. Some crimping processes do this automatically; with others it may be of value to compress a sample between two pan bases or using a flat pan lid as an insert.

- Take care not to deform a pan and discard any pans that are obviously deformed before use. Sometimes pans of slightly thicker aluminium can give better heat transfer because they retain a flatter base.

1.3.1.8 *Spillage*

A frequent cause of contamination is from sample attaching to the outside of the pan. Check and remove any contaminant sticking to the outside of the pan, particularly the base of the pan. A soft brush is good for removing powders.