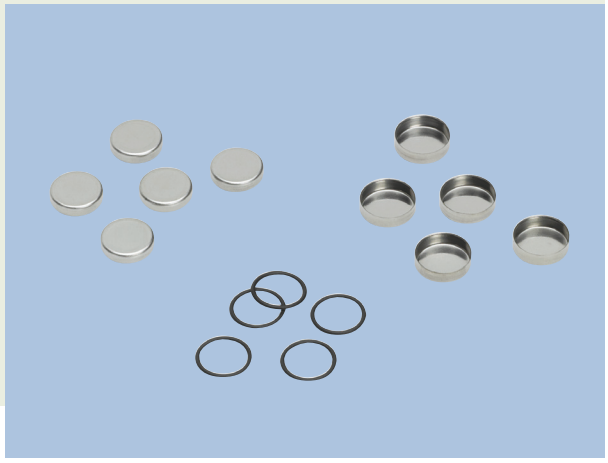
TECHNICAL NOTE

Thermal Analysis

Guide to Selection of Differential Scanning Calorimetry (DSC) Sample Pans



DSC measurement requires that samples are enclosed in a sample pan in order to avoid the direct contact between sample and furnace and/or sensor, because if the samples come into contact with the furnace/sensor problems can occur on the baseline. Memory can be affected and damage to the furnace/sensor can occur (in the worst case the furnace/sensor can be destroyed).

All of our DSC (DSC 4000/6000/8000/8500) include a pack of 100 standard sample pans in aluminum (N5390123), but many other different sample pans are available in different shapes and in different materials to handle any kind of samples and for all possible measurements. What follows is some advice on how to select the proper sample pan.

PerkinElmer®
For the Better

Solids and Powders (not decomposing, not sublimating/evaporating)

1. The standard aluminum sample pans and covers (02190041) can be used for solids and powders that do not decompose or boil in the range of -170° to 600 °C. They should not be used over 600 °C, as that approaches the melting point of aluminum at 660 °C. The typical measurements are glass transition; melting; curing; and oxidative induction test; and most samples are organic samples, such as plastics and polymers. These pans can also be used for metals and for inorganics (i.e. cement or rocks) provided that they do not react with aluminum. The standard pans are not suitable for liquids due to continuous evaporation. In addition, standard pans are not suitable for greases or creams because they can overflow from the pan due to their high fluidity. Standard pans can be closed manually bending the edges with tweezers or using the standard crimper press (Figure 1) or universal crimper press (Figure 2) with the adapter (B0508921).

2. If it is necessary to go above 600 °C, or if aluminum is reacting with the sample, use the standard sample pans and covers in copper, gold, graphite, and alumina (part numbers appear in Table 1). Please note that only the standard copper pans and covers can be crimped (using the same crimpers as standard aluminum pans), while in case of gold, graphite and alumina, the sample is placed inside the pan with the uncrimped cover on top of the sample.

3. If high sensitivity and high reproducibility of results are required, use the heat treated, high purity standard aluminum sample pans (N5340188). They are the same as part number 02190041, but they are cleaned with a solvent and then dried in an oven at a high temperature in order to remove any organic residue to get a better baseline.

4. When running an experiment with HyperDSC® (high heating and cooling rates: 300 °C/min, or more) it is possible to use pans with reduced mass (N5203115) in order to improve performance of the DSC. These pans are made of very thin aluminum foil and do not require a crimper. Of course, they are not sealed and the user must take care to avoid sample decompositions and/or overflows from the pan.

Figure 1. Standard Sample Pan Crimper Press are used to crimp covers on standard DSC pans of aluminum, gold, and copper. Design incorporates a replaceable crimper head for use with Crimp Pans (02190041 and 03190026) and Replacement Crimper Head (02191171).
Part No. 02190048

Figure 2. The Universal Crimper Press provides high quality pan sealing for the autosampler sample pans/hermetically sealed pans and can also crimp standard aluminum pans, large volume stainless steel (24 bar), and volatile pans. This is achieved simply by using the appropriate sealing insert. The Universal Crimper Press includes sample pan suction tool and inserts to seal 10, 30, and 50 µL sample pans.

Requires:
Insert (B0144637) for Volatile Sample Pans (02190062, 02190080, and N5190788); insert (B0505340) for Stainless Steel Pans (03190218 and 03190029); insert (B0508921) for Standard Aluminum Pans (02190041); and insert (B0139033) for hermetically sealed 10, 30, and 50 µL Pans are included.
Part No. B0139005

Table 1. Sample pans and covers (not-hermetically sealed).

Description	Part No.	Temp. Range	Vol. (µL)	Max. Pressure	Qty.	Crimper
Standard aluminum sample pans and covers	02190041	-170° to 600 °C	40	Ambient	400	02190048 or B0139005 with B0508921
Alumina pans and covers	N5190180	-170° to 750 °C	40	Ambient	6	Not required
Standard copper pans and covers	03190026	-170° to 750 °C	40	Ambient	200	02190048 or B0139005 with B0508921
Standard gold pans and covers	02190042	-170° to 750 °C	40	Ambient	10	02190048 or B0139005 with B0508921
Graphite pans and covers	03190025	-170° to 750 °C	40	Ambient (inert)	4	Not required
HyperDSC aluminum sample pans, low mass	N5203115	-170° to 600 °C	100	Ambient	100	Not required
Standard aluminum sample pans and covers, heat treated, high purity	N5340188	-170° to 600 °C	100	Ambient		02190048 or B0139005 with B0508921
Aluminum pans for Photocalorimeter	B0196858	-170° to 600 °C		Ambient	100	Not required
Aluminum pans with quartz cover for Photocalorimeter	B0198030	-170° to 600 °C		Ambient	100	Not required
Quartz covers for Photocalorimeter	B0181091				10	Not required

5. Photocalorimeter and the hyphenated Raman/DSC require special open aluminum pans that are available with a quartz cover (B0198030) or without a quartz cover (B0196858). The quartz cover is used to avoid the sample from overflowing from the pan. With or without the quartz cover, these pans are not crimped.

6. Greases, creams, and all other samples that produce foams (or that melts in very fluid liquids) must be closed in sample pans that are not sealed, but contain the sample better. For this, use the volatile aluminum sample pans (B3143015, B3143016, or B3143017) with pierced cover (B7001014). They all require the universal crimper (B0139005).

Solids/powders (that can have decomposition, sublimation, evaporation), liquids

7. In order to avoid decomposition, evaporation, sublimation, we have to use the volatile aluminum sample pans, which have a wide range of dimensions, volumes, and pressures. The aluminum sample pan (02190062) has an internal volume of 20 microliters seal, which can resist up to 2 bar of internal pressure and requires the crimper (part no. 02190061 as shown in Figure 3), or the universal crimper (B0139005) with the adapter (B0144637). Typical application use would be for analyzing purities of pharmaceutical compounds. Please note that these sample pans are not suitable for autosamplers.

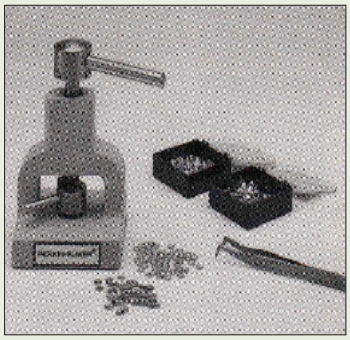


Figure 3. Volatile Sealer Assembly
Part No. 02190061

Replacement Lower Die for Sealer
Part No. 02191295

Replacement Upper Die for Sealer
Part No. 02191296

Table 2. Volatile sample pans and covers suitable for autosamplers.

Description	Part No.	Temp. Range	Vol. (µL)	Max. Pressure	Qty.	Crimper
Aluminum pans and covers starting kit	B0510800	-170° to 600 °C	assorted	3 bar	50 each of 7 types	B0139005
Aluminum (vented) pans	B0143018	-170° to 600 °C	30	Vented	400	B0139005
Aluminum (vented) pans	B0143019	-170° to 600 °C	50	Vented	400	B0139005
Aluminum pans	B0143015	-170° to 600 °C	10	1 Bar	400	B0139005
Aluminum pans	B0143016	-170° to 600 °C	30	1 Bar	400	B0139005
Aluminum pans	B0143017	-170° to 600 °C	50	1 Bar	400	B0139005
Aluminum covers (pierced)	B7001014	-170° to 600 °C		0.05 mm Hole	400	B0139005
Aluminum covers	B0143003	-170° to 600 °C		for 1 Bar Pans	400	B0139005
Aluminum pans and covers	B0169319	-170° to 600 °C	10	3 Bar	400	B0139005
Aluminum pans and covers	B0169320	-170° to 600 °C	30	3 Bar	400	B0139005
Aluminum pans and covers	B0169321	-170° to 600 °C	50	3 Bar	400	B0139005
Aluminum pans for Photocalorimeter	B0196858	-170° to 600 °C		ambient	100	Not required
Volatile sample pans and covers *NOT* suitable for autosamplers						
Aluminum pans and covers	02190062	-170° to 600 °C	20	2 bar	400	02190061 or B0139005 with B0144637
Aluminum pans and pierced covers (0.05 mm hole)	N5190788	-170° to 600 °C	20	Vented	100	02190061 or B0139005 with B0144637

8. The volatile sample pans and covers suitable for an autosampler (Table 2) have similar applications as 02190062 but have better versatility, as they can be used with or without the autosampler. They are available in different volumes (10-30-50 microliters) and can resist up to 1 or 3 bar of internal pressure, according to the thickness of the pan. They require the universal crimper (B0139005).

9. There are applications that require resistance to more than 3 bar, for example, the study of denaturation of proteins in water solution, or water dispersions, or samples with a big concentration of water inside. In these cases, it is recommended to use the large volume stainless steel sample pan (03190218 or 03190219) package of 1.000 units. These sample pans include a base and a cover in stainless steel and an O-ring in Viton® with an operating range from -40 °C up to 300 °C and an internal pressure resistance of up to 24 bar. The typical applications for these pans are to study water solutions, suspensions, and emulsions. These pans require the "Quick press" (03190021), shown in Figure 4 or the Universal Crimper (B0139005) with the adapter (B0505340).

Figure 4. Large Volume Stainless Steel Capsule Kit
Part No. 03190021

Kit Includes:

Quick Press
Part No. 09908467

Quick Press Spacer Die
Part No. 03191047

Guard Ring Inserts, 1 pair
Part No. 03190236

20 Pans, Covers, and O-Rings
Part No. 03190218

10. 24 bar or 300 °C limits may not be enough for some applications such as polymerizations with by-product evolution, polymerization in water solutions or suspensions, study of water solutions at high temperatures or when we want to avoid decomposition of explosive samples. In these cases use the high pressure re-usable sample pans. These sample pans are practically a mini-autoclave, they include a base, a gasket and a cover, are available in stainless steel, stainless steel gold plated and titanium while gaskets are in gold plated stainless steel or in titanium. The internal volume is 30 microliters and they can resist an internal pressure of up to 150 bar. The high pressure pans are the only re-usable pans in the provided list, all other pans are disposable, with the exception of alumina and gold standard pans that can be cleaned and re-used. High pressure pans require the press (B0182864) shown on Figure 5 while the part numbers of pans are listed on Table 3. Please note that considering the mass of these pans, the heating rate should not exceed 20 °C/min.

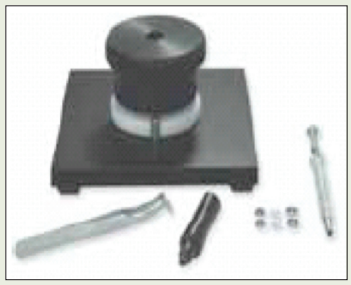


Figure 5. High-Pressure Capsule Sealing Tool

This reliable sealing tool is fast and precise. It employs a built-in torque clutch, which guarantees that all capsules are closed with the same torque.

For use with Seal Pans (B0182901, B0182902, and B0182903).
Part No. B0182864

Table 3. Large Volume S/S Volatile Sample Pans.

Description	Part No.	Temp. Range	Vol. (µL)	Max. Pressure	Qty.	Crimper
Stainless steel pans, covers and O-rings	03190218	-40° to 300 °C	60	24 Bar	20	03190021 or B0139005 with B0505340
Stainless steel pans, covers and O-rings	03190029	-40° to 300 °C	60	24 Bar	1000	03190021 or B0139005 with B0505340
High Pressure Re-Usable Sample Pans.						
Stainless steel pans and covers plus gold plated seals	B0182901	-170° to 400 °C	30	150 Bar	5 Pans and 20 Seals	B0182864
Gold plated pans, covers and seals	B0182902	-170° to 400 °C	30	150 Bar	5 Pans and 20 Seals	B0182864
Titanium pans, covers and seals	B0182903	-170° to 400 °C	30	150 Bar	5 Pans and 20 Seals	B0182864
Gold plated seals	B0182905				10	
Titanium seals	B0185054				10	

Special measurements and recommendations

11. Measurement of latent heat of evaporation: the problem related with this measurement is that liquids are evaporating at all temperatures. For example, if a drop of water is placed on a standard sample pan evaporation starts at 40 °C and at 80 °C. The only way to measure latent heat of evaporation for liquids is to use the autosampler volatile pans (B0143015, B0143016, B0143017) with pierced covers (B7001014) and the universal crimper (B0139005).

12. Oxidative Induction Test (OIT): the best solution is to use the standard aluminum sample pan 02190041 without cover. DSC 4000/6000 pans without a cover can be used with or without an autosample, while the sampler pans for the DSC 8000/8500 can only be used without an autosampler. If it is necessary to perform OIT with DSC 8000/8500 and autosampler, use the aluminum vented pan (B0143018 or B0143019).

13. Measurements with the DSC high pressure cell (N5340288): the most common application is the oxidation test under pressure. In this case the furnace is pressurized and the pressure is applied to the sealed sample. The pan must not be hermetically sealed, but it must allow an exchange of gas. The sample pans to use are the standard aluminum pans (02190041) for solids or the autosampler aluminum volatile pans (B0143015, B0143016, B0143017) with pierced covers (B7001014) for liquids.

14. The vented aluminum pans (B0143018, BO143019) should only be used when performing OIT experiments in the autosampler version of the DSC 8000/8500. Because of the side vents in these pans, there is a greater risk of spilling material and containing the head than with the center hole version. However, they are the only vented pans safe to use with an DSC 8000/8500 autosampler.

15. Decomposition/combustion experiments: DSC is not the best instrument to perform these experiments because evolved products can pollute and also damage the furnace/sensor. It is better to use Thermogravimetric analysis (TGA) that is specifically designed to study decompositions and combustions. If it is mandatory to perform decompositions and combustions inside a DSC, use the standard aluminum sample pans without the cover, use very low scan rate 5 °C/min or maximum 10 °C/min, and a high flow rate of purge gas 50 mL/min or more. In case of DSC 8000/8500 it is also recommended to use the flow-through cover (N5340287) in order to immediately extract all evolved products.

16. Regarding shape and amount of sample: when using standard pans and solids, it is better to use very small pieces that are distributed on the bottom of the pan or use thin films trying not to exceed one half of the height of pan. When using the volatile aluminum pans with pierced covers, use a smaller sample size in order to avoid overflowing from the hole of the cover, which can occur during melting. When using volatile sealed aluminum pans, fill the pan with the sample in order to avoid trapped air that can deform the sealed pan by the effect of expansion due to the increase in temperature. These pans are available with different internal volumes to select the ones with the correct volume to be completely filled by sample without trapping air.

Figure 6. PerkinElmer sample pans

Figure 6, above, summarizes all of the available sample pans. Please note that platinum pans and gold volatile pans are no longer available. As an alternative to platinum, you can use copper, graphite, or alumina.

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