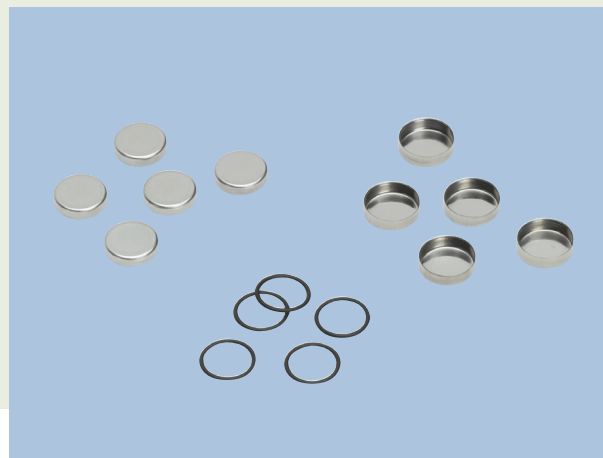


Thermal Analysis

Guide to Selection of DSC Sample Pans



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 effected 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 (Part No. 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. Here is some advice on how to select the proper sample pan.

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

1. The standard aluminum sample pans and covers (#02190041 sold in lots of 400) 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, oxidative induction test and most samples are organic samples such as plastics, polymers. These pans can be used also for metals and for inorganic (cement, rocks) provided that they do not react with aluminum. The standard pans are not suitable for liquids (because liquids are continuously evaporating, material is lost and endothermic evaporation is all that will show) and 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 preferably using the standard crimper press 02190048 (Figure 1) or the universal crimper press B0139005 (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 are on Figure 3. 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, 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 same as 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 and to get a better baseline.
4. When running an experiment with Hyper DSC (high heating and cooling rates: 300 °C/min or more) it is possible to use pans with reduced mass N5203115 in order to improve performances 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. 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. Universal Crimper Press. 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. 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 10, 30, and 50 µL Pans included.

Part No. B0139005

| 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 |
| Hyper DSC 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 |

Figure 3. Sample pans and covers (not-hermetically sealed).

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 quartz cover, these pans are not crimped.

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

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

7. In order to avoid decomposition or evaporation or 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 02190061 (Figure 4) 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.

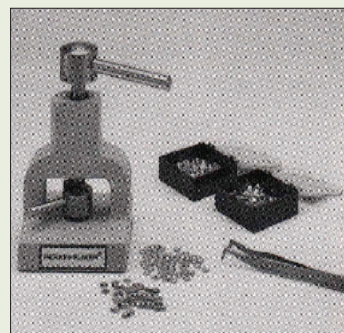


Figure 4. Volatile Sealer Assembly
Part No. 02190061

Replacement Lower Die for Sealer
Part No. 02191295

Replacement Upper Die for Sealer
Part No. 02191296

| 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 | B3143015 | -170° to 600 °C | 10 | 1 Bar | 400 | B0139005 |
| Aluminum pans | B3143016 | -170° to 600 °C | 30 | 1 Bar | 400 | B0139005 |
| Aluminum pans | B3143017 | -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 | B3169319 | -170° to 600 °C | 10 | 3 Bar | 400 | B0139005 |
| Aluminum pans and covers | B3169320 | -170° to 600 °C | 30 | 3 Bar | 400 | B0139005 |
| Aluminum pans and covers | B3169321 | -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 <i>NOT</i> 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 |

Figure 5. Volatile sample pans and covers suitable for autosamplers.

8. The volatile sample pans and covers suitable for an auto-sampler (Figure 5 – Lines 3-13) 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 package of 20 units 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 6 or the Universal Crimper B0139005 with the adapter B0505340.



Figure 6. 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 our price list, all other pans are disposable, with the only exception of alumina and gold standard pans that can be cleaned and re-used. High pressure pans require the press B0182864 shown on Figure 7 while the part numbers of pans are listed on Figure 8. Please note that considering the mass of these pans, the heating rate should not exceed 20 °C/min.



Figure 7. 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

| Description | Part No. | Temp. Range | Vol. (μL) | Max. Pressure | Qty. | Crimper |
|--|----------|-----------------|-----------|---------------|---------------------|------------------------------------|
| Stainless steel pans, covers and tygon O-rings | 03190218 | -40° to 300 °C | 60 | 24 Bar | 20 | 03190021 or B0139005 with B0505340 |
| Stainless steel pans, covers and tygon 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 | |

Figure 8. Large Volume S/S Volatile Sample Pans.

Special measurements and recommendations

- 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 B3143015/16/17 with pierced covers B7001014 and the universal crimper B0139005.
- Oxidative Induction Test: 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 and without an autosampler, 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, so 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 B3143015/16/16 with pierced covers B7001014 for liquids.
- 14) The vented aluminum pans numbered B0143018 and B0143019 should be only used when performing OIT experiments in the auto-sampler version of the DSC 85/8000. Because of the side vents in these pans, there is a greater risk of spilling material and containing the head that with the center hole version. However, they are the only vented pans safe to use with an DSC8500 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 a TGA that is specifically designed to study decompositions and combustions. If it is mandatory to perform decompositions/combustions inside a DSC use the standard aluminum sample pans without the cover, use very low scan rate 5 or maximum 10 °C/min and a high flow rate of purge gas 50 mL/min or more. In case of DSC 8000/85000 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, 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 that 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 9. PerkinElmer sample pans.

Figure 9 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 we can use copper, graphite or alumina.