Thermal Analysis

Crucibles Overview

DSC

TGA/SDTA

TGA/DSC

Crucibles

for Thermal Analysis

METTLER TOLEDO

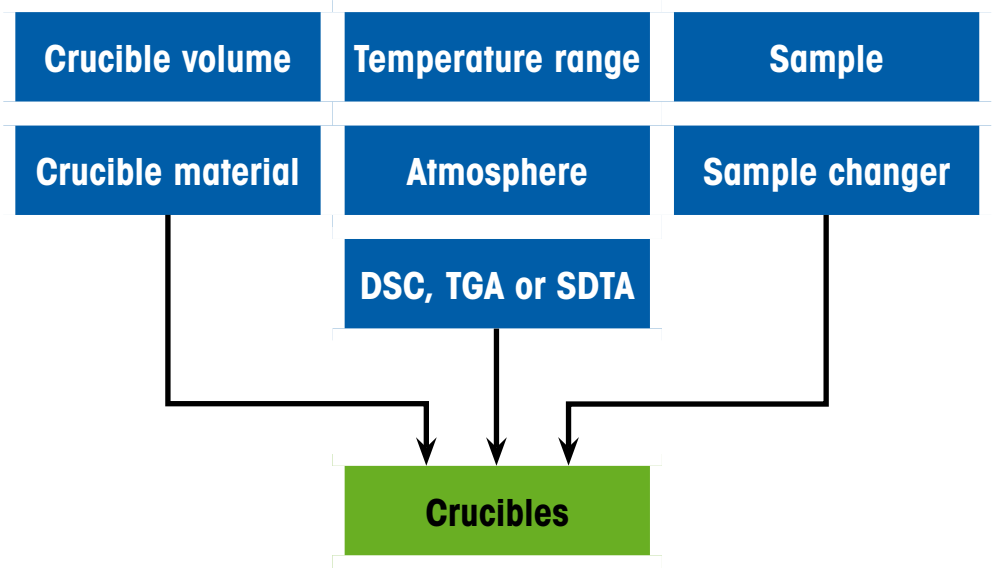
Crucibles for Thermal Analysis

Crucibles serve as containers for samples during thermoanalytical measurements. The type of crucible used for a measurement can have a large effect on the quality of the results obtained, and in addition, also influences important characteristics of the DSC measuring cell. Considering the relevant factors before the measurement can often help to save time later on when interpreting the curve.

The following points are particularly important for DSC and SDTA measurements:

- Make sure that the sample does not come into direct contact with the measuring cell, i.e. there is no contamination of the DSC measuring sensor or the SDTA/DSC sample holder.
- In the DSC measuring cell, the shape and the heat capacity of the crucible influence the specifications of the measurement system, such as for example the calorimetric sensitivity and the signal time constant. A short time constant results in sharp and well-defined peaks, and hence the best possible resolution (separation) of neighboring effects.
- Crucibles with a flat base that are made of materials of good thermal conductivity ensure optimum heat transfer with the lowest possible temperature gradients between the sample and the sample holder.
- The crucible material should normally be inert, i.e. it should not react with the sample in the temperature range used. Exceptions to this are the "copper stability" of polyolefines or lubricant greases and oils in copper crucibles, as well as certain reactions in platinum crucibles that also have a catalytic effect.
- The crucible material should not exhibit any physical transitions in the temperature range used, and the melting point should be sufficiently high.

Factors affecting the choice of a crucible



The Sample Robot
Precise and Reliable Like a Swiss Watch

The sample robot is very robust and operates reliably 24 hours a day and throughout the whole year.

Simple robust design

Universal gripper

Unique "wasp" crucible lid piercing device

Automatic and efficient
All DSC 1 models can be automated. The sample robot can process up to 34 samples even if every sample requires a different method and a different crucible.

No sample reaction before measurement
The sample robot can remove the protective crucible lid from the crucible or pierces the lid of hermetically sealed aluminum crucibles immediately before measurement. This unique feature prevents the sample taking up or losing moisture between weighing-in and measurement. It also protects oxygen-sensitive samples from oxidation.

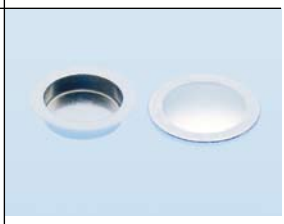
Extremely Wide Crucible Range

Aluminum crucible standard

40 µl crucibles with lids
Sets of 100 pcs
without pin ME-00026763
with pin ME-00027331

40 µl crucibles without lids
Set of 400 pcs
without pin ME-51119870

Piercing lid
Set of 400 pcs
ME-51119873



This is the standard type of pan for DSC measurements – it is very shallow (low in height) and has a strong flat base (this ensures that temperature gradients are as low as possible).
Hermetically sealed: to suppress the endothermic evaporation, vaporization or sublimation of volatile substances in the DSC. The maximum pressure is 0.2 MPa.
Comment: Particularly with TGA measurements using the sample changer, it is possible that the sample can partially dry out or take up moisture or oxygen from the laboratory air. This can be prevented with an aluminum lid. The lid (see cover) is automatically pierced before transfer to the measuring cell (3 needle diameters: 0.1 mm, 0.7 mm and 1.0 mm).
50 µm hole in the lid: for measurements in a self-generated atmosphere. Overlapping decomposition reactions are often better separated.
Large hole in the lid (0.35 mm to 2 mm): the atmosphere in the pan is practically the same as in the furnace, but substances are prevented from creeping out of the pan or spluttering.

Aluminum crucible light

20 µl crucible with lids
Set of 100 pcs
without pin ME-51119810

The light aluminum pan gives the shortest signal time constant, especially when using helium as a purge gas. The pan is particularly suitable for measuring polymer films, disks and powders – the samples are pressed down tightly against the base of the pan. It is less suitable for liquid samples because liquids might be squeezed out of the pan on sealing.
The narrow space between the pan and the lid leads to the formation of a self-generated atmosphere. Piercing the lid beforehand allows contact with the atmosphere. A special die set is required for the crucible sealing press.

Aluminum crucible

160 µl Tiegel mit Deckels
Set of 40 pcs
with pin ME-00027811

This very large crucible is used for DSC measurements of samples that exhibit very weak effects. Temperature gradients within the sample are to be expected because of the height of the crucible, which is the reason why measurement peaks are somewhat broader. For the same reason, heating rates of more than 10 K/min should not be used. The crucible is sealed just like a standard pan.

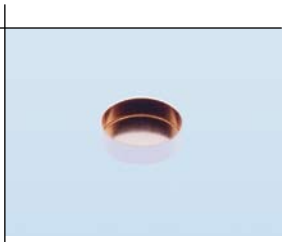
Aluminum crucible

100 µl crucible without lids
400 pcs
without pin ME-51119872

This crucible allows you to use larger quantities of sample if the signal obtained from the sample in a 40 µl pan is too weak.
The crucible is sealed just like a standard pan.

Copper crucible

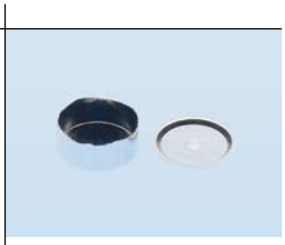
40 µl crucible without lids
100 pcs
without pin ME-51140407



The copper pan is supplied without a lid. It is almost exclusively used for the determination of oxidative stability (OIT) in the presence of copper, which exerts a catalytic effect. Usually the induction time measured in this way is compared with the value obtained with an inert aluminum pan.

Platinum crucible

Pt crucible with lids
Set of 4 pcs
without pin
30 µl ME-51140842
70 µl ME-51119654
150 µl ME-00024126



Platinum crucibles are mainly used for TGA or DSC measurements at temperatures above 640°C. SDTA and DSC curves measured with platinum crucibles are usually better than those obtained using crucibles made of alumina, which has a poorer thermal conductivity.
They can also be reused. After mechanical cleaning store them, if need be, in water (or even in 10% hydrochloric acid) because many salts are soluble in water. Oxides form chlorides in hydrochloric acid, which can then be removed by rinsing. After drying, heat the crucibles to red heat to ensure that no weight loss occurs when they are used.
Warning: Molten metals form alloys with platinum very easily. This can result in a hole being formed in the bottom of the crucible. And soot (or carbon black) is a so-called platinum poison in a non-oxidizing atmosphere. In the 1600°C furnace, a crucible can stick to the crucible holder, which is also made of platinum. This can be prevented by placing a sapphire disk (ME-00017759) on the crucible holder.

Gold crucible

40 µl crucibles with lids
Set of 6 pcs
without pin ME-00027220

The gold pan is chemically resistant and would be used much more frequently if it were not so expensive. Apart from some types of the aluminum pans, it is the only pan that can be hermetically sealed by cold welding. However, the gold surface becomes dirty during longer periods of storage, which makes cold welding more difficult. To clean the pan and lid, we recommend that you heat them to about 500°C for a short time prior to use (heat cleaning). The maximum pressure is 0.3 MPa. Warning: Molten metals easily from alloys with gold; this can result in a hole being formed in the bottom of the pan.

Medium pressure crucible
Stainless steel

120 µl crucibles with lids and Viton® O-rings
Set of 25 pcs
with pin ME-00026929
without pin ME-00029990

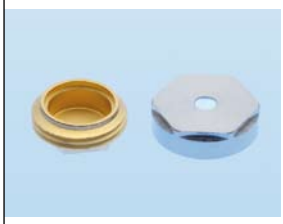
The medium pressure crucible is sealed with a Viton O-ring (a fluorinated elastomer made by DuPont). Viton is slightly permeable to water vapor. If this causes problems with aqueous solutions, O-rings made of Kel-F (polytrifluorochloroethylene, PCTFE from 3M) are also available (ME-00026933); PCTFE however shows a DSC melting peak at about 220°C. The maximum pressure is 2 MPa.
In order to seal the crucible, the crucible sealing press must be equipped with a special die set.
The crucible can also be sealed without using an O-ring (self-generated atmosphere). Finally, the crucible (and the lid) can be used individually as open crucibles (e.g. for TGA measurements).

High pressure crucible
Stainless steel

30 µl crucibles with lids,
seals not included
Set of 3 pcs
without pin ME-51140404

High pressure crucible
Stainless steel, gold plated

30 µl crucibles with lids,
seals not included
Set of 3 pcs
without pin ME-51140405



Seal
Copper, gold-plated

Set of 60 pcs
ME-51140403

The relatively light and flat construction of the crucible results in low temperature gradients. Compared to the larger pressure tight crucibles, it gives better DSC signals. The thread and the sealing tool with defined torque enable the crucibles to be easily and securely sealed. After the measurement, the crucible can be opened, cleaned and reused about 20 times using a new gold-plated copper seal each time. If the gold-plated crucible is used at temperatures above 350 °C, the crucible and the seal are welded together.

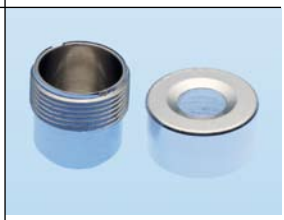
High pressure crucible
Stainless steel, gold plated

40 µl crucibles with lids and
seals
Set of 25 pcs
without pin ME-00026731
with pin ME-00026732

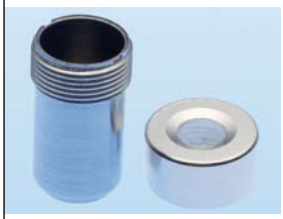
These gold-plated high pressure crucibles, which can be pressed together, have proven to be very useful for safety investigations. They can only be used for one measurement. The maximum pressure is 15 MPa.
The lid is pressed into the crucible with a pressure of about a ton so that the seal tightens the crucible.
A toggle press is used to close the crucible.

High pressure crucible
nimonic

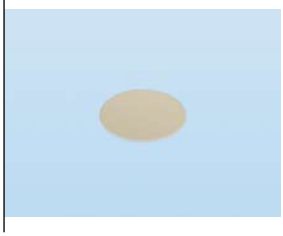
270 µl crucible with lid
1 pce
with pin ME-00650072



500 µl crucible with lid
1 pce
with pin ME-00650066



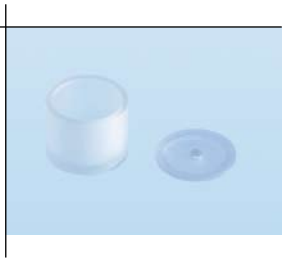
Seal
1 pce
ME-00027216



Nimonic 80A is a temperature resistant alloy made of Ni, Cr, Ti and Al. The crucible can be sealed thanks to its thread using a special sealing tool. After the measurement, it can be opened, cleaned and reused about 20 times with a seal disk each time. The maximum pressure is 10 MPa.
When sealed, the 270 µl crucible has a height of about 10 mm and is therefore too high for the DSC (the furnace body can be extended using the so-called furnace expander (ME-51140735) provided the sample changer is not used). The 500 µl crucible is 16 mm high and is therefore only suitable for use with TA4000 measuring cells with the flat lid.

Sapphire crucible
70 µl crucibles with lids

4 pcs
ME-51140845



Sapphire is a very pure form of monocrystalline aluminum oxide. This is the reason why a sapphire crucible is chemically more resistant than a polycrystalline alumina (aluminum oxide) crucible. The crucible is also recommended for melting metals such as Fe and Ni.

Alumina crucible
30 µl crucibles with lids
20 pcs ME-51140843

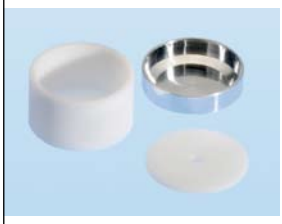
Special aluminum lids
40 pcs ME-51119649

Alumina crucible
70 µl crucibles with lids
20 pcs ME-00024123

Special aluminum lids
40 pcs ME-51119649

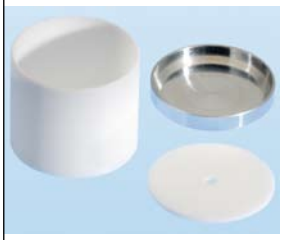
Alumina crucible
150 µl crucibles with lids
20 pcs ME-00024124

Special aluminum lids
40 pcs ME-51140477



Alumina crucible
900 µl crucibles with lids
4 pcs ME-51119960

Special aluminum lids
40 pcs ME-51140469

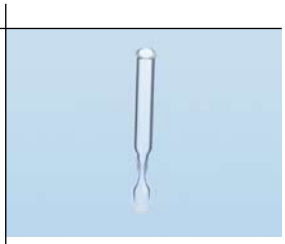


Alumina (aluminum oxide) **crucibles** are the crucibles that are normally used for TGA measurements, above all when the TG signal, and not the SDTA signal, is important. These crucibles can be reused. After mechanical cleaning, store them, if need be, in water (or possibly even in 10% hydrochloric acid) because many salts are soluble in water. Oxides form chlorides in hydrochloric acid, which can then be removed by rinsing. After drying, heat the crucibles to red heat to ensure that no weight loss occurs when they are used.

Special aluminum lids for the alumina crucibles and the sapphire crucible. They are removed by the sample changer during the TG measurement.

Glass crucible
100 µl

just crucibles, 50 pcs
without pin ME-00027812



These Duran glass crucibles have the advantage that they are transparent and are chemically resistant. The sample is filled through the neck of the glass crucible. The crucible is sealed by melting the neck in a small flame. A special holder (ME-00027815) is available that allows the sample to be cooled during sealing. The maximum pressure is 5 MPa.
When sealed, the crucible has a height of about 10 mm and is therefore too high for the DSC82x (the furnace body can be extended using the so-called furnace expander (ME-51140735) provided the sample changer is not used).

Crucible Sealing Press

The press allows the pan to be sealed very easily. Under the pressure of the plunger the pan is cold welded hermetically with the lid. After changing plunger and die you can use the press for other crucibles.

A

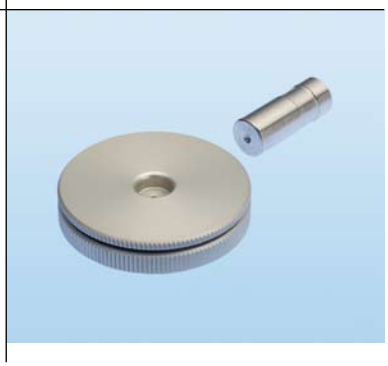
With these exchangeable assemblies you adjust the sealing press to the various crucibles.

A1

Die and plunger
for Al crucible light

A2

Die and plunger
for crucible, sealable by cold welding (included in delivery)



A3

Die and plunger
for medium pressure crucible

High Pressure Crucible Sealing Tool for Reusable Crucibles

B

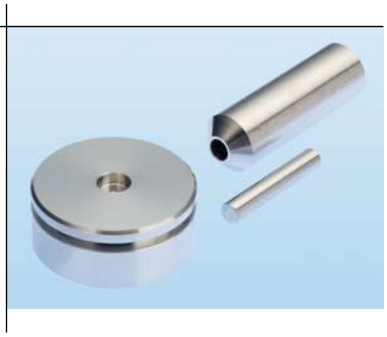
It consists of a lower part to keep the hexagonal crucible and of an upper part (turning head) that fits on the crucible cover. You close the crucible with the seal disk by turning until a sliding clutch is activated. After the measurement you open the crucible with the same tool.
ME-51119915

High Pressure Crucible Sealing Tool

C

This is the tool for cylindrical crucibles made of Nimonic. You fix the crucible in one of the tolls and the cover in the other to seal them with the required torque.
ME-00650067

Tool Kit for Toggle Press



D

This assembly fits in the Jossi press used to seal the 40 µl high pressure crucibles.
ME-00026733

Type	Designation	Part number
A	Crucible sealing press (incl. A2)	00119410
1	Die and plunger for Al crucible light	51140547
2	Die and plunger for cold hermetically welded crucible	00027809 and 00027386
3	Die and plunger for medium pressure crucible	00119428
B	High pressure crucible sealing tool, reusable	51119915
C	High pressure crucible sealing tool	00650067
D	Tool kit for toggle press (Jossi press, KP2.1N)	00026733

Crucible Datas

Part no	Designation	Quantity	Volume µl	With pin	Without pin	With lid	Without lid	Weight [mg]	Max. pressure MPa	Max. temperature °C	∅ without lid mm	Height mm without lid	Sealing tool
51119810	Aluminum crucible light	100	20		•	•		20	0.2	640	6	1.6	**A**1
00026763	Aluminum crucible standard	100	40		•	•		50	0.2	640	6	1.6	**A**2
51119870	Aluminum crucible standard	400	40		•		•	50	0.2	640	6	1.6	**A**2
00027331	Aluminum crucible standard	100	40	•		•		50	0.2	640	6	1.6	**A**2
51119872	Aluminum crucible medium	400	100		•		•	80	0.2	640	6	4.2	**A**2
00027811	Aluminum crucible large	40	160	•		•		100	0.2	640	6	6.4	**A**2
51119871	Aluminum lid standard	400						16		640			
51140832	Aluminum lid pierced 50 µm	400						16		640			
51119873	Aluminum piercing lid	400						16		640			
51140407	Cu crucible	100	40		•		•	70		750	6	1.65	
51140842	Platinum crucible small	4	30		•	•		220		1600	6	2.3	
51119654	Platinum crucible medium	4	70		•	•		285		1600	6	4.2	
00024126	Platinum crucible large	4	150		•	•		480		1600	7.3	4.2	
00027220	Gold crucible	6	40		•	•		380	0.25	750	6	1.65	**A**2
00026929	Medium pressure crucible	25	120	•		•		300	2	250	6	5.5	**A**3
00029990	Medium pressure crucible	25	120		•	•		300	2	250	6	5.5	**A**3
00026933	Seal for medium pressure crucible	30						—		230			
51140404	High pressure crucible	3	30		•	•		650	15	750	7	2.3	**B**
51140405	High pressure crucible gold plated	3	30		•	•		650	15	350	7	2.3	**B**
51140403	Seal for high pressure crucible	60						—					
00026732	High pressure crucible	25	40	•		•		1500	15	750	7	5.9	**D**
00026731	High pressure crucible	25	40		•	•		1500	15	750	7	5.9	**D**
00650072	High pressure crucible	1	270	•		•		2200	10	750	7.6	8.4	**C**
00650066	High pressure crucible	1	500	•		•		2600	10	750	7.6	14.4	**C**
00027216	Seal for high pressure crucible	1						—		750			
51140845	Sapphire crucible medium	4	70		•	•		220		2000	6	4.5	
51140843	Alumina crucible small	20	30		•	•		70		2000	6	2.6	
00024123	Alumina crucible medium	20	70		•	•		181		2000	6	4.5	
00024124	Alumina crucible large	20	150		•	•		380		2000	8	4.5	
51119960	Alumina crucible large	4	900		•	•		840		2000	12	10	
00027812	Glass crucible	50	100		•		•	400	5	500	6	13	

E: Only with large furnace (900 µl alumina crucible: only without the lid piercing kit)
F: Only with DSC20, 25, 27HP, 30; STARe DSC82x with furnace expander (without sample changer)

Material	for DSC	for TGA	suitable for sample robot	Turn table U1	Turn table U2	Page
Al 99.99%	•	•	•	•		4
Al 99.99%	•		•			4
Al 99.99%	•	•	•	•		4
Al 99.99%	•					4
Al 99.99%	•	•	•	•		4
Al 99.99%	F					4
Al 99.99%	•					4
Al 99.99%	•	•		•		4
Al 99.99%	•	•		•		4
E-Cu 99.90%	•	•	•	•		5
Pt Rh 20%	•	•	•	•		5
Pt Rh 20%	•	•	•	•		5
Pt Rh 20%	•	E	•	E		5
Au 99.99%	•	•	•	•		5
X5 CrNi 18 10	•					5
X5 CrNi 18 10	•		•			5
KEL-F						-
X2 CrNiMo18143	•		•			6
X2 CrNiMo18143, 5 µm gold plated	•		•			6
Cu. 2 µm gold plated						6
X2 CrNiMo18143, 5 µm gold plated	•					6
X2 CrNiMo18143, 5 µm gold plated	•		•			6
NiCr 20 TiAl	F					6
NiCr 20 TiAl	F					6
Au 700/531						6
Sapphire		•	•	•		7
Al_2O_3 99.7%		•	•	•		7
Al_2O_3 99.7%		•	•	•		7
Al_2O_3 99.7%		E	•	E		7
Al2O3 99.7%		E	•	E		7
Glass Duran®	F					7

Crucible handling set complete consisting of:	**51142765**
• Funnel	00026783
• Tweezers	51191865
• Needle standard	00029772
• Needle thin (0.35 mm)	51140833
• Crucible holder	51142312

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