Thermal Analysis Excellence

Crucibles

STAR^{e} System

Innovative Technology

Versatile Modularity

Swiss Quality

High Quality Crucibles

For Best Thermal Analysis Results

METTLER TOLEDO

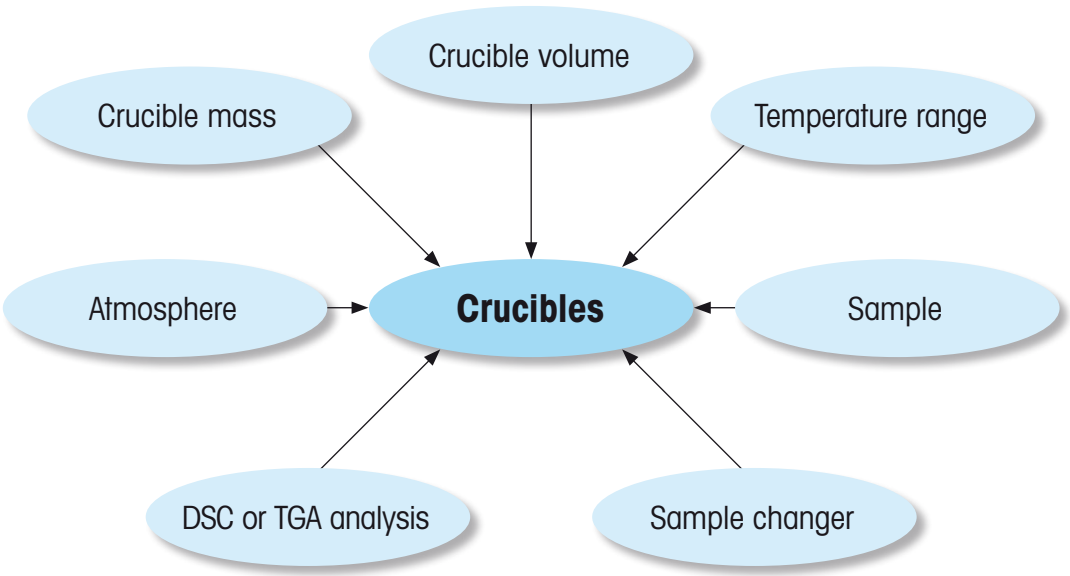
Crucibles for Thermal Analysis
The Optimal Type for Every Application

Crucibles serve as containers for samples during DSC and TGA measurements. They guarantee that the sensor is not contaminated during the measurement. The type of crucible used for a measurement can have a large effect on the quality of the results obtained, and also influences important characteristics of the measuring cell. Considering the relevant factors before the measurement can often help save time later on when interpreting the curve.

Features and benefits of the METTLER TOLEDO thermal analysis crucibles:

- **Large volume crucibles** – increase the sensitivity
- **Low mass crucibles** – push the resolution to the limit
- **Highest purity** – no reactions between sample and crucible caused by impure materials
- **Material** – guarantees high thermal conductivity and dimensional stability
- **Flat crucible base** – ensures perfect thermal contact and minimizes artifacts

For crucible selection, the following factors have to be considered:



Choosing the right crucible guarantees the best results for DSC, TGA and TGA/DSC measurements.

www.mt.com/ta-crucibles

Reliable Automation
24 Hours a Day, Just Like a Swiss Watch

The sample robot is extremely robust and operates reliably 24 hours a day, 7 days a week. All DSC and TGA 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. The universal gripper can handle all types of METTLER TOLEDO crucibles or even new crucible types that have been defined by the user.

No sample reaction before measurement

The sample robot can remove the protective aluminum lid from the alumina crucibles. It prevents loss or uptake of water of samples in alumina crucibles. It also protects oxygen-sensitive samples from oxidation.

Unique lid-piercing device

The lid piercing device (wasp) is a unique feature. Samples releasing volatiles or being hygroscopic can be enclosed in hermetically sealed crucibles to prevent mass changes prior to analysis. With the lid piercing kit hermetically sealed aluminum crucibles can be opened just before analysis in the DSC or TGA instrument.

Robot also works with 900 µL crucibles

Even 900 µL crucibles can be handled by the robot (special grippers with a longer swiveling arm – ArtNo: 30293250 – are necessary). These special grippers are limited to TGA/SDTA (LF/HT) sensors.

For Pt crucible handling, special grippers for Pt (ArtNo: 30479641) are available.

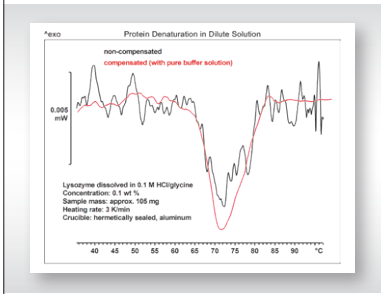
All our DSC and TGA models can be automated. The sample robot can handle any crucible in any sequence.

www.mt.com/ta-automation

Wide Range of Crucibles
Obtain the Optimal Measurement Results

Crucibles serve as containers for samples during DSC, TGA/DSC and TGA measurements. They prevent the direct contact of the test specimen with the sensor and thus prevent sensor contamination. Furthermore, intentional choice of the crucibles can contribute to optimum measurement results.

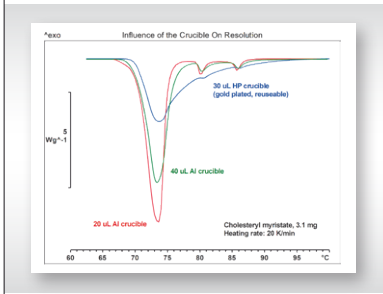
Increase crucible volume/sample mass for higher sensitivity



In the above example, the "weak peak" (black curve: about 10 times the noise level of the system) could only be detected because a 100 µL crucible was used instead of a standard 40 µL crucible.
If larger crucibles are used it is recommended to limit the heating rate to a maximum of 10 K/min as temperature gradients within the sample are expected.

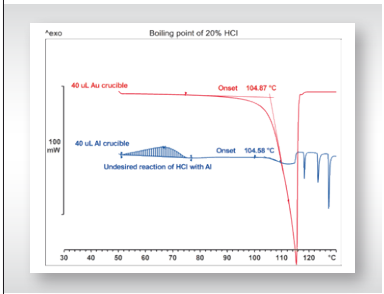
To improve the curve quality, the same experiment was repeated; this time with a reference 100 µL crucible that was filled with 105 mg buffer solution (red curve).

Improve resolution with low-mass crucibles



Especially in DSC, the crucible impacts the DSC signal response time and thus the temperature or time resolution. The lower the crucible mass, the better the resolution. The low-mass, 20 µL light aluminum crucible has the best signal time constant, i.e. peaks become sharper and close-lying peaks can be better separated.

Prevent undesired interaction between sample and crucible



Samples can react with the crucible. In most cases this is an unwanted effect. It can be prevented by choosing a suitable inert crucible. For typical reactive samples such as acid or alkaline materials, inert crucibles made of gold, platinum or glass are recommended. Also gold-plated crucibles from aluminum or steal are considered to be inert.

In some cases the interaction between the test specimen and the crucible is of interest (e.g. insulating material/copper).

METTLER TOLEDO offers a wide range of precision designed and manufactured crucibles with different volumes and materials. You can choose which is best suited for your application. Don't forget how important the sample preparation is.

www.mt.com/ta-sampleprep

Typical Crucibles for DSC
Appropriate for Your Application

Aluminum is the most commonly used crucible material for DSC measurements. The most frequently used crucible type for DSC (and TGA/DSC) measurements is the 40 µL aluminum (standard) crucible. It is very shallow (low in height) and has a strong flat base, which ensures that temperature gradients are as low as possible by having a well defined thermal contact with the sensor. A variant with pin is also available for the 40 µL volume, resulting in highest reproducibility (only for manual insertion). All aluminum crucibles can be combined with various lid types, depending on the application.

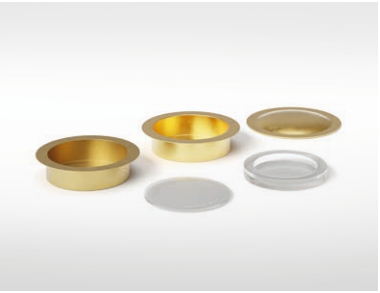
Aluminum crucibles with various volumes

Aluminum is the most frequently used crucible material for DSC measurements. METTLER TOLEDO offers aluminum crucibles of various volumes from 20 to 160 µL. The 20 µL light Al crucible has the shortest signal time constant. However, it cannot be sealed hermetically. The 25 µL Al crucible is optimized for enthalpy measurements with high reproducibility requirements. It can be sealed hermetically to suppress endothermic evaporation. Larger volume Al crucibles (100 and 160 µL) are used then large sample masses are needed.

Choice of 3 lid types

The standard lid is used either to hermetically seal the crucible (to suppress evaporation of solvents) or by piercing a hole in the lid to prevent test specimen from spilling over the crucible. In this case, the atmosphere is practically identical to the atmosphere in the furnace. Lids for robot piercing by the piercing device: The lid is pierced before the transfer to the furnace; 3 needle types – 0.1; 0.7; 1 mm diameter – are available. Lids with prepierced 50 µm-hole: For measurements in a self-generated atmosphere; evaporation of e.g. water is shifted to its boiling point.

Inert crucibles – gold-plated, pure gold or glass



Gold or gold-plated crucibles (40 µL) are chemically resistant and the optimal choice if strong acids or bases have to be analyzed. These pans are the only non-aluminum types that can be hermetically sealed by cold-welding; maximum pressure: 0.25 MPa (gold) and 0.2 MPa (gold-plated Al).

One type of glass crucible is offered (volume 15 µL). This crucible cannot be sealed, however, a glass cover disk, 7 mm in diameter, can be applied that closes the pan.

Alternatives to standard 40 µL Al crucibles

Alternative crucible types are described that improve sensitivity, resolution or inertness compared to standard 40 µL Al crucibles.

Technique	Crucible selection			
DSC, HP DSC, TGA/DSC, TGA	Standard crucible 40 µL aluminum → select the crucible that fits best:			
Operation mode \ Reason	**Improvement of sensitivity**	**Improvement of resolution and reproducibility**	**Inertness critical**	**Catalytic effect**
Robotic use	• 100 µL Al crucible For detection of weak signals.	• 20 µL Al crucible Maximal separation of close-lying effects (peak separation). Best for flat materials and powders, less suited for liquids.	• 40 µL Au-plated Al crucible Lid can be hermetically sealed, for reactive samples (e.g. strong acids and strong bases) at temperatures below 350 °C.	• 40 µL Cu pan Strong catalytic effects, often used for OIT determinations, catalytic effects are desired.
		• 25 µL Al crucible Lid can be hermetically sealed, maximal separation of close-lying effects (peak separation), for liquids.	• 40 µL Au crucible Lid can be hermetically sealed), for reactive samples (e.g. strong acids and strong bases) and high temperatures up to 750 °C.	
			• 30 µL or 70 µL Al_2O_3 For TGA/DSC measurements in the high temperature range up to 1600 °C.	
			• 70 µL sapphire crucibles For C_p measurements, for ash testing and wet chemistry applications.	
Manual use (DSC only)	• 160 µL Al crucible with pin Manual operation, crucible of choice if large quantities must be applied.	• 40 µL Al crucible with pin Maximal reproducibility.	• 15 µL glass pan For reactive samples (e.g. strong acids and strong bases).	

Selection of the aluminum lid types

Alternatives to standard lids are described.

Technique	Lid selection			
DSC, HP DSC, TGA/DSC, TGA	Select lid type to be combined with Al pans (25 µL, 40 µL, 100 µL, 160 µL)			
Material \ Lid type	**Standard lid**	**Lid with 50 µm hole**	**Piercing lid (sample protection)**	**No lid**
Aluminum	• Lid pierced with needle Proctects the sensor from material spilling over; atmosphere outside and inside identical.	• Lid with 50 µm hole and in comabination with standard 40 µL pans Self-generated atmosphere reduces gas exchange, shifts vaporization to the boiling point.	• Piercing lids in combination with standard 40 µL pans Makes it possible to limit gas exchange to the start of the experiment; no drying or water uptake of the sample during the waiting period.	• No lid Optimal gas exchange, typically applied for OIT and OOT measurements (particularly for HP DSC).
	• Lid hermetically sealed			
Aluminum, gold-plated lids, gold lids	• Hermetically sealed lid Al, Au, gold-plated; no hole, typically for liquids.			

Typical Crucibles for TGA
Accurate TGA Measurements

The most frequently used crucible type for TGA (and TGA/DSC) measurements is the 70 µL alumina (standard) crucible. All alumina crucibles are provided with aluminum oxide lids. Aluminum oxide crucibles can be reused; appropriate cleaning procedures have to be defined (depending on the degree and type of contamination).

Alumina crucibles – from 30 to 900 µL

The most often used crucibles for TGA measurements are the aluminum oxide (or alumina) crucibles. They allow measurements up to 1600 °C. In addition, aluminum oxide is highly chemically inert – side reactions are very seldom. The volume range of our offering from 30 to 900 µL is enormous.

Aluminum lids for alumina crucibles

All alumina crucibles are provided with alumina lids. These lids have a hole. For applications with samples that contain volatile substances, drying out or moisture uptake is critical. For such cases, aluminum lids are available that can be removed by the sample robot before the crucible is transferred to the furnace.

Sapphire crucibles

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

Alternatives to standard 70 µL Al_2O_3 (alumina) crucibles

Alternative crucible types that improve sensitivity, resolution or inertness are described compared to standard 70 µL Al_2O_3 crucibles.

Technique	Crucible selection		
TGA, TGA/DSC, DSC	Standard crucible 70 µL alumina → select the crucible that fits best:		
Furnace type \ **Reason**	**Improvement of sensitivity**	**Improvement of resolution and reproducibility**	**Inertness-critical**
Small TGA or TGA/DSC furnace		• 40 µL (25 µL) Al crucibles Al crucibles for best sensitivity also for TGA up to 640 °C.	• 40 µL Au crucibles Up to 750 °C, Au crucibles if resolution, sensitivity and inertness are critical. • 40 µL Au-plated Al crucibles Up to 350 °C.
		• 30 µL Al_2O_3 crucibles Close-lying effects, peak separation. • 30 µL Pt crucibles High temperature range up to 1600 °C.	• 70 µL sapphire crucible C_p measurements; measurements of alloys, temperatures up to 1100 °C.
TGA: Large or HT furnace	• 100 µL Al crucibles Al Crucibles for best sensitivity also for TGA up to 640 °C. • 150 µL, 300 µL, 600 µL Al_2O_3 crucibles Weak signal, increase of sample mass required. • 150 µL Pt crucibles Lid not sealed, temperature range > 600 °C, weak signal, reproducibility critical.	• 30 µL Al_2O_3 crucibles Close-lying effects, peak separation. • 30 µL Pt crucibles High temperature range up to 1600 °C.	• 70 µL sapphire crucibles C_p measurements; measurements of alloys, temperatures up to 1600 °C.
Large furnace / special grippers	• 900 µL Al_2O_3 crucibles Special grippers for robot, weak signal, large mass/volume needed (EGA measurements).		
Sapphire disks (TGA)		Sapphire disks are always necessary if Pt crucibles are in contact with Pt sensors (DTA).	

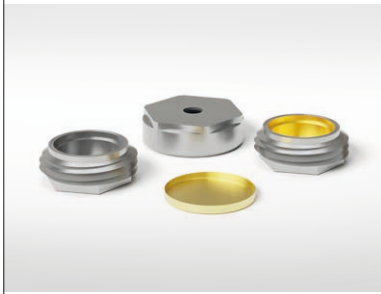
Typical applications intended for Pt crucibles:

- TGA/DSC experiments with a focus on DSC signals at higher temperatures.
- Heat capacity measurements at temperatures > 600 °C.
- Metal characterization measurements (mainly below the melting temperature of the metal sample).
- Humidity measurements with salts.

High-Pressure Crucibles
For the Investigation of Chemical Reactions

METTLER TOLEDO offers a variety of high-presssure crucibles that serve as sample containers for thermoanalytical measurements. These hermetically sealed HP crucibles withstand pressures up to 15 MPa.

High-pressure (HP) crucible – for reusage



The relatively light and flat construction of the HP steel crucible results in low temperature gradients. Compared to larger HP crucibles, the obtained DSC results are better. The thread and the sealing tool with defined torque enable the crucible to be easily and securely sealed. After the measurement, the crucible can be cleaned for reusage (about 20 times, using a new gold-plated copper seal). NiCr (NiCr20TiAl) HP Crucibles: For manual usage and large volumes, two types of NiCr crucibles (270 und 500 µL) are available.

High-pressure crucibles, stainless steel, single-use

These HP gold-plated steel crucibles (25 µL and 40 µL volume) have proven highly effective in the field of safety investigations. These are the optimal single-use crucibles for a pressure up to 15 MPa. The crucible lid is pressed (pressure of about a ton) into the crucible with the aid of a special toggle press so that the rupture disk (gold) hermetically seals the crucible.

Medium-presssure crucibles, single-use

The medium pressure crucible allows a maximum pressure to be generated of about 2 MPa. To use the standard sealing press, the die set (type A3) must be used.

The medium pressure crucibles can be sealed with either a FPM O-ring (that allows water-vapor to permeate) or with a PCTFE O-ring. However, this polymer shows a DSC melting peak at 220 °C.

Selection of high-pressure crucibles

The high-pressure portfolio is shown.

Technique	Crucible selection		
DSC, (HP DSC)	High pressure crucibles		
Lid type / Material	**Reusable HP crucibles for moderate reactive samples**	**Single-use HP crucibles**	**Reusable, inert HP crucibles**
Steel, max. pressure 15 MPa	• 30 µL crucible with burst disc - Suppression of boiling, for highly reactive samples. - Reusable, up to 20 times.		
Gold-plated steel, max. pressure 15 MPa		• 25 µL, 40 µL crucibles with lid and burst disc - Safety measurements. - Not reusable.	• 30 µL crucible with lid [1] - Suppression of boiling, inert material necessary, hightly reactive material. - Reusable, up to 20 times.
NiCr [2] alloy, max. pressure 10 MPa			• 270 µL, 500 µL crucible with lid [3] and seal - Reusable, no limitation.

[1] Seals necessary, not included.
[2] NiCr: alloy of Ni, Cr, Ti, Al.
[3] Furnace expander required; after each measurement, a new seal disk is used.

Crucible material alternatives to aluminum (ambient pressure)

Alternative crucible materials and their advantages compared to the predominantly used aluminum are shown.

Technique	Crucible selection			
DSC, HP DSC, TGA/DSC, TGA	Select crucible material other than aluminum			
Material / Reason	**Platinum (Pt80Rh20)**	**Al_2O_3 (alumina)**	**Sapphire**	**Gold**
Reactivity / chemical resistance	• 30 µL, 70 µL, 150 µL Catalytic effects possible (often undesired). Normally used at temperatures up to 1600 °C.	• 30 µL, 70 µL, 150 µL, 300 µL, 600 µL, 900 µL Inert material, easy to handle, Alumina is critical in combination with some metals such as iron. Not optimal for hightest resolution and reproducibility.	• 70 µL Optimal for C_p measurements and measurements at very high temperatures, sapphire is chemically extremely resistant. Sapphire is the optimal choice for many metals at higher temperatures.	• 40 µL Inert material, often used in combination with acid or alkaline samples.
Degree of reactivity	Low and undesired. Pt is the optimal choice for substances that react with Al_2O_3 or sapphire.	Low reactivity (critical with certain metals such as iron).	None (highest degree of inertness).	Low reactivity

Platinum crucibles: Platinum crucibles are mainly used for TGA/DSC or TGA measurements with end 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. In addition, Pt crucibles are often used for accurate C_p determinations (DSC and TGA instruments).

Innovative Accessories for Crucibles
Sealing Press Types and Accessory Box

The standard sealing press allows the pan to be sealed very easily. Under the pressure of the plunger, the pan is cold-welded hermetically with the lid. There is a variety of plungers and dies for various crucible types.

Sealing of many crucible types

A1: Die and plunger set for the aluminum light 20 µL crucibles. Hermetical sealing is not possible with this crucible type.

A2: Die and plunger for all crucibles that can be sealed by cold-welding: 40, 100 and 160 µL aluminum crucibles; 40 µL gold crucibles. All these crucibles can be hermetically sealed.

A1a: Die and plunger for the 40 µL gold-plated aluminum crucibles; they can also be hermetically sealed.

Crucible handling set

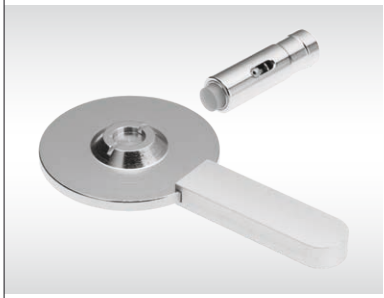
The crucible handling set provides a range of tools that are helpful for sample preparation:

- A funnel for filling the pan with sample
- Tweezers for handling of sample, pans and lids
- Standard needle (1 mm) and a thin needle (0.35 mm thick)
- Crucible holder for crucible handling and safe transfer to the instrument.

Sealing of high-pressure crucibles

High-pressure crucibles (25 µL and 40 µL) can be sealed by using a special sealing press (Maeder press). A particular plunger/die set is included for these two types of crucibles.

Die and plunger

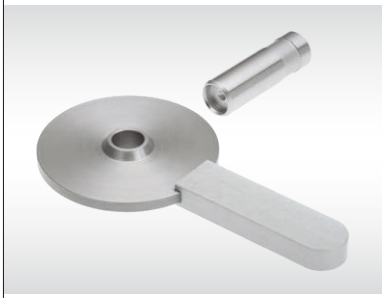


A1: Die and plunger for Al crucible light.
ArtNo: 51140547

A1a: Die and plunger for gold-plated aluminum crucibles.
ArtNo: 51142272

20 µL 40 µL

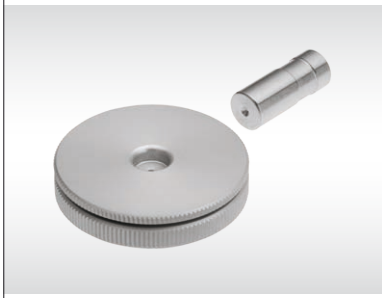
Die and plunger



A2: Die and plunger; sealable by cold welding (for hermetical sealing; included in delivery).
ArtNo: 27809 / 27386

25 µL 40 µL 100 µL 160 µL

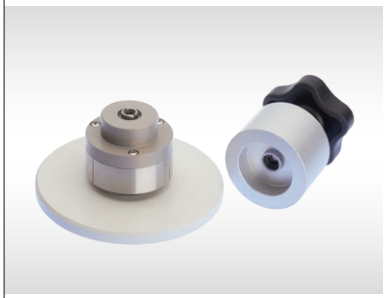
Die and plunger



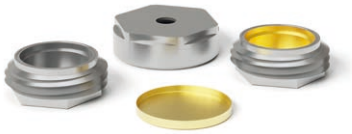
A3: Die and plunger for medium pressure crucible.
ArtNo: 119428

120 µL

High pressure crucible sealing tool for reusable crucibles



B: This assembly consists of a lower part to keep the hexagonal crucible and an upper part (turning head) that fits on the crucible cover. The crucible is closed with the seal disk by turning until a sliding clutch is activated. After the measurement, the crucible is opened with the same tool.
ArtNo: 51119915



30 µL

High pressure crucible sealing tool for reusable NiCr crucibles

C: This is the tool for the NiCr crucibles. They are fixed in one of the tolls and the cover in the other to seal them with the required torque.
ArtNo: 650067

500 µL 270 µL

Tool kit for toggle press

D: This assembly fits in the Maeder press used to seal the 25 and 40 µL high pressure crucibles.
ArtNo: 26733

25 µL 40 µL

Technical Data

Article no.	Designation	Quantity	Volume [µL]	With pin	Without pin	With lid	Without lid	Pan weight [mg]	Lid weight [mg]	Max. pressure [MPa]	Max. temperature [°C]	∅ Base [mm]	Height [mm] without lid
26763	**Aluminum crucible (standard)**	**100**	**40**		•	•		**33**	**17**	**0.2**	**640**	**6**	**1.6**
51119810	Aluminum crucible light	100	20		•	•		14	9	0.2	640	6	1.6
30085850	Aluminum crucible	100	25		•	•		28	17	0.2	640	4	1.6
27331	Aluminum crucible standard	100	40	•		•		33	17	0.2	640	6	1.6
51119870	Aluminum crucible standard	400	40		•		•	33	–	0.2	640	6	1.6
51141448	Aluminum crucible / light lid	100	40	•		•		33	9	0.2	640	6	1.6
51143092	Aluminum crucible large	100	100		•		•	63	–	0.2	640	6	4.2
51119872	Aluminum crucible large	400	100		•		•	63	–	0.2	640	6	4.2
27811	Aluminum crucible large	40	160	•		•		80	17	0.2	640	6	6.4
51119871	Aluminum lid standard	400	lid					–	17		640		
51140832	Aluminum lid pierced 50 µm	400	lid					–	17		640		
51119873	Aluminum piercing lid	400	lid					–	16		640		
51142973	Aluminum crucible gold plated	10	40		•	•		70	40	0.2	350	6	1.6
27220	Gold crucible	6	40		•	•		270	130	0.25	750	6	1.65
51140407	Copper crucible	100	40		•		•	75	–		750	6	1.65
17780	Glass crucible	5	15					55			500	7	1
17782	Glass cover disk (7 mm)	5	disk						15			7	0.2
24123	**Alumina crucible (standard)**	**20**	**70**		•	•		**185**	**60**		**2000**	**6**	**4.5**
51140843	Alumina crucible small	20	30		•	•		95	60		2000	6	2.6
24124	Alumina crucible large	20	150		•	•		280	110		2000	8	4.5
30267108	Alumina crucible large	10	300		•	•		520	100		2000	8	9
30077260	Alumina crucible large	4	600		•	•		880	230		2000	12	9.2
51119960	Alumina crucible large	4	900		•	•		770	270		2000	12	10
51119649	Aluminum lid for Al_2O_3 30/70	40	lid			•			50				
51140477	Aluminum lid for Al_2O_3 150/300	40	lid			•			75				
30077266	Aluminum lid for Al_2O_3 600	40	lid			•			205				
51140469	Aluminum lid for Al_2O_3 900	40	lid			•			185				
51140842	Platinum crucible small	4	30		•	•		180	85		1600	6	2.3
51119654	Platinum crucible medium	4	70		•	•		275	85		1600	6	4.2
24126	Platinum crucible large	4	150		•	•		420	140		1600	7.3	4.2
51140845	Sapphire crucible	4	70		•	•		200	60		2000	6	4.5
51140404	High pressure crucible	3	30		•	•		300	270	15	750	7	2.5
51140405	High pressure crucible gold plated	3	30		•	•		325	270	15	350	7	2.5
51140403	Seal for high pressure crucible	60	seal					–	–				
30077139	High pressure crucible small	25	25		•	•		620	280	15	400	6.4	4.7
26732	High pressure crucible / pin	25	40	•		•		1150	280	15	400	7	5.9
26731	High pressure crucible	25	40		•	•		1150	280	15	400	7	5.9
650072	High pressure crucible	1	270	•		•		1060	1070	10	750*	7.6	8.4
650066	High pressure crucible	1	500	•		•		–	–	10	750*	7.6	14.4
27216	Seal for high pressure crucible	1	seal					–	–		750*		
26929	Medium pressure crucible	25	120	•		•		170	140	2	250	6	5.5
29990	Medium pressure crucible	25	120		•	•		170	140	2	250	6	5.5
26933	Seal for medium pressure crucible	30	seal					–	–		230		

* Above 500 °C and in the presence of oxygen, scaling of the material may occur.

Article no.	Designation	Material	Sealing tool	For DSC	For TGA	Suitable for sample robot	Turn table U1	Turn table U2	Turn table U3
26763	**Aluminum crucible (standard)**	**Al 99.99%**	**A2**	•	•	•		•	•
51119810	Aluminum crucible light	Al 99.99%	**A**1	•	•	•	•	•	•
30085850	Aluminum crucible	Al 99.99%	**A**2	•	•	•	•	•	•
27331	Aluminum crucible standard	Al 99.99%	**A**2	•					
51119870	Aluminum crucible standard	Al 99.99%	**A**2	•	•	•	•	•	•
51141448	Aluminum crucible / light lid	Al 99.99%	**A**2/**A**1	•					
51143092	Aluminum crucible large	Al 99.99%	**A**2	•	•	•	•	•	•
51119872	Aluminum crucible large	Al 99.99%	**A**2	•	•	•	•	•	•
27811	Aluminum crucible large	Al 99.99%	**A**2	H					
51119871	Aluminum lid standard	Al 99.99%		•	•	•	•		•
51140832	Aluminum lid pierced 50 µm	Al 99.99%		•	•	•	•		•
51119873	Aluminum piercing lid	Al 99.99%		•	•	•	•		•
51142973	Aluminum crucible gold plated	Al 99.5%, 5 µm gold plated	**A**1a	•	•	•	•	•	•
27220	Gold crucible	Au 99.99%	**A**2	•	•	•	•	•	•
51140407	Copper crucible	E-Cu 99.90%		•	•	•	•	•	•
17780	Glass crucible	Borosilicate glass		•	•				
17782	Glass cover disk (7 mm)	Borosilicate glass		•	•				
24123	**Alumina crucible (standard)**	**Al_2O_3 99.7%**		•	•	•	•	•	•
51140843	Alumina crucible small	Al_2O_3 99.7%		•	•	•	•	•	•
24124	Alumina crucible large	Al_2O_3 99.7%			E	•	•		•
30267108	Alumina crucible large	Al_2O_3 99.7%			E	•	•	•	•
30077260	Alumina crucible large	Al_2O_3 99.5%			E	•	•	•	•
51119960	Alumina crucible large	Al_2O_3 99.8%			E	G			
51119649	Aluminum lid for Al_2O_3 30/70	Al 99.5%		•	•				
51140477	Aluminum lid for Al_2O_3 150/300	Al 99.5%			E				
30077266	Aluminum lid for Al_2O_3 600	Al 99.5%			E				
51140469	Aluminum lid for Al_2O_3 900	Al 99.5%			E				
51140842	Platinum crucible small	Pt Rh 20%		•	•	•	•	•	•
51119654	Platinum crucible medium	Pt Rh 20%		•	•	•	•	•	•
24126	Platinum crucible large	Pt Rh 20%		•	E	•	•		•
51140845	Sapphire crucible	α-Al_2O_3 99.9%		•	•	•	•	•	•
51140404	High pressure crucible	X2 CrNiMo18143	**B**	•		•			•
51140405	High pressure crucible gold plated	X2 CrNiMo18143, 5 µm gold plated	**B**	•		•			•
51140403	Seal for high pressure crucible	Cu, 2 µm gold plated							
30077139	High pressure crucible small	X2 CrNiMo18143, 5 µm gold plated	**D**	•		•			
26732	High pressure crucible/pin	X2 CrNiMo18143, 5 µm gold plated	**D**	•					•
26731	High pressure crucible	X2 CrNiMo18143, 5 µm gold plated	**D**	•		•			•
650072	High pressure crucible	NiCr 20 TiAl	**C**	F					
650066	High pressure crucible	NiCr 20 TiAl	**C**	F					
27216	Seal for high pressure crucible	Au 700/531							
26929	Medium pressure crucible	X5 CrNi 18 9	**A**3	•					
29990	Medium pressure crucible	X5 CrNi 18 9	**A**3	•		•		•	•
26933	Seal for medium pressure crucible	PCTFE							

E: Only with large furnace (900/600 µL alumina crucible: only without the lid piercing kit)
F: Only with DSC20, 25, 27HP, 30; S**TA**R® DSC82x and S**TA**R® System DSC 1/2/3 with furnace expander (without sample changer)
G: Only with the special gripper (30293250) for sample robot
H: The crucible can be used without furnace expander if the lid is reversed

Extremely Wide Application Range
Crucibles for Every Sample Type

Good thermal analysis measurements also depend also on the correct choice of crucibles and lids. Lids seal the crucible hermetically to avoid evaporation of the sample and to avoid interferences with the surrounding atmosphere. In contrast, open crucibles without a lid (or with a lid with a big hole) allow the ambient atmosphere to come into contact with the sample.

Sealing possibilities

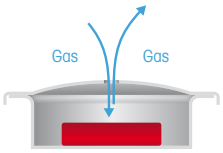
Open crucible, no lid

Optimal access for gas molecules

Removal of gaseous reaction products

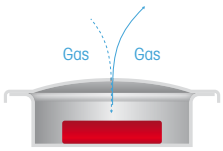
Lid pierced, large holes

Sealed lid, lid pierced, hole diameter 0.4 to 1 mm → atmosphere in the inner part almost corresponds to the outer atmosphere. Sensor is protected from substance creeping out. Evaporation is only slightly slowed down.



Lid pierced, 50 µm hole

Crucible sealed with a pierced lid (50 µm hole). Inside forms a self-generated atmosphere, evaporation is shifted to the boiling point.



Hermetically sealed

Lid hermetically sealed: Sample is measured without gas exchange during the heating or cooling. Typical case for samples that contain volatile substances.

Perfect gas exchange ⟶ no gas exchange

Evaporation: normal ⟶ slightly slowed down ⟶ slowed down ⟶ suppressed

Crucibles are fundamental for the studies of materials such as thermoplastics, thermosets, elastomers, composites, adhesives, foodstuffs, pharmaceuticals and chemicals.

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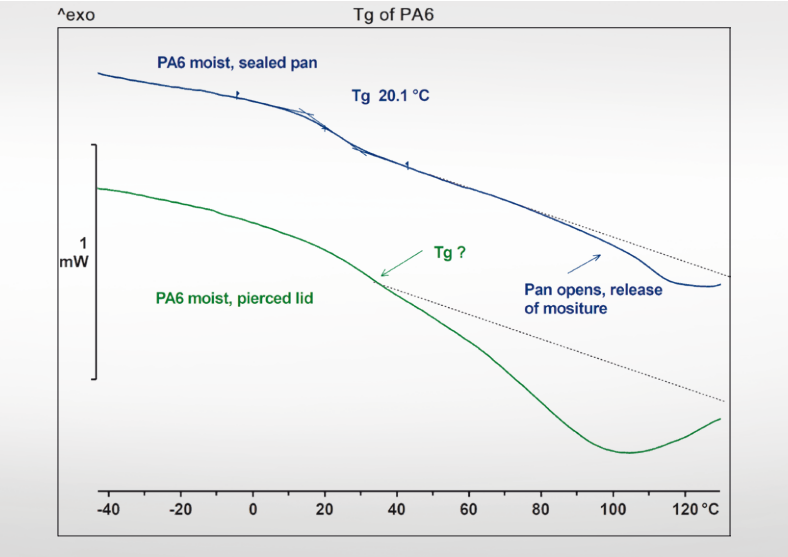
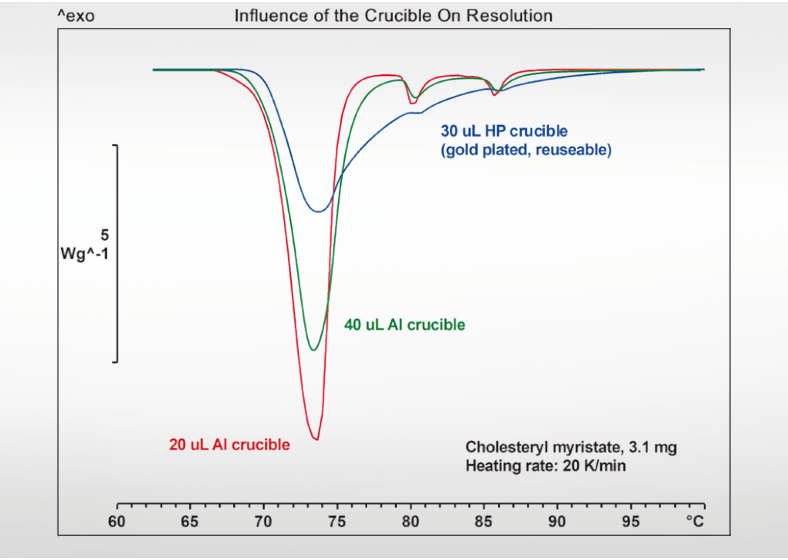
Influence of crucible sealing



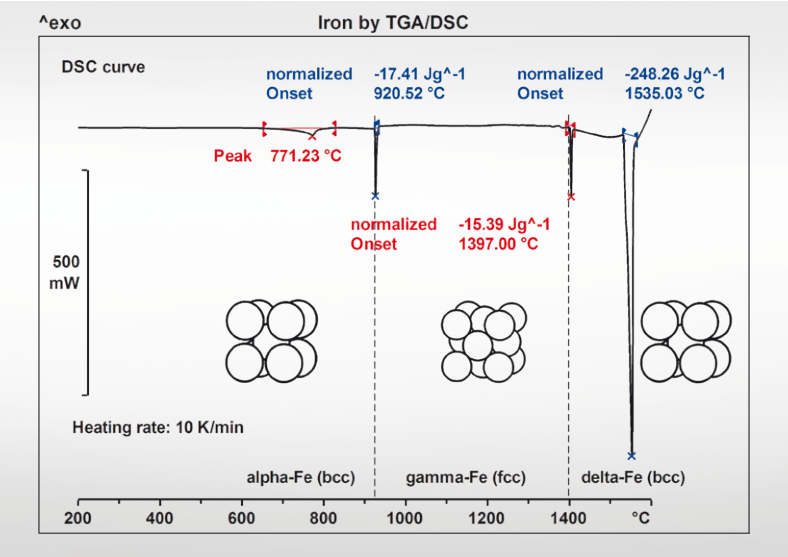
This example describes the influence of crucible sealing on the resulting measurement curves obtained from a DSC experiment. Two polyamide samples were measured using standard 40 µL aluminum crucibles; one was left open (see the green curve), and the other was hermetically sealed (see the blue curve). At room temperature, the moisture in the sample begins to evaporate in the open crucible; this results in a broad endothermic peak on the green DSC curve and the glass transition at about 30 °C is hardly detectable. In contrast, evaporation is suppressed in the hermetically sealed crucible and the glass transition can be easily identified.

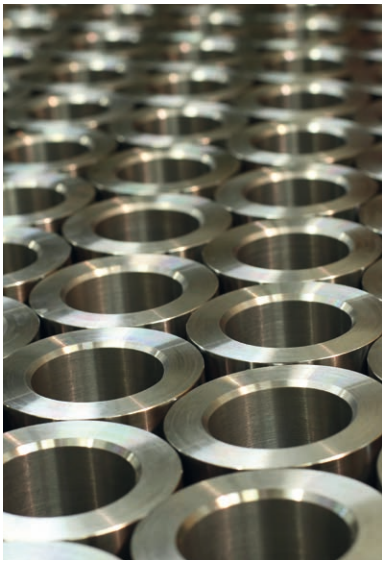
Cholesteryl myristate measured with various crucibles



This example shows the DSC curves of cholesterol myristate – a liquid crystal that undergoes two liquid-liquid transitions after melting. The only difference between the measurements was the type of crucible used. The blue curve measured using a 30 µL gold plated high-pressure crucible shows a broad melting peak at about 74 °C, followed by two weak effects corresponding to a liquid-liquid transition at about 81 and 86 °C, respectively. These latter two effects exhibit sharper peaks when the sample is measured in a 20 or 40 µL aluminum crucible.

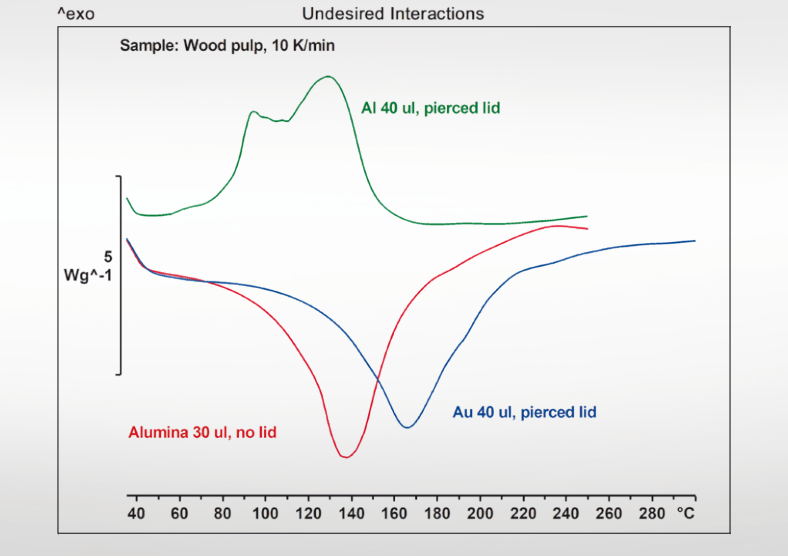
Sapphire crucibles used to study iron





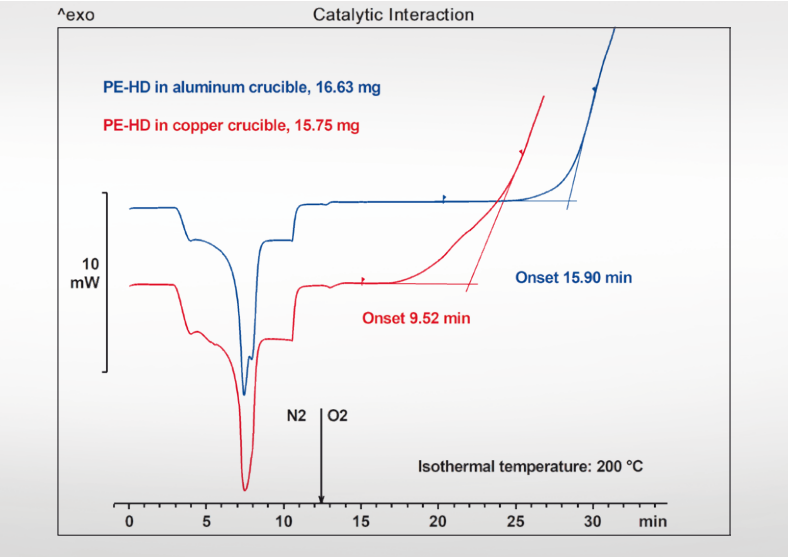
In this example, the phase transitions of iron are illustrated. The measurements were performed in argon using a 70 µL sapphire crucible. These crucibles are of Al_2O_3 material that has been crystallized at very high temperature and is resistant towards molton iron (in contrast to conventional alumina crucibles). The examples shows 3 modifications of iron (α-modification prevalent below 920 °C; γ-modification formed at about 920 °C and a δ-modification formed at about 1397 °C). The iron melts at about 1540 °C.

Inert crucibles suppress undesired interactions



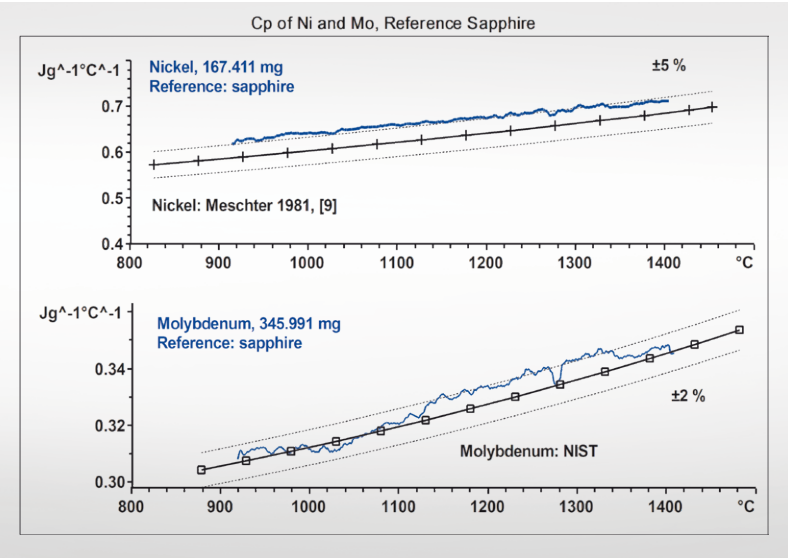
For most applications, a reaction between the crucible material and the sample is undesired. Aluminum crucibles are suitable for most samples but may react with certain acids or alkaline materials. In this example, a wood pulp specimen containing sodium hydroxide and sodium sulfide reacted with the aluminum crucible to produce a large exothermic peak. This is clearly illustrated by the green curve. In contrast, when the same sample is measured in an inert crucible such as alumina or gold, the desired evaporation peak of the water is clearly observed as an endothermic peak; see red and blue curves.

Copper crucibles enhance catalytic effect



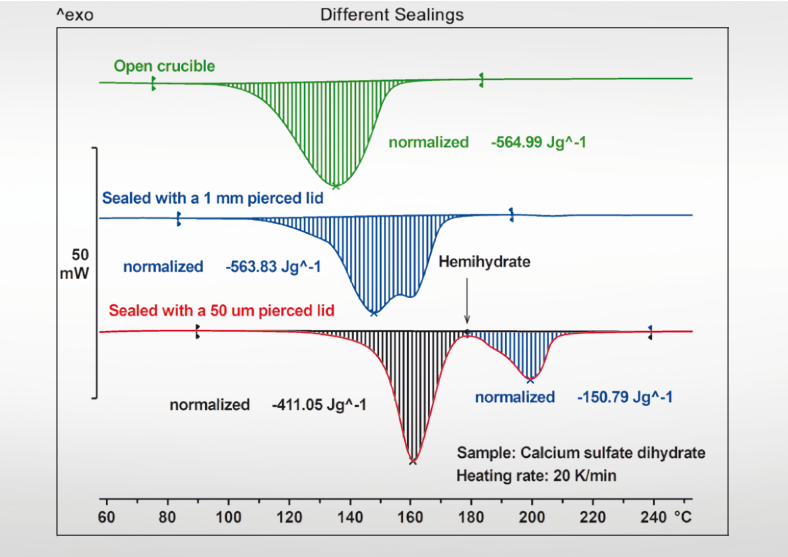
OIT experiments – short for oxidative induction time – provide information about the oxidative stability of a sample. Here, a high-density PE sample was heated to 200 °C in an inert atmosphere. After 12.5 min at 200 °C, the gas is switched from nitrogen to oxygen. The time from the gas switching to the onset of the oxidation reaction is defined as OIT. The results show a big difference in the stability of the sample with respect to the pan material. The sample with the copper pan has a significantly lower OIT than the sample with the aluminum pan as copper increases the reaction time. Such experiments are important for the cable industry (ASTM D3895).

Platinum crucibles for c_p measurements



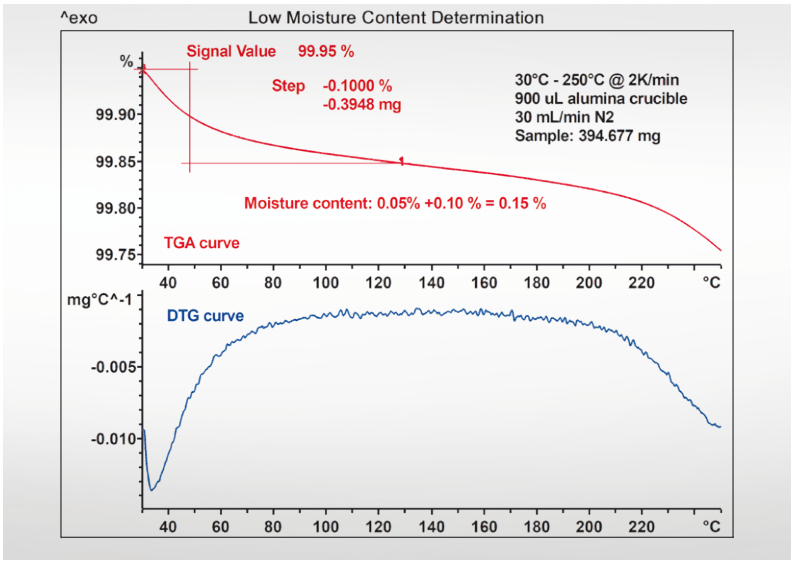
Using a TGA/DSC, this example illustrates the determination of the specific heat capacities (C_p) of two metals (molybdenum and nickel; blue line) and the comparison with the literature value (black line with symbol). For C_p measurements, crucibles of platinum are recommended as they have an excellent thermal conductivity. In general, the crucible should be used with lid and its size should be as small as possible. As Pt can stick to Pt sensors at temperatures around 1100 °C and above, we recommend placing sapphire disks between the crucibles and sensor.

Gypsum measured with different pan-lid combinations



The type of sealing also influences a DSC measurement. This can be illustrated using calcium sulfate dehydrate, commonly referred to as gypsum. When gypsum is heated, dehydration occurs in two steps to form hemihydrate followed by anhydrite. These dehydration steps were studied in an open crucible and two sealed crucibles, one pierced with a 1 mm hole and the other with a 50 µm hole. As shown here, different types of sealings have a significant influence on the results. The sample measured in an open crucible (top curve) exhibits one broad endothermic peak due to dehydration. When the same sample is measured in sealed crucibles with pierced lids, the two dehydration steps become visible (see middle and bottom curves). The different phases are best separated when a very small hole is pieced in the lid – in this case 50 µm.

Moisture determination in a polymer membrane

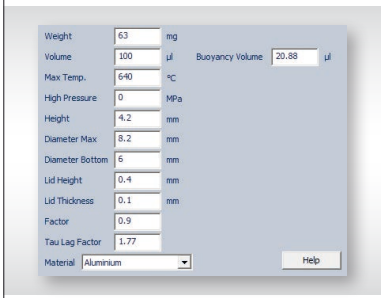


When a low moisture polymer membrane sample is heated in a TGA, a large amount of sample is needed to accurately determine its moisture content. Therefore, about 400 mg of sample was measured in a 900 µL crucible. Between sample preparation and the actual measurement, the sample loses about 0.05% of its initial mass due to evaporation. Upon heating to 130 °C, an additional 0.1% is lost. This corresponds to about 400 µg of sample. In contrast, a 70 µL crucible could only support an initial sample mass of about 30 mg, and the overall mass loss step would correspond to 45 µg. This results in a measurement uncertainty of about 10% in contrast to only 0.3% when using a 900 µL crucible filled with 400 mg of sample.

Simple, Intuitive Operation
Straightforward, Efficient and Secure

STARe is the most comprehensive thermal analysis software on the market and provides unrivalled flexibility and unlimited evaluation possibilities. The flexible STARe software consists of the base software and a large number of application-specific options to satisfy your current and future requirements.

Crucible definition in Install Window

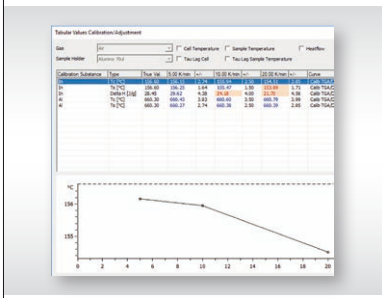


Crucible types are defined in the Install Window and belong to every method. The STARe software allows users to define their own crucible types with new dimensions.

FlexCal™: Automatically get the correct adjustment parameters

a) FlexCal database for the highest accuracy: STARe software stores the complete adjustment data in the database for every crucible, gas and module combination.
b) FlexCal factors for fast results: For the case that there is no specific adjustment data available, the system takes a base adjustment dataset and calculates a dataset for another crucible, gas and module combination.

TabVal calibration and adjustment



Calibration and adjustment has been revised and optimized so that the possibilities offered by FlexCal are more advantageous.

For each calibration substance, there is now a result value with details of the tolerance for the particular heating rate. The evaluation of the adjustment or calibration measurement is fully automatic.

A complete thermal analysis system consists of the basic six complementary measuring techniques, each of which brings fast and accurate results. Additional knowledge can be obtained by means of several hyphenated techniques.

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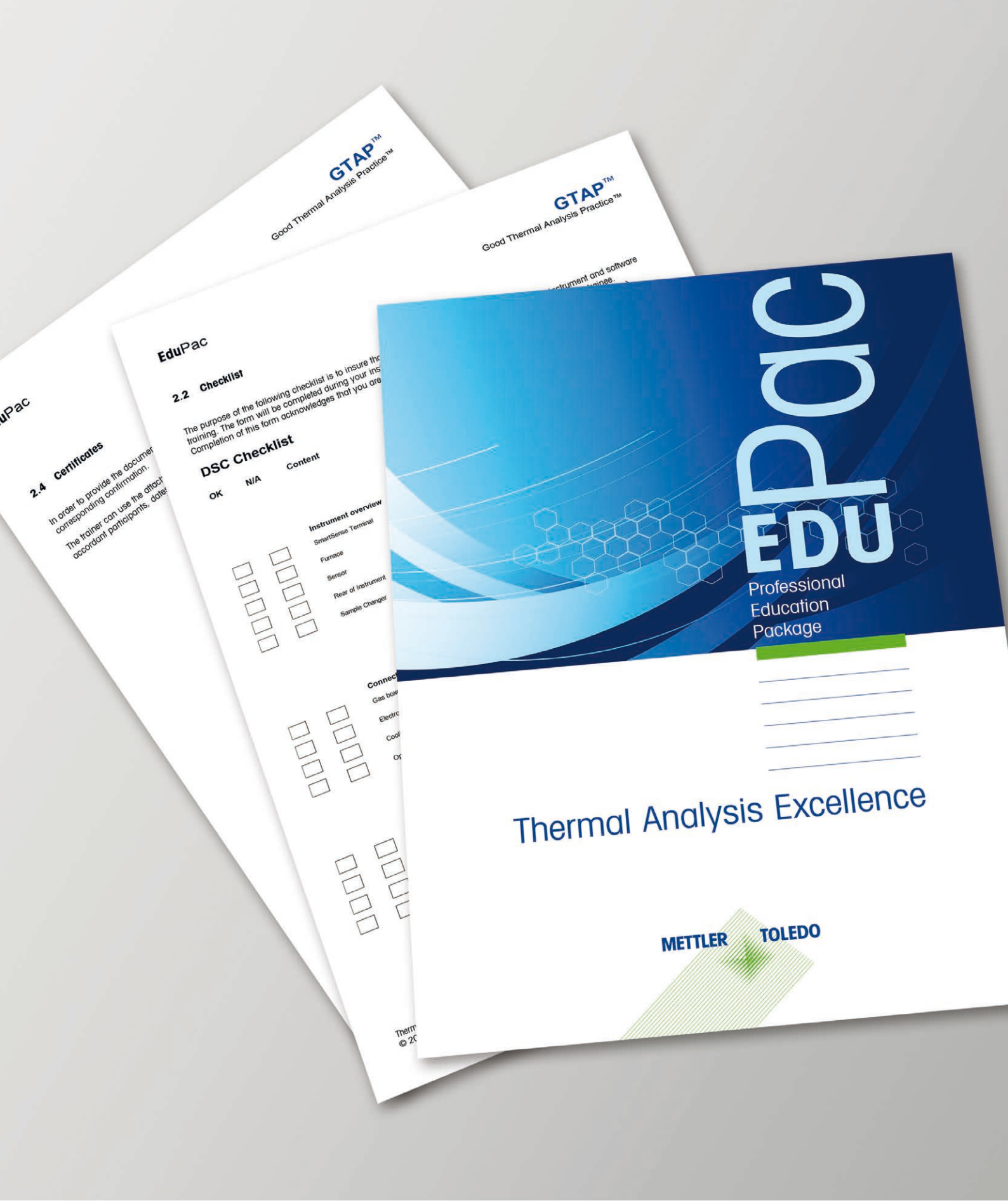

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