

Accessories for Differential Scanning Calorimeters and Thermobalances

Crucibles, Sensors, Sample Carriers, Calibration Kits for DSC, TGA and STA Systems



Introduction – Table of Contents

Accessories for Thermal Analysis – DSC/DTA, TGA and STA

At NETZSCH, we always strive to be a step ahead. We place as much importance on the development and production of accessories as on that of new instruments. Accessories in contact with the sample or in close proximity to it require special attention. Potential reactions between the sample material and instrument parts must be prevented while ensuring that the test results remain reliable and accurate. For these reasons, one of our primary areas of focus is crucibles and sensors for DTA/DSC, TGA and STA instruments.

This catalogue provides an overview of all such crucibles and sensors for DTA, DSC, TGA and STA measurements. You will find many different crucible materials listed, and a variety of types

and special shapes. From among these, we can help you find the right crucible size and material for any application, be it standard or special. In addition to standard aluminum crucibles, our portfolio includes autoclaves with low to medium or high pressure-tightness, as well as ones for determining the oxidative-induction time (OIT) and solid fat index (SFI). We offer crucibles made of ceramics or metals covering a wide temperature range. In the high-temperature range, special TGA and DTA crucibles, slip-on plates, meshes and baskets are available to accommodate specific sample dimensions and densities.

Lately, the demand for special crucibles has been increasing. Of course,

measurements can only be carried out when the right sensor or sample carrier for these special crucibles is available. We have therefore listed these special cases here, often providing application examples to demonstrate their characteristic advantages.

Our accessories can open up a world of possibilities for your thermoanalytical needs. It is our hope that this catalogue will serve to acquaint you with these. If you have any questions, or if you require something specific which you cannot find in the following tables, please simply contact us – NETZSCH welcomes the challenge of working out a solution tailored to your application, and we are always happy to hear from you.

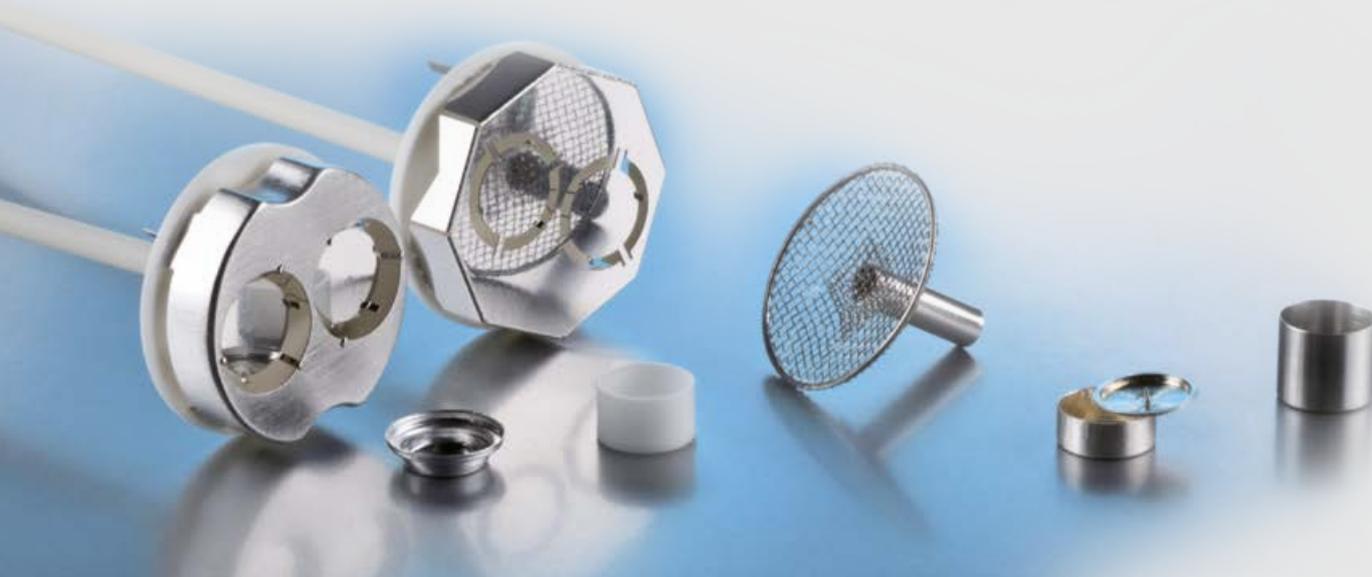


Table of Contents

| | | | |
|---|----|--|----|
| Crucibles | | Sensors and Sample Carriers | 40 |
| Selection of Crucibles..... | 4 | DSC 204 F1 Phoenix [®] Sensors, TG 209 F1 Iris [®] / Libra [®] and TG 209 F3 Tarsus [®] Sample Carriers..... | 42 |
| DSC 204 F1 Phoenix [®] /DSC 200 F3 Maia [®] /DSC 3500 Sirius [®] /DSC 214 Polyma | 6 | c-DTA [®] – More Than an Easy Calibration Routine | 44 |
| Standard Crucibles made of Aluminum | 7 | DTA and DSC Sensors for the DSC 404 F1/F3 Pegasus [®] | 46 |
| Crucibles for General and Special Applications | 8 | TGA Sample Carriers and TGA-DTA Sensors for the STA 449 F1/F3 Jupiter [®] | 48 |
| Sealing Press, Sample Preparation and Pressure-Tight Crucibles | 10 | TGA-DTA and TGA-DSC Sensors for the STA 449 F1/F3 Jupiter [®] | 50 |
| Crucibles for Automatic Sample Changer (ASC) | 12 | Radiation Shield for STA Systems..... | 52 |
| Special Crucibles for Special Applications | 16 | TGA and TGA-DSC Sensors for C _p Determination and Operation with the Automatic Sample Changer (ASC) for the STA 449 F1/F3 Jupiter [®] | 54 |
| TG 209 F1 Libra [®] /TG 209 F3 Tarsus [®] | 18 | Different Sensor Types – The Right Sensor for Each Application | 56 |
| Crucibles for General Applications | 19 | Tungsten for Highest Temperature Applications..... | 58 |
| Crucibles for Automatic Sample Changer (ASC) | 20 | Special Sample Carriers for Special Applications – OTS [®] | 60 |
| Applications..... | 23 | Hanging Samples..... | 62 |
| STA 2500 Regulus | 24 | Calibration | 64 |
| STA F1/F3/F5 Jupiter [®] and DSC 404 F1/F3 Pegasus [®] | 26 | Calibration Materials..... | 66 |
| Crucibles Made of Aluminum..... | 27 | Calibration Kits for DSC and DTA..... | 67 |
| DSC Crucibles for Measurements in a Pressure-Tight Environment | 28 | Calibration Kits for TGA and STA | 70 |
| Large TGA Crucibles and Slip-On Plates..... | 29 | Chemical Behavior of Pt, Al₂O₃ and Graphite Crucibles and Sensors | 72 |
| Knudsen Cells (TGA); TGA and TGA-DTA Crucibles Through to the Highest Temperature Range | 30 | Material Compatibility – Sample in Crucible | 74 |
| TGA-DTA, DSC and TGA-DSC Crucibles | 32 | Recommendations for Cleaning Al₂O₃ and Pt Crucibles | 76 |
| Crucibles for Automatic Sample Changer (ASC) | 34 | | |
| Applications – Special Crucibles for Large Samples, Oxygen-Sensitive Alloys..... | 36 | | |
| Applications – Special Crucibles for High Temperatures | 38 | | |

Crucibles

Selection of Crucibles

Crucibles and Their Selection

Crucibles and their lids are made of materials resistant to high temperatures, usually porcelain or an inert metal. Ceramics such as alumina, zirconia, and especially magnesia will tolerate the highest temperatures. One of the first metals used in crucible production was platinum; more recently, metals

such as nickel and zirconium have also been used. The type of crucible used for thermoanalytical measurements can have a strong influence on the measurement results obtained. Additionally, the crucible can also influence the characteristics of the instrument's measuring cell.



Important Factors for Selection of Suitable DSC, TGA and STA Crucibles for Your Sample

- The DSC crucible should have a flat bottom and be made of a material with a high thermal conductivity. This guarantees optimum heat transfer and low temperature gradients between the sample, crucible and sensor.
- The crucible should be made of an inert material in order to prevent reactions with the sample in the programmed temperature range. Exceptions are crucibles for which a catalytic effect on the sample is desired (e.g., copper crucible for OIT tests, etc.).
- The crucible should not exhibit any phase transitions or other effects in the programmed temperature range; the melting point or fusion temperature must exceed the maximum application temperature to a sufficient degree.
- The dimension, shape and specific heat of the crucible should be optimized to achieve and/or maintain the highest caloric sensitivity and lowest time constant for the measuring system. Optimized parameters will result in sharp, well-defined and clearly separated peaks.
- Crucibles should be reusable, especially special ones for specific applications.



Standard Aluminum Crucibles

Crucibles for the DSC 204 **F1 Phoenix**[®], DSC 200 **F3 Maia**[®], DSC 3500 *Sirius* and DSC 214 *Polyma*

The following tables list the crucibles for the low-temperature DSC systems DSC 204 **F1 Phoenix**[®], DSC 200 **F3 Maia**[®], DSC 3500 *Sirius* and DSC 214 *Polyma*. Many of these crucibles can also be used for the high-temperature DSC and STA systems and can be found in those tables as well.

Please note that the temperatures listed indicate the maximum temperature ranges of the crucible material but not of the DSC systems.

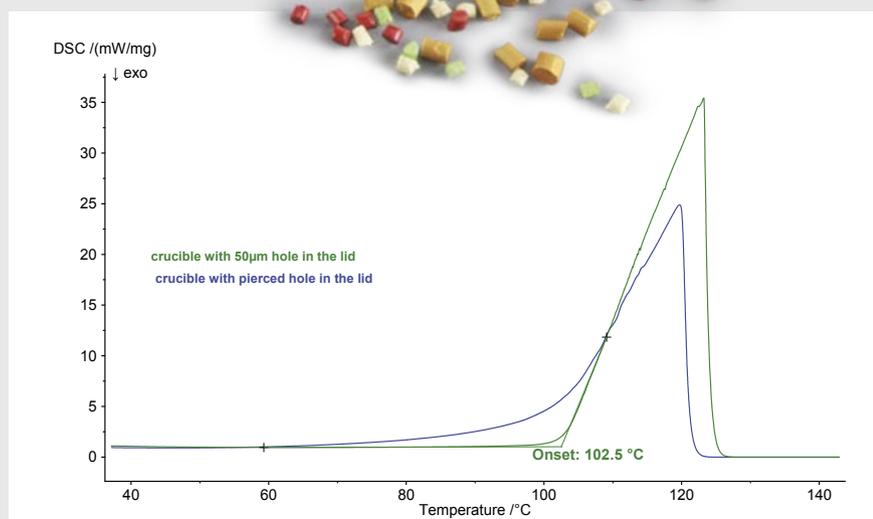
Some of the crucibles made of aluminum can be cold-welded with a

sealing press. The standard Al crucibles and lids can be sealed to obtain a volume of either 40 μl or 25 μl by simply reversing the lid. This ensures that samples of various shapes, such as pellets or fibers, can make optimum contact with the crucible bottom.

The premium *Concavus* pans have a unique geometry which features a concave bottom. The pans are delivered in the antistatic 3in1 Box. This elaborate packing prevents deformation of the pans, while allowing easy access of them and providing a fully functional archiving system.

For special applications such as boiling point determination for volatile substances or gypsum investigations, Al lids with a laser-cut hole (50 μm) can be used. The lids can be cold-welded to the standard Al crucible.

Our high-pressure crucibles are capable of withstanding a maximum of 100 bar of internal pressure. A single sealing tool handles the job identically for all high-pressure crucible varieties. Each crucible comes with a seal. The gold seals will be expended but can be ordered separately in order to continue using the crucible. The medium-pressure crucible withstands pressures up to 20 bar.



Using Al crucibles and lid with laser-cut hole (\varnothing 50- μm hole) suppresses evaporation of solvents and other volatiles. Furthermore, these lids can be used for gypsum investigations.

Al Lid with Laser-Cut Hole

Evaporation of volatile substances usually starts before the boiling point of the volatile is reached.

This measurement shows that the evaporation of water can be suppressed by using a cold weldable aluminum lid with a defined hole (\varnothing 50 μm , green curve). The comparison measurement uses a lid with a punched hole. Here, evaporation already begins at slightly above 60°C (blue curve).

Cold weldable lids with laser-cut holes can also be used for gypsum applications. The separation of dihydrate (DH) and half hydrate (HH) can be achieved.

Standard Crucibles Made of Aluminum for the DSC 204 F1 Phoenix®, DSC 200 F3 Maia®, DSC 3500 Sirius and DSC 214 Polyma

| Material (Purity) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|-------------------|-------------------|--|-------------------|--|---------------------------|
| Al (99.5) | Max. 600°C | Crucible + lid | ø 6 mm; 25/40 µl* | Set of 100 pcs., cold weldable** | 6.239.2-64.5.00 |
| Al (99.5) | Max. 600°C | Crucible + lid | ø 6 mm; 25/40 µl* | Set of 500 pcs., cold weldable** | 6.239.2-64.51.00 |
| Al (99.5) | Max. 600°C | Crucible | ø 6 mm; 25/40 µl* | 100 pieces | 6.239.2-64.5.01 |
| Al (99.5) | Max. 600°C | Lid | | Set of 100 pcs. for 6.239.2-64.5.01 | 6.239.2-64.5.02 |
| Al (99.5) | Max. 600°C | Crucible + lid with laser-cut hole (50 µm) | ø 6 mm; 40 µl | Set of 100 pcs., cold weldable** | 6.239.2-64.8.00 |
| Al (99.5) | Max. 600°C | Crucible + lid with laser-cut hole (50 µm) | ø 6 mm; 40 µl | Set of 500 pcs., cold weldable** | 6.239.2-64.81.00 |
| Al (99.5) | Max. 600°C | 96 <i>Concavus</i> pans and lids*** | ø 5 mm; 30/40 µl | 3in1 Box (archiving system) | DSC21400A66.010.00 |
| Al (99.5) | Max. 600°C | Crucible + lid | ø 5 mm; 30/40 µl | Set of 5x96 pieces incl. 3in1 box | DSC21400A66.020.00 |
| Al (99.5) | Max. 600°C | <i>Concavus</i> pan*** | ø 5 mm; 30/40 µl | 96 pieces including 3in1 box | NGB814672 |
| Al (99.5) | Max. 600°C | <i>Concavus</i> lid*** | | Set of 96 pieces | NGB814673 |
| Al (99.5) | Max. 600°C | Crucible + lid | ø 5 mm; 30/40 µl | Set of <i>Concavus</i> pans and lids, 100 pieces each, without tray | DSC21400A66.030.00 |
| Al (99.5) | Max. 600°C | Crucible + lid | ø 5 mm; 30/40 µl | Set of <i>Concavus</i> pans and lids, 500 pieces each, without tray | DSC21400A66.040.00 |
| Al (99.5) | Max. 600°C | Crucible + lid with laser-cut hole (50 µm) | ø 5 mm; 30/40 | Set of 100 <i>Concavus</i> pcs. + lid, cold weldable** | DSC21400A66.050.00 |
| Al (99.5) | Max. 600°C | Lid with laser-cut hole (50 µm) | | Pierced lids for <i>Concavus</i> crucibles, 100 pcs. | DSC21400A66.051.00 |
| Al (99.5) | Max. 600°C | Slide-in lid | | For <i>Concavus</i> pans; allows sample to be pressed onto crucible bottom, set of 100 pcs., especially for foils and fibers; requires tool kit DSC21400A80.030-00 for sealing press 6.240.10-80.0.00A | NGB815051 |
| Al (99.5) | Max. 600°C | Crucible | ø 6.7mm; 85 µl | Set of 100 pieces | NGB810405 |
| Al (99.5) | Max. 600°C | Set | | 2 x 3in1 boxes, <i>Concavus</i> sealing tool, <i>Concavus</i> calibration set | DSC21400A91.010.00 |
| Al (99.5) | Max. 600°C | Lid | | 100 pieces, for NGB810405 | NGB810406 |

* Crucible volumes of either 25 µl or 40 µl can be achieved by simply reversing the lid.

** One and the same sealing press is capable of sealing all standard Al crucibles; order no. 6.240.10-80.0.00A.

***Using *Concavus* pans with ASC instrument which were delivered before 2014 may require re-adjustment of the ASC gripper.



Concavus pans with lids



Al crucibles with lids



Slide-in lid for Concavus pan; for demonstration purposes, the covers are colored



Al crucible with laser-cut Al lid

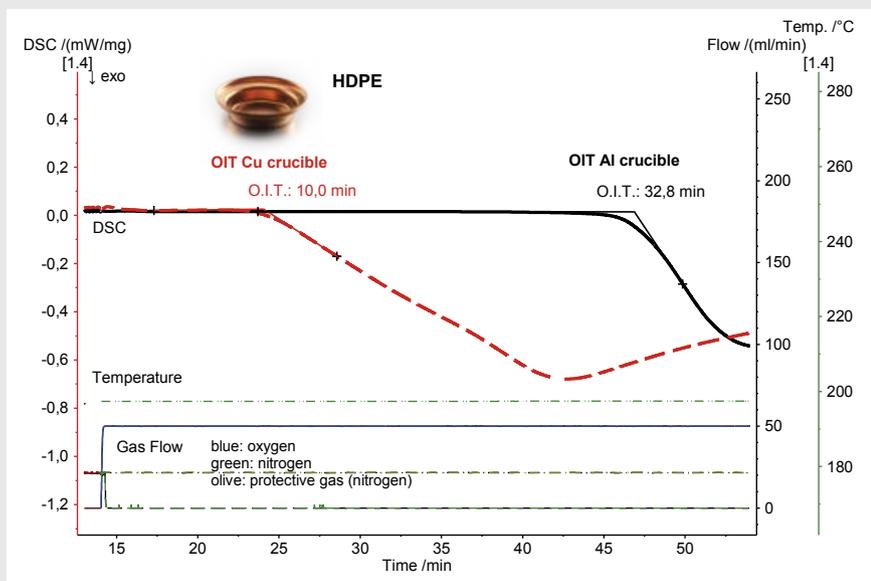
Crucibles for General and Special Applications

Crucibles for General Applications – DSC 204 **F1 Phoenix**[®], DSC 200 **F3 Maia**[®], DSC 3500 **Sirius** and DSC 214 **Polyma**

| Material (Purity) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|---------------------------------------|-------------------|----------------|-------------------|----------------------------|------------------------|
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | ø 6.8 mm / 85 µl | | GB399972 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | For GB399972 | GB399973 |
| Fused silica | Max. 1000°C | Crucible | ø 6.7 mm / 85 µl | | GB399974 |
| Fused silica | Max. 1000°C | Lid | | For GB399974 | GB399975 |
| Pt/Rh (80/20) | Max. 1700°C | Crucible | ø 6.8 mm / 85 µl | | GB399205 |
| Pt/Rh (80/20) | Max. 1700°C | Crucible | ø 6.8 mm / 190 µl | | NGB801556 |
| Pt/Rh (80/20) | Max. 1700°C | Lid | | For GB399205 and NGB801556 | GB399860 |
| Gold (99.9) | Max. 900°C | Crucible + lid | ø 6.7 mm / 85 µl | | 6.225.6-93.3.00 |
| Silver | Max. 750°C | Crucible + lid | ø 6.7 mm / 85 µl | | 6.225.6-93.4.00 |



Ampoules made of Duran glass or fused silica are available.



Influence of the Crucible on the Oxidative-Induction Time (OIT)

The oxidative-induction time (OIT) can be determined in standard aluminum or in open copper crucibles in accordance with ASTM D3895.

This plot represents an OIT measurement of HDPE carried out in open copper (red) and Al (black) crucible respectively. It can clearly be seen that, under isothermal conditions, oxidation of HDPE begins approximately 23 min earlier in the copper crucible than in the Al crucible.

Comparison of the oxidative-induction time in open copper and aluminum crucibles.



Special crucibles made of copper and aluminum for OIT tests

Special Crucibles for Special Applications – DSC 204 *F1 Phoenix*[®], DSC 200 *F3 Maia*[®], DSC 3500 *Sirius* and DSC 214 *Polyma*

| Material (Purity) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|---|-------------------|--|-------------------|---|-------------------------|
| Cu | Max. 600°C | Crucible | ø 6 mm / 25/40 µl | For OIT measurements according to ASTM D3895 (Oxidative-Induction Time), Set of 25 | 6.239.2-64.6.00 |
| Duran glass | Max. 600°C | Ampoule | 80 µl | Set of 100, not available for DSC 214 <i>Polyma</i> , after sealing ampoule length ≈ 19 mm | 6.240.1-93.2.00 |
| Fused silica | Max. 1000°C | Ampoule | 80 µl | After sealing ampoule length ≈ 19 mm | NGB815223 |
| For DSC 204 F1 : Auxiliary kit for glass ampoules | | <ul style="list-style-type: none"> ▪ Seal-off support for glass ampoules ▪ Small welder with butane and oxygen gas bottles ▪ Inner lid for measuring cell for use with glass ampoules | | | 6.240.10-93.1.00 |
| For DSC 200 F3 <i>Maia</i> [®] : Auxiliary kit for glass ampoules | | <ul style="list-style-type: none"> ▪ Seal-off support for glass ampoules ▪ Small welder with butane and oxygen gas bottles ▪ Inner lid for measuring cell for use with glass ampoules ▪ 100 ampoules | | | 6.240.20-93.1.00 |
| For DSC 204 F1 <i>Phoenix</i> [®] with τ-sensor and DSC 214 <i>Polyma</i> : Al (99.5) | Max. 600°C | Crucible | ø 6.7 mm / 85 µl | Especially for determination of the OIT of lubricants and grease in accordance to ASTM D5483-5 and Solid Fat Index (SFI) in accordance to ASTM D6186 Required accessories to obtain OIT/SFI crucible design: <ul style="list-style-type: none"> ▪ Sealing press 6.240.10-80.0.00A ▪ Stamping toolkit 6.240.10-84.0.00 ▪ Lid NGB810406 | NGB810405 |

Sealing Press, Sample Preparation and Pressure-Tight Crucibles

Sealing Press/Tools

- The sealing press (order no. 6.240.10-80.0.00A) can be equipped with five toolkits for sealing and stamping of different crucible types:
 - Toolkit for *Concavus* pan: order no. 6.240.10-85.0.00
 - Toolkit for Al crucible: order no. 6.240.10-81.0.00
 - Toolkit for low-pressure Al crucible: order no. 6.240.10-82.0.00
 - Toolkit for sealing of medium-pressure crucibles: order no. 6.240.10-83.0.00
 - Stamping toolkit 6.240.10-84.0.00 for SFI crucible NGB810405
 - Toolkit for inserting slide-in lid (order no. NGB815051) into *Concavus* pan: order no. DSC21400A80.030-00
- Sealing tool for the high-pressure crucibles, order no. 6.239.2-92.4.00



Sample Preparation

For the sample preparation, we offer two kits for the low- and high-temperature range:

- Kit for low-temperature range: cutting board, scissors, hollow punch, scribe, glass-fiber brush, tweezers, spatula, scalpel, 5 scalpel blades, 5 single-edge blades, ball pen, post-it, space for business card of the NETZSCH contact, manual order no. 6.240.12-99.0.00



- Kit for high-temperature range: cutting board, scissors, hollow punch, scribe, glass-fiber brush, tweezers, spatula, scalpel, 5 scalpel blades, 5 single-edge blades, shaping tool, crucible holder, weighing support, crucible box with 4 Al₂O₃ crucibles, post-it, manual order no. DSC404F1A99.000-00



- *SampleCutter* tool for cutting and creating of plane-non-metallic samples (e.g., polymer granulates), order no. NGB814695
 - 10 spare blades for *SampleCutter*, order no. NGB 814821

Crucibles Made of Aluminum – Low-Pressure*

| Material (Purity) | Temperature Range | Consisting of | Dimension/Volume/Pressure | Remarks | Order Number |
|-------------------|-------------------|----------------|---------------------------|----------------------------------|--------------------------|
| Al (99.5) | Max. 600°C | Crucible + Lid | ø 6 mm / 35 µl; 3 bar | Set of 100 pieces, cold weldable | 6.240.10-65.1.00 |
| Al (99.5) | Max. 600°C | Crucible + Lid | ø 6 mm / 35 µl; 3 bar | Set of 500 pieces, cold weldable | 6.240.10-65.11.00 |
| Al (99.5) | Max. 600°C | Crucible | ø 6 mm / 35 µl; 3 bar | 100 pieces | NGB810419 |
| Al (99.5) | Max. 600°C | Lid | For crucibles of ø 6 mm | 100 pieces | NGB810420 |

*Please note, the low-pressure crucibles made of Al are not available for the automatic sample changer (ASC) for 192 sample pans.

Medium- and High-Pressure Crucibles

| Material (Purity) | Temperature Range | Consisting of | Dimension/Volume/Pressure | Remarks | Order Number |
|------------------------|-------------------|---|------------------------------|---|-------------------------|
| CrNi steel (AISI 304) | Max. 250°C | Crucible + lid + elastomer seal | 120 µl; 20 bar | Set of 25 pieces; seal made of FPM | 6.240.1-68.1.00 |
| O-ring seal | Max. 230°C | O-ring | Max. 20 bar | Seal made of PCTFE | NGB801765 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, gold-plated surface, reusable | ø 6 mm, 27 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.3.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, gold-plated surface, reusable | ø 6 mm, 27 µl, max. 100 bar | 10 crucibles + 50 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.31.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, gold-plated surface, reusable | ø 6 mm, 100 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.8.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 27 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-93.3.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 27 µl, max. 100 bar | 10 crucibles + 50 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-93.31.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 100 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-93.4.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 100 µl, max. 100 bar | 10 crucible + 50 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-93.41.00 |
| Titanium (Grade 4) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 27 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.6.00 |
| Titanium (Grade 4) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 100 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.9.00 |
| Au seals (Au 585) | Max. 500°C | For all high-pressure autoclaves, alloy: Au 58.5%, Ag 26%, Cu 15.5% | | | 6.240.1-91.1.00 |
| Sealing press | | For crucibles: 6.239.2-92.3.00, -92.31.00, -92.6.00, -92.8.00, -92.9.00, -93.3.00, -93.31.00, -93.4.00, -93.41.00 | | | 6.239.2-92.4.00 |



Medium-pressure crucible (right) and high-pressure crucible (left)

Crucibles for Operation with the Automatic Sample Changer (ASC)

Crucibles for DSC 200 **F3 Maia**[®], DSC 3500 *Sirius* and DSC 214 *Polyma* in ASC Operation

| Crucible Material (Purity/%), Volume Lid | ASC Gripper DSC 200 F3 (TG 209 F3) | Carousel Standard Crucibles | Carousel Large Crucibles | Order Number |
|--|---|-----------------------------|---------------------------|---------------------------|
| Al (99.5), 30/40 µl (<i>Concavus</i> pan) | x* | x | x | DSC21400A66.010.00 |
| Crucible with welded lid | x* | x | x | |
| Al (99.5), 25/40 µl | x | x | x | 6.239.2-64.5.00 |
| Crucible with welded lid | x | x | x | |
| Al (99.5), 25/40 µl (500/pkg.) | x | x | x | 6.239.2-64.51.00 |
| Crucible with welded lid | x | x | x | |
| Al (99.5), 25/40 µl | x | x | x | 6.239.2-64.8.00 |
| Crucible and lid with laser-cut-hole | x | x | x | |
| Al (99.5), 25/40 µl (500/pkg.) | x | x | x | 6.239.2-64.81.00 |
| Crucible and lid with laser-cut-hole | x | x | x | |
| Al (99.5), low-pressure, 35 µl | x | x | x | 6.240.10-65.1.00 |
| Crucible with welded lid | x | x | x | |
| CrNi steel (AISI 304), medium-pressure, 120 µl, 20 bar | x | x | Only for manual operation | 6.240.1-68.1.00 |
| Crucible with lid | x | x | | |
| Copper, 25 µl | x | x | Only for manual operation | 6.239.2-64.6.00 |
| CrNi steel (AISI 316L), high-pressure, 27 µl, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.3.00 |
| CrNi steel (AISI 316L), high-pressure, 27 µl, 100 bar | x | x | Only for manual operation | 6.239.2-93.3.00 |
| CrNi (AISI 316L), high-pressure, 100 µl, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.8.00 |
| CrNi (AISI 316L), high-pressure, 100 µl, 100 bar | x | x | Only for manual operation | 6.239.2-93.4.00 |
| Ti (Grade 4), high-pressure, 27 µl, 100 bar | x | x | Only for manual operation | 6.239.2-92.6.00 |
| Ti (Grade 4), high-pressure, 100 µl, 100 bar | x | x | Only for manual operation | 6.239.2-92.9.00 |
| Al ₂ O ₃ (99.7), ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399972 |
| Crucible with lid (note: lid order no.) | x | x | | GB399973 |
| Fused silica, ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399974 |
| Crucible with lid (note: lid order no.) | - | x | | GB399975 |
| Pt/Rh (80/20), ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399205 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |

| Crucible Material (Purity/%), Volume Lid | ASC Gripper DSC 200 F3 (TG 209 F3) | Carousel Standard Crucibles | Carousel Large Crucibles | Order Number |
|--|--|-----------------------------------|-----------------------------|------------------------|
| Pt/Rh (80/20), \varnothing 6.8 x 6 mm, 190 μ l | x | x | Only for manual operation | NGB801556 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |
| Al (99.5), \varnothing 6.7 mm, 85 μ l | x | x | Only for manual operation | NGB810405 |
| Crucible with lid (note: lid order no.) | - | x | | NGB810406 |
| Graphite, \varnothing 6.8mm, 85 μ l | x | x | Only for manual operation | GB399956 |
| Crucible with lid (note: lid order no.) | - | x | | GB399957 |
| Gold (99.9), \varnothing 6.8 mm, 85 μ l, | x | x | Only for manual operation | 6.225.6-93.3.00 |
| Crucible with lid (99.9) | - | x | | |
| Silver, \varnothing 6.8 mm, 85 μ l | x | x | Only for manual operation | 6.225.6-93.4.00 |
| Crucible with lid | - | x | | |
| ZrO ₂ , \varnothing 6.8mm, 85 μ l | x | x | Only for manual operation | GB397053 |
| Crucible with lid (note: lid order no.) | - | x | | GB397052 |
| Y ₂ O ₃ , \varnothing 6.8mm, 85 μ l | x | x | Only for manual operation | NGB812636 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812635 |
| MgO, \varnothing 6.8mm, 85 μ l | x | x | Only for manual operation | NGB812639 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812637 |
| Al ₂ O ₃ (99.7), \varnothing 8 x 8 mm, 300 μ l | x | - | x | NGB803698 |
| Crucible with lid (note: lid order no.) | - | - | x | NGB808209 |
| Pt/Rh/Al ₂ O ₃ system with lid | - | x | Only for manual operation | 6.225.6-93.2.00 |

* Using *Concavus* pans with ASC instrument which were delivered before 2014 may require re-adjustment of the ASC gripper.



Concavus pans with lids, 30 μ l or 40 μ l



Al crucibles with lids, 25 μ l or 40 μ l



Crucibles for Operation with the Automatic Sample Changer (ASC)

Crucibles for DSC 204 **F1** Phoenix® (TG 209 **F1** Libra®) in ASC Operation, possible for μ - and τ -sensor

| Crucible Material (Purity/%), Volume Lid | ASC Gripper DSC 204 F1 (TG 209 F1) | Carousel Standard Crucibles | Carousel Large Crucibles | Order Number |
|---|---|-----------------------------|---------------------------|---------------------------|
| Al (99.5), 30/40 μ l (Concavus pan) | x* | x | x | DSC21400A66.010.00 |
| Crucible with welded lid | x* | x | x | |
| Al (99.5), 25/40 μ l | x | x | x | 6.239.2-64.5.00 |
| Crucible with welded lid | x | x | x | |
| Al (99.5), 25/40 μ l (500/pkg.) | x | x | x | 6.239.2-64.51.00 |
| Crucible with welded lid | x | x | x | |
| Al (99.5), 25/40 μ l | x | x | x | 6.239.2-64.8.00 |
| Crucible and lid with laser-cut-hole | x | x | x | |
| Al (99.5), 25/40 μ l (500/pkg.) | x | x | x | 6.239.2-64.81.00 |
| Crucible and lid with laser-cut-hole | x | x | x | |
| Al (99.5), low-pressure, 35 μ l | x | x | x | 6.240.10-65.1.00 |
| Crucible with welded lid | x | x | x | |
| CrNi steel (AISI 304), medium-pressure, 120 μ l, 20 bar | - | x | Only for manual operation | 6.240.1-68.1.00 |
| Crucible with lid | - | x | | |
| Copper, 25 μ l | x | x | Only for manual operation | 6.239.2-64.6.00 |
| CrNi steel (AISI 316L), high-pressure, 27 μ l, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.3.00 |
| CrNi steel (AISI 316L), high-pressure, 27 μ l, 100 bar | x | x | Only for manual operation | 6.239.2-93.3.00 |
| CrNi (AISI 316L), high-pressure, 100 μ l, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.8.00 |
| CrNi (AISI 316L), high-pressure, 100 μ l, 100 bar | x | x | Only for manual operation | 6.239.2-93.4.00 |
| Ti (Grade 4), high-pressure, 27 μ l, 100 bar | x | x | Only for manual operation | 6.239.2-92.6.00 |
| Ti (Grade 4), high-pressure, 100 μ l, 100 bar | x | x | Only for manual operation | 6.239.2-92.9.00 |



Concavus pans in a 3in1 Box

| Crucible Material (Purity/%), Volume Lid | ASC Gripper DSC 204 F1 (TG 209 F1) | Carousel Standard Crucibles | Carousel Large Crucibles | Order Number |
|---|--|-----------------------------------|-----------------------------|-----------------|
| Al ₂ O ₃ (99.7), ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399972 |
| Crucible with lid (note: lid order no.) | x | x | | GB399973 |
| Fused silica, ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399974 |
| Crucible with lid (note: lid order no.) | - | x | | GB399975 |
| Pt/Rh (80/20), ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399205 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |
| Pt/Rh (80/20), ø 6.8 mm, 85 µl | x | x | Only for manual operation | NGB801556 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |
| Al (99.5), ø 6.7 mm, 85 µl | x | x | Only for manual operation | NGB810405 |
| Crucible with lid (note: lid order no.) | - | x | | NGB810406 |
| Graphite, ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399956 |
| Crucible with lid | - | x | | GB399957 |
| Gold, ø 6.8 mm, 85 µl | x | x | Only for manual operation | 6.225.6-93.3.00 |
| Crucible with lid | - | x | | |
| Silver, ø 6.8 mm, 85 µl | x | x | Only for manual operation | 6.225.6-93.4.00 |
| Crucible with lid (note: lid order no.) | - | x | | |
| ZrO ₂ , ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB397053 |
| Crucible with lid (note: lid order no.) | - | x | | GB397052 |
| Y ₂ O ₃ , ø 6.8 mm, 85 µl | x | x | Only for manual operation | NGB812636 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812635 |
| MgO, ø 6.8 mm, 85 µl | x | x | Only for manual operation | NGB812639 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812637 |
| Al ₂ O ₃ (99.7), ø 8 x 8 mm, 300 µl | - | - | x | NGB803698 |
| Crucible with lid (note: lid order no.) | - | - | x | NGB808209 |
| Pt/Rh/Al ₂ O ₃ system with lid | - | x | Only for manual operation | 6.225.6-93.2.00 |

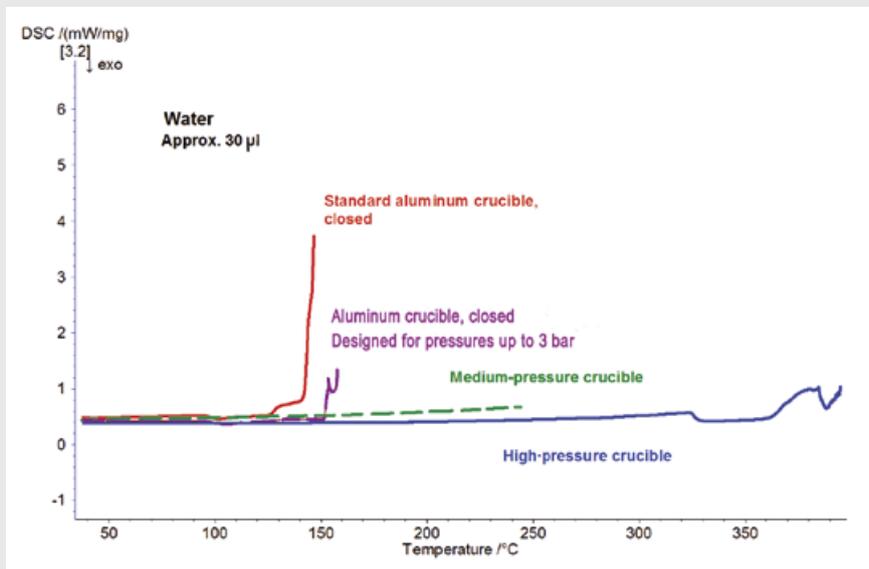
* Using *Concavus* pans with ASC instrument which were delivered before 2014 may require re-adjustment of the ASC gripper.



Special Crucibles for Special Applications

Internal Pressure Capabilities of Sealed Al, Fine Steel, and CrNi Steel Crucibles

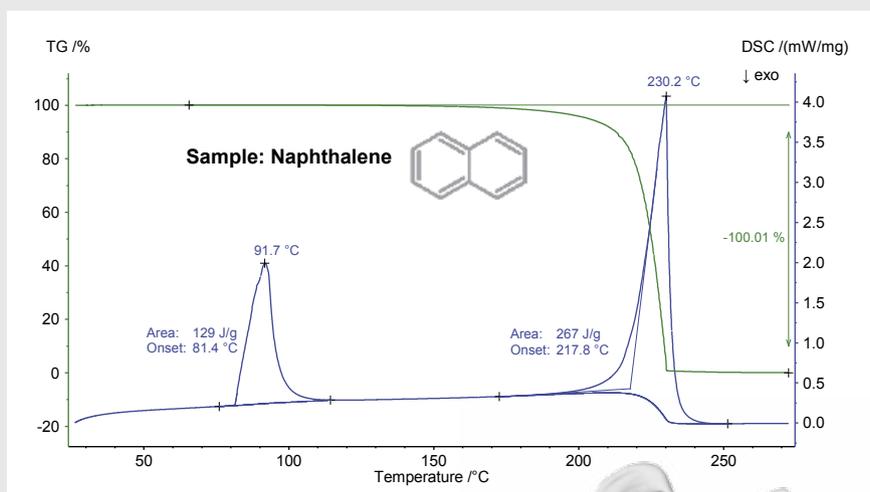
A material's potential reaction and/or type of investigation being conducted (e.g., denaturation of starch, curing of phenolic resin), may warrant the use of crucibles capable of certain levels of internal pressure. The plot shows four measurements on water, of approximately 30 μl each, in different crucible types: Al standard, Al low-pressure (max. 3 bar), fine steel medium-pressure, and CrNi steel autoclaves. It can be observed that the standard Al crucible starts to leak at approximately 125°C, while the low-pressure Al pan opens above 150°C when water is trapped inside. The medium-pressure crucibles (20 bar) withstand the inner pressure increase up to their maximum temperature. The high-pressure crucibles begin to leak around 300°C.



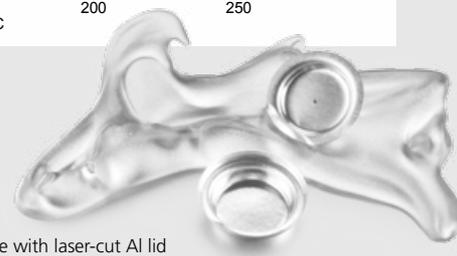
Comparison of measurements done in crucibles capable of withstanding various levels of internal pressure

Melting and Boiling Point Determination for a Polycyclic Aromatic Hydrocarbon

Polycyclic aromatic hydrocarbons (PAHs), such as naphthalene, are formed during natural and anthropogenic processes like volcanic eruptions, forest fires, etc. They are released into the atmosphere and, due to their hydrophobicity, accumulate in soil and may enter into the food chain. This measurement was designed to determine the melting and boiling point of naphthalene. A sealed Al crucible with a 50- μm laser-cut hole was used. At an extrapolated onset temperature of 81°C, melting occurred with an enthalpy of 129 J/g. A mass-loss step of 100% took place between 150°C and 230°C; this reflects evaporation of the sample. This effect was accompanied by an endothermic DSC peak with an enthalpy of 267 J/g and an extrapolated onset of 218°C. The latter reflects the boiling point of naphthalene. These results correspond to the nominal values.



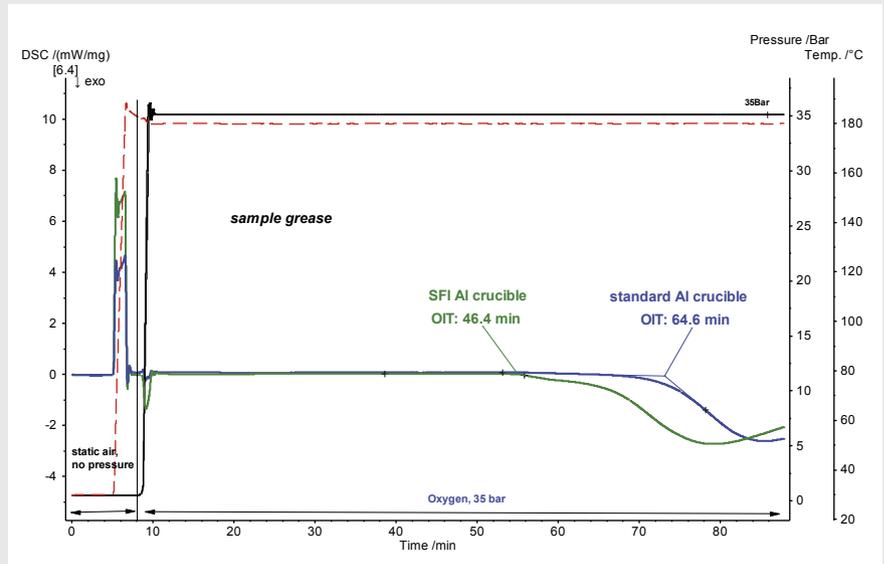
STA 449 **F3 Jupiter**[®] measurement using the TGA-DSC sensor for determination of the melting and boiling points of naphthalene (sample mass 22.66 mg, heating rate 10 K/min, 70 ml/min nitrogen atmosphere)



Al crucible with laser-cut Al lid

Oxidative-Induction Time (OIT) and Solid Fat Index (SFI) Tests on Lubricating Greases

Grease and wax samples can be prepared with more consistent surface areas when using SFI crucibles instead of flat-bottom crucibles. The results show an increased reproducibility. In this example, an OIT measurement on grease was carried out with the DSC 204 HP *Phoenix*® with a standard Al crucible and the SFI crucible under an oxygen pressure of 35 bar as per ASTM D5483-5. The difference in the OIT is clearly visible. The oxidative-induction time of the grease in the SFI crucible is approximately 21 min shorter than the one in the standard Al crucible. This effect results from the more consistent sample surface area made possible by use of the SFI crucible.



Oxidation stability in two different Al crucibles



SFI crucible for an OIT test of lubricants and greases on special stamping tool for sealing press 6.240.10-80.0.00A

Crucibles for General Applications

Crucibles for the TG 209 **F1** *Libra*[®] and TG 209 **F3** *Tarsus*[®]

The TG 209 **F1** *Libra*[®] and TG 209 **F3** *Tarsus*[®] both offer sample carriers for regular and large sample masses, guaranteeing broad applications in the temperature range from room temperature to 1000°C and 1100°C, respectively.

A large variety of crucibles is available to cover the temperature range for different materials, including large crucibles required for substances with a high sample mass and low density (e.g., foams, aerogels, etc.).



Crucibles for General Applications – TG 209 F1 Libra® and TG 209 F3 Tarsus®

| Material (Purity/%) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|---------------------------------------|-------------------|----------------|------------------------------------|--|------------------------|
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | ø 6.8 mm / 85 µl | | GB399972 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | For GB399972 | GB399973 |
| Fused silica | Max. 1000°C | Crucible | ø 6.7 mm / 85 µl | | GB399974 |
| Fused silica | Max. 1000°C | Lid | | For GB399974 | GB399975 |
| Pt/Rh (80/20) | Max. 1700°C | Crucible | ø 6.8 mm / 85 µl | | GB399205 |
| Pt/Rh (80/20) | Max. 1700°C | Lid | | For GB399205 and NGB801556 | GB399860 |
| Pt/Rh (80/20) | Max. 1700°C | Crucible | ø 6.8 mm / 190 µl | | NGB801556 |
| Al (99.5) | Max. 610°C | Crucible | ø 6.7 mm / 85 µl | Set of 100 pieces | NGB810405 |
| Al (99.5) | Max. 610°C | Lid | | For NGB810405 | NGB810406 |
| Gold (99.9) | Max. 900°C | Crucible + lid | ø 6.7 mm / 85 µl | | 6.225.6-93.3.00 |
| Silver | Max. 750°C | Crucible + lid | ø 6.7 mm / 85 µl | | 6.225.6-93.4.00 |
| ZrO ₂ | Max. 2000°C | Crucible | 85 µl | CaO-stabilized | GB397053 |
| ZrO ₂ | Max. 2000°C | Lid | | For GB397053 | GB397052 |
| Graphite | Max. 2200°C | Crucible | 85 µl | | GB399956 |
| Graphite | Max. 2200°C | Lid | | For GB399956 | GB399957 |
| Al ₂ O ₃ (99.8) | Max. 1700°C | Crucible | ø 9 mm, height 7 mm, volume 350 µl | Sample holder for large samples required | NGB800453 |
| Al ₂ O ₃ (99.8) | Max. 1700°C | Lid | | | NGB800454 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | ø 8 mm, height 8 mm, volume 300 µl | Sample holder for large samples required, ASC-compatible | NGB803698 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | For crucible NGB803698 | NGB808209 |



Large crucible made of Al₂O₃, volume 350 ul



Large crucible made of Al₂O₃, volume 300 ul



Crucibles made of Pt

Crucibles for Operation with the Automatic Sample Changer (ASC)

| Crucibles for TG 209 F1 <i>Libra</i> [®] (& DSC 204 F1 <i>Phoenix</i> [®]) in ASC Operation | | | | |
|--|---|-----------------------------|---------------------------|---------------------------|
| Crucible Material (Purity/%), Volume Lid | ASC Gripper TG 209 F1 (DSC 204 F1) | Carousel Standard Crucibles | Carousel Large Crucibles | Order Number |
| Al (99.5), 30/40 µl (<i>Concavus</i> pan)* | x* | x | x | DSC21400A66.010-00 |
| Crucible with welded lid | x | x | x | |
| Al (99.5), 25/40 µl | x | x | x | 6.239.2-64.5.00 |
| Crucible with welded lid | x | x | x | |
| Al (99.5), 25/40 µl | x | x | x | 6.239.2-64.8.00 |
| Crucible and lid with laser-cut-hole | x | x | x | |
| Al (99.5), low-pressure, 35 µl | x | x | x | 6.240.10-65.1.00 |
| Crucible with welded lid | x | x | x | |
| CrNi steel (AISI 304), medium-pressure, 120 µl, 20 bar | - | x | Only for manual operation | 6.240.1-68.1.00 |
| Crucible with lid | - | x | | |
| Copper, 25 µl | x | x | Only for manual operation | 6.239.2-64.6.00 |
| CrNi steel (AISI 316L), high-pressure, 27 µl, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.3.00 |
| CrNi steel (AISI 316L), high-pressure, 27 µl, 100 bar | x | x | Only for manual operation | 6.239.2-93.3.00 |
| CrNi (AISI 316L), high-pressure, 100 µl, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.8.00 |
| CrNi (AISI 316L), high-pressure, 100 µl, 100 bar | x | x | Only for manual operation | 6.239.2-93.4.00 |
| Ti (Grade 4), high-pressure, 27 µl, 100 bar | x | x | Only for manual operation | 6.239.2-92.6.00 |
| Ti (Grade 4), high-pressure, 100 µl, 100 bar | x | x | Only for manual operation | 6.239.2-92.9.00 |
| Al ₂ O ₃ (99.7), 6.8 mm, 85 µl | x | x | Only for manual operation | GB399972 |
| Crucible with lid (note: lid order no.) | - | x | | GB399973 |
| Fused silica, ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399974 |
| Crucible with lid (note: lid order no.) | - | x | | GB399975 |

* DSC21400A66.020-00: Set with 5 x 96 pans/lids

| Crucible Material (Purity/%), Volume Lid | ASC Gripper TG 209 F1 (DSC 204 F1) | Carousel Standard Crucibles | Carousel Large Crucibles | Order Number |
|---|------------------------------------|-----------------------------|---------------------------|------------------------|
| Pt/Rh (80/20), ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399205 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |
| Pt/Rh (80/20), ø 6.8 x 6 mm, 190 µl | x | x | Only for manual operation | NGB801556 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |
| Al (99.5), ø 6.7 mm, 85 µl | x | x | Only for manual operation | NGB810405 |
| Crucible with lid (note: lid order no.) | - | x | | NGB810406 |
| Graphite, ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399956 |
| Crucible with lid (note: lid order no.) | - | x | | GB399957 |
| Gold, ø 6.8 mm, 85 µl | x | x | Only for manual operation | 6.225.6-93.3.00 |
| Crucible with lid | - | x | | |
| Silver, ø 6.8 mm, 85 µl | x | x | Only for manual operation | 6.225.6-93.4.00 |
| Crucible with lid | - | x | | |
| ZrO ₂ , ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB397053 |
| Crucible with lid (note: lid order no.) | - | x | | GB397052 |
| Y ₂ O ₃ , ø 6.8 mm, 85 µl | x | x | Only for manual operation | NGB812636 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812635 |
| MgO, ø 6.8 mm, 85 µl | x | x | Only for manual operation | NGB812639 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812637 |
| Al ₂ O ₃ (99.7), ø 8 x 8 mm, 300 µl | x | - | x | NGB803698 |
| Crucible with lid (note: lid order no.) | - | - | x | NGB808209 |
| Pt/Rh/Al ₂ O ₃ system with lid | - | x | Only for manual operation | 6.225.6-93.2.00 |

* Using *Concavus* pans with ASC instrument which were delivered before 2014 may require re-adjustment of the ASC gripper.



Crucibles for Operation with the Automatic Sample Changer – Applications

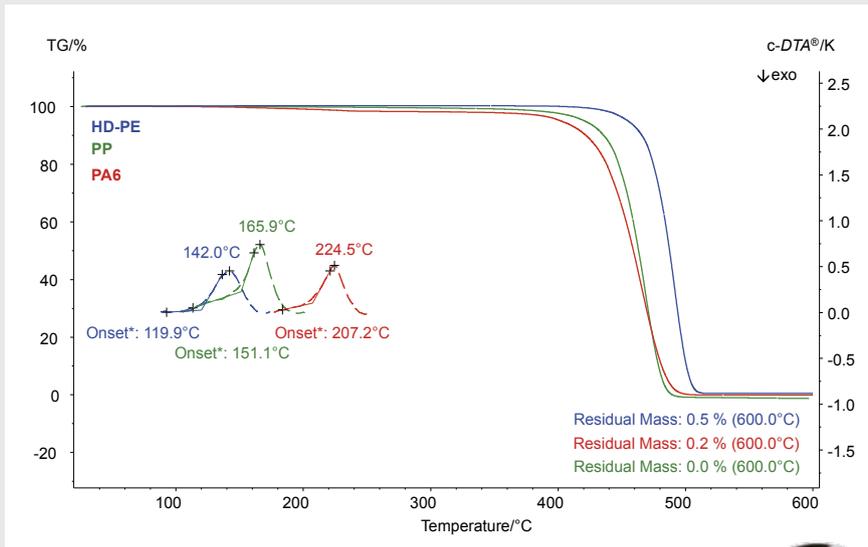
Crucibles for TG 209 **F3** *Tarsus*[®] (& DSC 200 **F3** *Maia*[®], DSC 3500 *Sirius* and DSC 214 *Polyma*) in ASC Operation

| Crucible Material (Purity/%), Volume Lid | ASC Gripper TG 209 F3 (DSC 200 F3) | Carousel Standard Crucibles | Carousel Large Crucibles | Order Number |
|---|---|-----------------------------|---------------------------|---------------------------|
| Al (99.5), 30/40 µl (<i>Concavus</i> pan) | x* | x | x | DSC21400A66.010.00 |
| Crucible with welded lid | x | x | x | |
| Al ₂ O ₃ (99.7), ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399972 |
| Crucible with lid (note: lid order no.) | x | x | | GB399973 |
| Fused silica, ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399974 |
| Crucible with lid (note: lid order no.) | - | x | | GB399975 |
| Pt/Rh (80/20), ø 6.8 mm, 85 µl | x | x | Only for manual operation | GB399205 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |
| Pt/Rh (80/20), ø 6.8 x 6 mm, 190 µl | x | x | Only for manual operation | NGB801556 |
| Crucible with lid (note: lid order no.) | - | x | | GB399860 |
| Al (99.5), ø 6.7 mm, 85 µl | x | x | Only for manual operation | NGB810405 |
| Crucible with lid (note: lid order no.) | - | x | | NGB810406 |
| Graphite, ø 6.8mm, 85 µl | x | x | Only for manual operation | GB399956 |
| Crucible with lid (note: lid order no.) | - | x | | GB399957 |
| Gold (99.9), ø 6.8 mm, 85 µl | x | x | Only for manual operation | 6.225.6-93.3.00 |
| Crucible with lid (99.9) | - | x | | |
| Silver, ø 6.8 mm, 85 µl | x | x | Only for manual operation | 6.225.6-93.4.00 |
| Crucible with lid | - | x | | |
| ZrO ₂ , ø 6.8mm, 85 µl | x | x | Only for manual operation | GB397053 |
| Crucible with lid (note: lid order no.) | - | x | | GB397052 |
| Y ₂ O ₃ , ø 6.8mm, 85 µl | x | x | Only for manual operation | NGB812636 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812635 |
| MgO, ø 6.8mm, 85 µl | x | x | Only for manual operation | NGB812639 |
| Crucible with lid (note: lid order no.) | x | x | | NGB812637 |
| Al ₂ O ₃ (99.7), ø 8 x 8 mm, 300 µl | x | - | x | NGB803698 |
| Crucible with lid (note: lid order no.) | - | - | x | NGB808209 |
| Pt/Rh/Al ₂ O ₃ system with lid | - | x | Only for manual operation | 6.225.6-93.2.00 |

* Using *Concavus* pans with ASC instrument which were delivered before 2014 may require re-adjustment of the ASC gripper.

Please note:

Other crucibles suitable for ASC operation are listed in the ASC table for the DSC 200 **F3** *Maia*[®], DSC 3500 *Sirius* and DSC 214 *Polyma*, see page 12

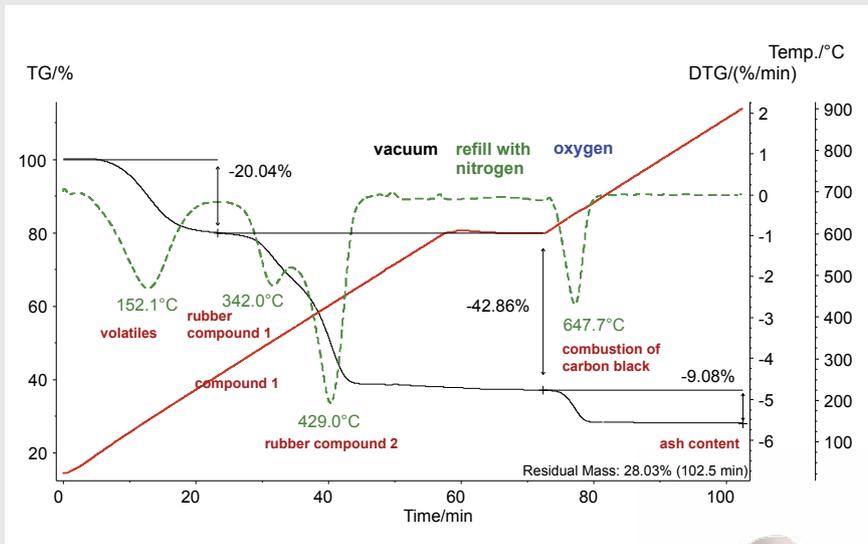


The measurements were carried out using approximately 10 mg of the rubber mixture in a platinum crucible under a nitrogen atmosphere.



c-DTA® for Polymer Identification

HD-PE, PP and PA 6 exhibit very similar decomposition behavior with regard to temperature and residual mass, which makes it difficult to distinguish between them. However, when using the TGA sensor for c-DTA® calibration in combination with a platinum crucible, the characteristic melting peaks of these components can be monitored – even in a TGA system. The melting peak detected at 142°C can be clearly ascribed to HD-PE; the peak at 166°C, to PP; and the peak at 225°C, to PA 6.



This measurement was performed in the TG **F1 Libra**®, using a 10-mg sample mass in an Al₂O₃ crucible.



Precise Determination of the Plasticizer Content of a Tire Mixture

The plasticizer content can be determined precisely by carrying out a TGA measurement under vacuum prior to the decomposition of the rubber mixture. Because the boiling point is depressed under vacuum, the release of the plasticizer at 152°C is clearly separated from the decomposition of the rubber – which consists of two compounds, as indicated by the DTG peaks at 342°C and 429°C. After the decomposition under vacuum, at 600°C, nitrogen is filled into the TGA system. After 20 min stabilization time, the atmosphere is changed to oxygen and the measurement proceeds at a linear heating rate (10 K/min). Under an oxygen atmosphere, carbon black immediately combusts to 9%. The residue in the crucible is the ash content, which was determined here to be 28%.

Crucibles for the STA 2500 *Regulus*



Crucibles for the STA 2500 *Regulus*

The STA 2500 *Regulus* is a standard simultaneous thermal analyzer with trendsetting technology. This top-loading unique differential balance

arrangement is delivered with slip-on plates which allow for nearly all kinds of sample pans. A large slip-on plate is available for a large volume crucible.

Crucibles, Lids/Caps and Slip-on Plates for the STA 2500 *Regulus*

| Material (Purity/%) | Temperature Range | Consisting of | Dimension (ø x Height)/ Volume | Remarks | Order Number |
|---------------------------------------|-------------------|--------------------------------|--------------------------------|---|-----------------|
| Al (99.5) | 600°C | Crucible | ø 5.2 x 2.6 mm / 45 µl | Set of 100 pieces | J1560179 |
| Al (99.5) | 600°C | Crucible | ø 5.2 x 5.1 mm / 95 µl | Large crucible, set of 100 pieces | J1560180 |
| Al (99.5) | 600°C | Lid | ø 4.8 mm | For Al crucibles, Set of 100 pieces | J1560184 |
| Al ₂ O ₃ (99.7) | 1700°C | Crucible | ø 5.2 x 2.6 mm / 45 µl | Set of 2 pieces | J1560181 |
| Al ₂ O ₃ (99.7) | 1700°C | Crucible | ø 5.2 x 5.1 mm / 95 µl | Large crucible, set of 2 pieces | J1560182 |
| Al ₂ O ₃ (99.7) | 1700°C | Lid | ø 5.7 x 2.25 mm | For Al ₂ O ₃ crucibles J1560181 and J1560182, set of 2 pieces | JA000981 |
| Al ₂ O ₃ (99.7) | 1700°C | Crucible | ø 8.0 x 10.0 mm / 400 µl | Only available for large slip-on plate JA004734 | JA004738 |
| Pt (90/10) | 1600°C | Crucible | ø 5.2 x 2.6 mm / 45 µl | Set of 2 pieces | J5900905 |
| Pt (90/10) | 1600°C | Crucible | ø 5.2 x 5.1 mm / 95 µl | Large crucible, set of 2 pieces | J5900879 |
| Pt (90/10) | 1600°C | Lid | ø 4.8 | For Pt crucibles, set of 2 pieces | J5900906 |
| | | Reshaping tool for Pt crucible | | | J7672306 |
| Fused silica | 1000°C | Crucible | ø 5.0 x 2.6 mm / 25 µl | Set of 10 pieces | J5900881 |
| Fused silica | 1000°C | Crucible | ø 5.0 x 5.1 mm / 55 µl | Set of 10 pieces | J1560183 |
| Al ₂ O ₃ (99.7) | 1700°C | Slip-on-plate | | Standard, set of 2 pieces | JA003598 |
| Al ₂ O ₃ (99.7) | 1700°C | Corrosive slip-on-plate | | Set of 2 pieces | J1670612 |
| Al ₂ O ₃ (99.7) | 1700°C | Large volume slip-on-plate | | For sample position | JA004734 |
| Al ₂ O ₃ (99.7) | 1700°C | Large volume slip-on-plate | | For reference position | JA004735 |
| Al ₂ O ₃ + Pt | 1600°C | Flat slip-on-plate | | set of 2 pieces, for crucibles with ø 6.8 mm | JA005724 |



Large crucible made of Al, order no. J1560180



Crucible made of Al₂O₃, order no. J1560181



Large Al₂O₃ crucible, order no. J1560182



Lids made of Al₂O₃, order no. JA000981



Crucible and lid made of Al, order no. J1560179 and J1560184



Crucible made of fused silica, order no. J5900881

Spare parts of the STA 2500 *Regulus*

| Spare parts | Remark | Order Number |
|---------------------------|---------------------------------|-----------------|
| Radiation shield | Standard version | JA003597 |
| Radiation shield | Special version | JA004827 |
| Sample carrier receiver | | JA003596 |
| TGA-DTA sample holder rod | Type P, 1100°C; set of 2 pieces | J1670610 |
| TGA-DTA sample holder rod | Type S, 1600°C; set of 2 pieces | JA004768 |



Large crucible made of Pt,
order no. J5900879



Reshaping tool for crucible made of Pt,
order no. J7672306



Standard radiation shield,
order no. JA003597



Special radiation shield,
order no. JA004827



Slip-on plates for measurements in corrosive
atmospheres, order no. JA004768



Sample carrier receiver,
order no. JA003596



Slip-on plates made of Al₂O₃,
order no. JA003598



Slip-on plates made of Al₂O₃, for
sample and reference position,
order no. JA004734 and JA004735



Large crucible made of Al₂O₃,
order no. JA004738

Crucibles Made of Aluminum

Crucibles for the STA 449 **F1/F3** Jupiter® and DSC 404 **F1/F3** Pegasus®

What sets the STA 449 **F1/F3** Jupiter® and DSC 404 **F1/F3** Pegasus® systems apart is not only their flexibility in furnaces and sensors but also the huge variety of possible crucibles. For the broad temperature range from -150°C to 2400°C, the crucible materials vary from aluminum to magnesium oxide and tungsten to zirconium oxide.

Besides DSC and TGA (for STA) crucibles, plates and net-shaped plates

are also available in different materials and dimensions.

For inhomogeneous samples and those with an extremely low density, special TGA sample holders and large crucibles are available. The automatic sample changer (ASC) with its gripper and carousel allows for the use of nearly any crucible type including specialties such as low-, medium- and high-pressure crucibles.

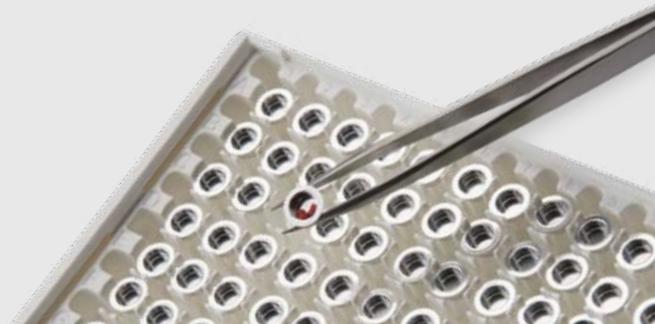


Crucibles Made of Aluminum for the STA 449 F1/F3/F5 Jupiter® and DSC 404 F1/F3 Pegasus®

| Material (Purity/%) | Temperature Range | Consisting of | Dimension/Volume | Remarks | Order Number |
|---------------------|-------------------|----------------------------------|-------------------|--------------------------------------|---------------------------|
| Al (99.5) | Max. 600°C | Concavus pan* + lid | ø 5 mm / 30/40 µl | Set of 96, 3in1 Box, cold weldable** | DSC21400A66.010.00 |
| Al (99.5) | Max. 600°C | Concavus pan* | ø 5 mm / 30/40 µl | 3in1 Box, 96 pieces | NGB814672 |
| Al (99.5) | Max. 600°C | Concavus lid | | 96 pieces | NGB814673 |
| Al (99.5) | Max. 600°C | Crucible + lid | ø 6 mm / 25/40 µl | Set of 100, cold weldable** | 6.239.2-64.5.00 |
| Al (99.5) | Max. 600°C | Crucible + lid | ø 6 mm / 25/40 µl | Set of 500, cold weldable** | 6.239.2-64.51.00 |
| Al (99.5) | Max. 600°C | Crucible + punched lid (0.05 mm) | ø 6 mm / 40 µl | Set of 100, cold weldable** | 6.239.2-64.8.00 |
| Al (99.5) | Max. 600°C | Crucible + punched lid (0.05 mm) | ø 6 mm / 40 µl | Set of 500, cold weldable** | 6.239.2-64.81.00 |
| Al (99.5) | Max. 600°C | Crucible | ø 6.7 mm / 85 µl | Set of 100 pieces, non-weldable | NGB810405 |
| Al (99.5) | Max. 600°C | Lid | | Set of 100 pieces, for NGB810405 | NGB810406 |

* Using Concavus pans with ASC instrument which were delivered before 2014 may require re-adjustment of ASC gripper.

** Please note: One and the same sealing press is capable of sealing all the Al crucibles, order no. 6.240.10-80.0.00A, see optional toolkits, page 12



Concavus pans in the antistatic 3in1 Box

Low-Pressure Crucibles Made of Aluminum for TGA-DSC Sensors of the STA 449 F1/F3/F5 Jupiter®¹ and DSC Sensors of the DSC 404 F1/F3 Pegasus®²

| Material (Purity/%) | Temperature Range | Consisting of | Dimension/Volume/Pressure | Remarks | Order Number |
|---------------------|-------------------|----------------|---------------------------|----------------------------------|--------------------------|
| Al (99.5) | Max. 600°C | Crucible + lid | ø 6 mm / 35 µl; 3 bar | Set of 100 pieces, cold weldable | 6.240.10-65.1.00 |
| Al (99.5) | Max. 600°C | Crucible + lid | ø 6 mm / 35 µl; 3 bar | Set of 500 pieces, cold weldable | 6.240.10-65.11.00 |
| Al (99.5) | Max. 600°C | Crucible | ø 6 mm / 35 µl; 3 bar | 100 pieces | 6.240.10-65.1.01 |
| Al (99.5) | Max. 600°C | Lid | For crucibles of ø 6 mm | 100 pieces | 6.240.10-65.1.02 |

¹ STA 449 F1/F3 Jupiter® sensors: HTP40000AXX.000-00 (XX = 78, 77, 73, 74, 79 or 63), STA 449 F5 Jupiter® sensor HTP40000AXX.000-00 (XX=69, 73)

² DSC 404 F1/F3 Pegasus® sensors: DSC404F1AXX.000-00 (XX = 78, 77, 73, 74, 79 or 75)

DSC Crucibles for Measurements in a Pressure-Tight Environment, Large TGA Crucibles and Slip-On Plates

Medium- and High-Pressure Crucibles for TGA-DSC Sensors (HTP 40000A69/70/71/72.000-00) of the STA 449 **F1/F3/F5** Jupiter® and DSC sensors of the DSC 404 **F1/F3** Pegasus®

| Material | Temperature Range | Consisting of | Dimension/Volume/Pressure | Remarks | Order Number |
|------------------------|-------------------|---|-----------------------------|--|-------------------------|
| CrNi steel (AISI 304) | Max. 250°C | Crucible + lid + seal | 120 µl; 20 bar | Set of 25 pieces, seal made of FPM | 6.240.1-68.1.00 |
| O-ring seal | Max. 230°C | O-ring | Max. 20 bar | Seal made of PCTFE | NGB801765 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, gold-plated surface, reusable | ø 6 mm, 27 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.3.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, gold-plated surface, reusable | ø 6 mm, 27 µl, max. 100 bar | 10 crucible + 50 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.31.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 27 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-93.3.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 27 µl, max. 100 bar | 10 crucible + 50 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-93.31.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6mm, 100 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-93.4.00 |
| CrNi steel (AISI 316L) | Max. 500°C | Crucible + lid, hexagonal, gold-plated surface, reusable | ø 6mm, 100 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.8.00 |
| Titanium (Grade 4) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6 mm, 27 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.6.00 |
| Titanium (Grade 4) | Max. 500°C | Crucible + lid, hexagonal, reusable | ø 6mm, 100 µl, max. 100 bar | 1 crucible + 5 Au sealing disks (sealing press 6.239.2-92.4) | 6.239.2-92.9.00 |
| Au seals (Au585) | Max. 500°C | For all high-pressure autoclaves, alloy: Au 58.5%, Ag 26%, Cu 15.5% | | | 6.240.1-91.1.00 |
| Sealing press | | For crucibles: 6.239.2-92.3.00, -92.31.00, -92.6.00, -92.8.00, -92.9.00, -93.3.00, -93.31.00, -93.4.00, -93.41.00 | | | 6.239.2-92.4.00 |



Sealing tool for the high-pressure crucibles, order no. 6.239.2-92.4.00



Medium-pressure crucible (right) and high-pressure crucible (left)

Crucibles for TGA Sample Carrier (HTP40000A58.000-00) of the STA 449 F1/F3/F5 Jupiter®

| Material (Purity/%) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|---------------------------------------|--|--------------------------|---|---------------------------------|---------------------------|
| Al ₂ O ₃ (99.7) | Max. 1700°C | Slip-on plate, flat | ø 10 mm | | GB343550 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Slip-on plate | ø 17 mm | | GB445240 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | 0.9 ml | | GB445215 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | For GB445215 | GB445216 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | 3.4 ml | | GB445213 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | Lid without hole, for GB445213 | GB445214 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | 5.0 ml | | NGB809163 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | Lid without hole, for NGB809163 | NGB814785 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | For sample dimension: ø 15 mm; hight 25 mm ø 18 mm; ø _{inner} 14.5 mm; | | NGB811934 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | total height 14.5 mm; filling height 3 mm; volume 0.53 ml | For glove box applications | NGB811194 |
| Repository | Crucible repository with handling tool, for glove box crucible NGB811194 | | | | HTP40000A91.000-00 |
| Pt/Ir (90/10) | Max. 1600°C | Net-shaped slip-on plate | ø 17 mm (100 meshes/cm ²) | | GB398029 |
| Pt/Ir (90/10) | Max. 1600°C | Net-shaped slip-on plate | ø 17 mm (225 meshes/cm ²) | | GB398030 |
| Pt/Ir (90/10) | Max. 1600°C | Slip-on plate | ø 10 mm | | GB348834 |
| Pt/Ir (90/10) | Max. 1600°C | Slip-on plate | ø 17 mm | | GB348835 |



Al₂O₃ crucibles with different volumes (0.9 ml, 3.4 ml, 5 ml)



Al₂O₃ slip-on plates (ø 10 mm, 17 mm)



Pt/Ir net-shaped slip-on plates (ø 17 mm, 100/225 meshes/cm²)



Al₂O₃ crucible for glove box applications

Knudsen Cells (TGA), TGA and TGA-DTA Crucibles Through to the Highest Temperature Range

Crucible for TGA Sample Carrier (HTP40000A64.000-00/-010.00)

| Material | Temperature Range | Sample Chamber Dimension | Remarks | Order Number |
|---------------|-------------------|--------------------------|---|------------------|
| Fused Silica* | Max. 1100°C | 3.4 ml | Only for TGA-sample carrier HTP40000A64.000-00 | NGB811507 |

* Not available for STA 449 **F5** Jupiter®



Crucibles for TGA Sample Carriers of the STA 449 **F1/F3** Jupiter® with Turbo Pump – Knudsen Cells

| Material (Purity/%) | Temperature Range | Sample Chamber Dimension | Remarks | Order Number |
|--|-------------------|---|---|------------------------|
| ZrO ₂ ¹ | Max. 2000°C | ø 12 mm x 3.7 mm high; orifice ø 2.896 mm (optically dimensioned) | Crucible + lid | 6.226.1-69.5.00 |
| Al ₂ O ₃ ² (99.7) | Max. 1700°C | ø 12 mm x 3.7 mm high; orifice ø 2.896 mm (optically dimensioned) | Crucible + lid | 6.226.1-67.1.00 |
| Al ₂ O ₃ ² (99.7) | Max. 1700°C | Outlet ø 0.3 mm (optically dimensioned) | Crucible + lid; also for carrier: 6.226.1-58.0.00+S | 6.226.1-69.1.00 |
| Al ₂ O ₃ ² (99.7) | Max. 1700°C | Outlet ø 0.1 mm (optically dimensioned) | Crucible + lid | 6.226.1-69.2.00 |
| Molybdenum ¹ | Max. 2000°C | ø 12 mm x 3.7 mm high; orifice ø 2.896 mm (optically dimensioned) | Crucible + lid | 6.226.1-69.4.00 |
| MgO ² | Max. 1500°C | Outlet ø 0.3 mm (optically dimensioned) | Crucible + lid* | 6.226.1-68.1.00 |

* depending on the atmosphere, the cell can potentially withstand 1650°C for short periods

¹ For sample carrier HTP40000A52.000-00

² For sample carrier HTP40000A58.000-00



Knudsen cells with different dimensions and orifice diameters

Crucibles for TGA Sample Carrier (HTP40000A52.000-00) of the STA 449 **F1/F3** Jupiter®

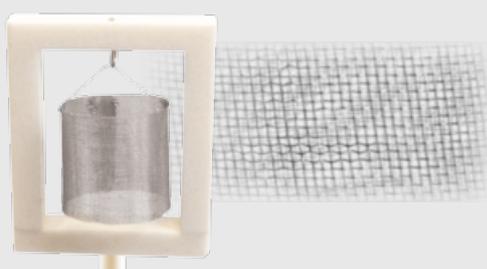
| Material | Temp. Range | Consisting of | Dimension/Volume | Remarks | Order Number |
|----------|-------------|---------------|------------------|---------|------------------|
| Tungsten | Max. 2400°C | Crucible | 0.3 ml | | NGB809001 |
| Graphite | Max. 2200°C | Crucible | 0.3 ml | | GB445333 |



Tungsten crucible (right) and graphite crucible (left)

Crucibles for TGA Sample Carrier (HTP40000A60.000-00) for Hanging Samples of the STA 449 F1/F3 Jupiter® – Cage

| Material | Temp. Range | Consisting of | Dimensions | Order Number |
|----------|--------------|------------------|--|------------------|
| Pt | Max. 1650°C | Cage, net-shaped | Cage ø 12 mm, height 12 mm, wire ø 0.04 mm, mesh width 0.12 mm | NGB813037 |
| Pt | Max. 1650° C | Cage, net-shaped | Cage ø 12 mm, height 12 mm, wire ø 0.06 mm, mesh width 0.25 mm | NGB813036 |



Crucibles for DTA Sensor¹ of the DSC 404 F1/F3 Pegasus® and for TGA², TGA-DTA³ Sensor of the STA 449 F1/F3/F5 Jupiter®

| Material (Purity/%) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|---------------------------------------|-------------------|------------------|------------------------------|---|------------------|
| Duran glass | Max. 600°C | Ampoule | Length after sealing ≈ 34 mm | | GB461185 |
| Fused silica | Max. 1000°C | Ampoule | Length after sealing ≈ 34 mm | | GB461177 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Slip-on plate | ø 10 mm | | GB343550 |
| Al ₂ O ₃ | Max. 1700°C | Crucible | 0.2 ml | | GB445172 |
| Al ₂ O ₃ | Max. 1700°C | Crucible | 0.3 ml | | NGB810411 |
| Al ₂ O ₃ | Max. 1700°C | Lid | | for NGB810411, GB445172; pierced, ø 0.5 mm hole | GB445175 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | 0.3 ml; inner ø 6 mm | sixfold pierced | NGB813974 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | 0.9 ml | | GB445215 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | for GB445215 | GB445216 |
| Pt/Ir | Max. 1600°C | Crucible | 0.3 ml | | GB280062 |
| Pt/Ir | Max. 1600°C | Lid for GB280062 | | | GB280066 |
| Graphite | Max. 2400°C | Crucible | 0.3 ml | > 2200°C evaporation may occur | GB399222 |
| Fine steel | Max. 600°C | Crucible | 0.3 ml | Only in inert atmosphere | GB440112 |
| Fine steel | Max. 600°C | Lid | | for GB440112 | GB440113 |

1 DTA sample carrier of the DSC 404 F1/F3 Pegasus®:

order no. DSC404F1AXX.000-00 (XX = 56, 57 or 68)

2 TGA sample carrier of the STA 449 F1/F3/F5 Jupiter®:

order no. HTP40000A58.000-00

3 TGA-DTA sample carrier of the STA 449 F1/F3/F5 Jupiter®:

order no. HTP40000AXX.000-00 (XX = 56, 68, 51, 50 or 65)



Ampoules for TGA and TGA-DTA measurements

TGA-DTA crucibles and slip-on plates



TGA-DTA, DSC and TGA-DSC Crucibles

Crucibles for TGA-DTA Sensor (HTP40000A53.000-00) of the STA 449 **F1/F3** Jupiter® and DSC 404 **F1/F3** Pegasus® (DSC404F1A53.000-00)

| Material | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|--------------------------------|-------------------|---------------|-------------------|--|------------------|
| Tungsten | Max. 2400°C | Crucible | 80 µl | Wall thickness 0.3 mm, only for STA 449 F3 Jupiter® | GB453520 |
| Tungsten | Max. 2400°C | Crucible | 80 µl | Wall thickness 0.2 mm, BN liner available | NGB808999 |
| Tungsten | Max. 2400°C | Lid | | Pierced, for NGB808999 | NGB809406 |
| BN ¹ | Max. 1600°C | Liner | 80 µl | For NGB808999 | NGB805943 |
| Glassy carbon | Max. 2000°C | Crucible | 60 µl | Up to 500°C oxidizing | GB395116 |
| Graphite | Max. 2400°C | Crucible | 0.15 ml | | GB427607 |
| Graphite | Max. 2400°C | Lid | | Pierced, for GB427607 | NGB814940 |
| Al ₂ O ₃ | Max. 1700°C | Crucible | 0.15 ml | | GB399147 |
| Al ₂ O ₃ | Max. 1700°C | Lid | | Pierced lid for GB399147 | GB399973 |
| ZrO ₂ | Max. 2000°C | Crucible | 0.15 ml | | GB399148 |



TGA-DTA crucibles for highest temperatures

Crucibles for All DSC and TGA-DSC Sensors of the STA 449 **F1/F3/F5** Jupiter® and DSC 404 **F1/F3** Pegasus®

| Material (Purity/%) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|---------------------------------------|-------------------|--|-------------------|--|------------------------|
| PtRh/Al ₂ O ₃ | Max. 1700°C | Crucible + liner + lid | | For metal melts and other reactive materials | 6.225.6-93.2.00 |
| Al ₂ O ₃ | Max. 1700°C | Crucible liner (spare for 6.225.6-93.2.00) | | | 6.225.6-93.1.00 |
| Yttriumoxid-Spray* | - | Content 360g | - | Coating of crucibles for increased oxidation stability | NGB804075 |
| MgO | Max. 1500°C | Crucible liner (spare for GB399205) | | Minimum sale 10 pieces | NGB812638 |
| Y ₂ O ₃ | Max. 1700°C | Crucible liner (spare for GB399205) | | Minimum sale 10 pieces | NGB812634 |
| Tool | | | | For reshaping of Pt crucibles | GB397964 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Crucible | ø 6.8 mm / 85 µl | | GB399972 |
| Al ₂ O ₃ (99.7) | Max. 1700°C | Lid | | For GB399972 | GB399973 |
| Duran glass | Max. 600°C | Ampoule | 80 µl | Length after sealing ≈19 mm | 6.240.1-93.2.00 |
| Fused silica | Max. 1000°C | Ampoule | 80 µl | Length after sealing ≈19 mm | NGB815223 |

* Hazardous material, shipment will cause additional costs.

DSC and TGA-DSC crucible variety



Crucibles for All DSC and TGA-DSC Sensors of the STA 449 F1/F3/F5 Jupiter® and DSC 404 F1/F3 Pegasus® (Continuation)

| Material (Purity/%) | Temperature Range | Consisting of | Dimension/ Volume | Remarks | Order Number |
|--------------------------------|-------------------|----------------|------------------------|--|------------------------|
| Fused silica | Max. 1000°C | Crucible | ø 6.7 mm / 85 µl | | GB399974 |
| Fused silica | Max. 1000°C | Lid | | For GB399974 | GB399975 |
| Pt/Rh (80/20) | Max. 1700°C | Crucible | ø 6.8 mm / 85 µl | | GB399205 |
| Pt/Rh (80/20) | Max. 1700°C | Crucible | ø 6.8 mm / 0.19 ml | Height 6 mm | NGB801556 |
| Pt/Rh (80/20) | Max. 1700°C | Lid | | For GB399205 and NGB801556 | GB399860 |
| Al (99.5) | Max. 600°C | Concavus pan* | ø 5 mm / 30/40 µl | | NGB814672 |
| Al (99.5) | Max. 600°C | Concavus lid | | | NGB814673 |
| Al (99.5) | Max. 600°C | Crucible | ø 6.7 mm / 85 µl | Set of 100 pieces | NGB810405 |
| Al (99.5) | Max. 600°C | Lid | | For NGB810405 | NGB810406 |
| Gold (99.9) | Max. 900°C | Crucible + lid | ø 6.7 mm / 85 µl | | 6.225.6-93.3.00 |
| Ag | Max. 750°C | Crucible + lid | ø 6.7 mm / 85 µl | | 6.225.6-93.4.00 |
| ZrO ₂ | Max. 2000°C | Crucible | 85 µl | CaO-stabilized | GB397053 |
| ZrO ₂ | Max. 2000°C | Lid | | For GB397053 | GB397052 |
| Graphite | Max. 2400°C | Crucible | 85 µl | >2200°C evaporation may occur | GB399956 |
| Graphite | Max. 2400°C | Lid | | For GB399956 | GB399957 |
| Glassy carbon | Max. 2400°C | Crucible | ø 6 mm / 50 µl | | NGB811386 |
| Glassy carbon | Max. 2400°C | Lid | | For NGB811386 | NGB812613 |
| Al ₂ O ₃ | Max. 1700°C | Washer | ø 6.8 mm, thick 0.2 mm | Inhibits sticking of Pt onto sensor type S and B | NGB811071 |
| MgO | Max. 1500°C | Crucible | ø 6.7 mm, 85 µl | Minimum sale 10 pieces | NGB812639 |
| MgO | Max. 1500°C | Lid | | For NGB812639, min. sale 10 pieces | NGB812637 |
| Y ₂ O ₃ | Max. 1700°C | Crucible | ø 6.7 mm, 85 µl | Minimum sale 10 pieces | NGB812636 |
| Y ₂ O ₃ | Max. 1700°C | Lid | | For NGB812636, min. sale 10 pieces | NGB812635 |
| BN ¹ | Max. 1600°C | Crucible | ø 6.7 mm, 85 µl | Inert gas required | NGB808836 |
| BN ¹ | Max. 1600°C | Lid | | For NGB808836 | NGB808835 |
| Mo | Max. 2000°C | Crucible | ø 6.8 mm, 85 µl | | NGB808165 |

¹ Maximum temperature when in contact with Pt, W and Rh or Pt/Rh alloys: 1600°C. For other materials getting in contact with BN, reactions have to be avoided which might damage crucible or liner. In the presence of He, Ar or vacuum, tungsten embrittles and hardens in contact with BN.

* Using *Concavus* pans with ASC instrument which were delivered before 2014 may require re-adjustment of the ASC gripper.



Crucibles for Operation with the Automatic Sample Changer (ASC)

| Crucibles for STA 449 F1/F3 & DSC 404 F1/F3 in ASC Operation | | | | |
|--|---|------------------------------------|---|-------------------------|
| Crucible Material (Purity/%), Volume Lid | ASC Gripper STA 449 F1/F3 DSC 404 F1/F3 | TGA-DSC Sensor* DSC Sensor** | TGA Sensor ² HTP40000A67.000-00 HTP40000A75.000-00 | Order Number |
| Al (99.5), 30/40 µl (Concavus pan) | - ³ | x | x | DSC214A66.010.00 |
| Crucible with welded lid | - ³ | x | x | |
| Al (99.5), 25/40 µl | - ¹ | x | x | 6.239.2-64.5.00 |
| Crucible with welded lid | - | x | x | |
| Al (99.5), 25/40 µl (500/pkg.) | x | x | x | 6.239.2-64.51.00 |
| Crucible with welded lid | x | x | x | |
| Al (99.5), 25/40 µl | - ¹ | x | x | 6.239.2-64.8.00 |
| Crucible and lid with laser-cut-hole | - | x | x | |
| Al (99.5), 25/40 µl (500/pkg.) | x | x | x | 6.239.2-64.81.00 |
| Crucible and lid with laser-cut-hole | x | x | x | |
| Al (99.5), low-pressure, 35 µl | - ¹ | x | x | 6.240.10-65.1.00 |
| Crucible with welded lid | - | x | x | |
| CrNi steel (AISI 302), medium-pressure, 120 µl | x | x | x | 6.240.1-68.1.00 |
| Crucible with lid | x | x | x | |
| Copper, 25 µl | - | x | x | 6.239.2-64.6.00 |
| CrNi steel (AISI 316L), high-pressure, 27 µl, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.3.00 |
| CrNi steel (AISI 316L), high-pressure, 27 µl, 100 bar | x | x | Only for manual operation | 6.239.2-93.3.00 |
| CrNi (AISI 316L), high-pressure, 100 µl, gold-plated, 100 bar | x | x | Only for manual operation | 6.239.2-92.8.00 |
| CrNi (AISI 316L), high-pressure, 100 µl, 100 bar | x | x | Only for manual operation | 6.239.2-93.4.00 |
| Ti (Grade 4), high-pressure, 27 µl, 100 bar | x | x | Only for manual operation | 6.239.2-92.6.00 |
| Ti (Grade 4), high-pressure, 100 µl, 100 bar | x | x | Only for manual operation | 6.239.2-92.9.00 |
| Al ₂ O ₃ (99.7), ø 6.8 mm, 85 µl | x | x | x | GB399972 |
| Crucible with lid (note: lid order no.) | x | x | x | GB399973 |
| Fused silica, ø 6.8 mm, 85 µl | x | x | x | GB399974 |
| Crucible with lid (note: lid order no.) | x | x | x | GB399975 |
| Pt/Rh (80/20), ø 6.8 mm, 85 µl | x | x | x | GB399205 |
| Crucible with lid (note: lid order no.) | x | x | x | GB399860 |
| Pt/Rh (80/20), ø 6.8 x 6 mm, 190 µl | x | x | x | NGB801556 |
| Crucible with lid (note: lid order no.) | x | x | x | GB399860 |

| Crucible Material (Purity/%), Volume Lid | ASC Gripper STA 449 F1/F3 DSC 404 F1/F3 | TGA-DSC Sensor* DSC Sensor** | TGA Sensor ² HTP40000A67.000-00 HTP40000A75.000-00 | Order Number |
|---|---|------------------------------------|---|-----------------|
| Al (99.5), ø 6.7 mm, 85 µl | x | x | x | NGB810405 |
| Crucible with lid (note: lid order no.) | x | x | x | NGB810406 |
| Graphite, ø 6.8mm, 85 µl | x | x | x | GB399956 |
| Crucible with lid (note: lid order no.) | x | x | x | GB399957 |
| Gold (99.9), ø 6.8 mm, 85 µl | x | x | x | 6.225.6-93.3.00 |
| Crucible with lid | x | x | x | |
| Silver, ø 6.8 mm, 85 µl | x | x | x | 6.225.6-93.4.00 |
| Crucible with lid | x | x | x | |
| ZrO ₂ , ø 6.8 mm, 85 µl | x | x | x | GB397053 |
| Crucible with lid (note: lid order no.) | x | x | x | GB397052 |
| Y ₂ O ₃ , ø 6.8 mm, 85 µl | x | x | x | NGB812636 |
| Crucible with lid (note: lid order no.) | x | x | x | NGB812635 |
| MgO, ø 6.8mm, 85 µl | x | x | x | NGB812639 |
| Crucible with lid (note: lid order no.) | x | x | x | NGB812637 |
| Al ₂ O ₃ (99.7), ø 8 x 8 mm, 300 µl | x | - | x | NGB803698 |
| Crucible with lid (note: lid order no.) | x | - | x | NGB808209 |
| Pt/Rh/Al ₂ O ₃ system with lid | x | x | x | 6.225.6-93.2.00 |
| Mo, ø 6.8 mm, 85 µl | x | x | - | NGB808165 |
| BN, ø 6.7 mm, 85 µl | x | x | only for manual operation | NGB808836 |
| Crucible with lid (note: lid order no.) | - | x | - | NGB808835 |

1 Special gripper for Al crucibles on request

2 For the listed crucibles, the TGA-Sensor requires the slip-on plate made of Al₂O₃, order no. NGB809010

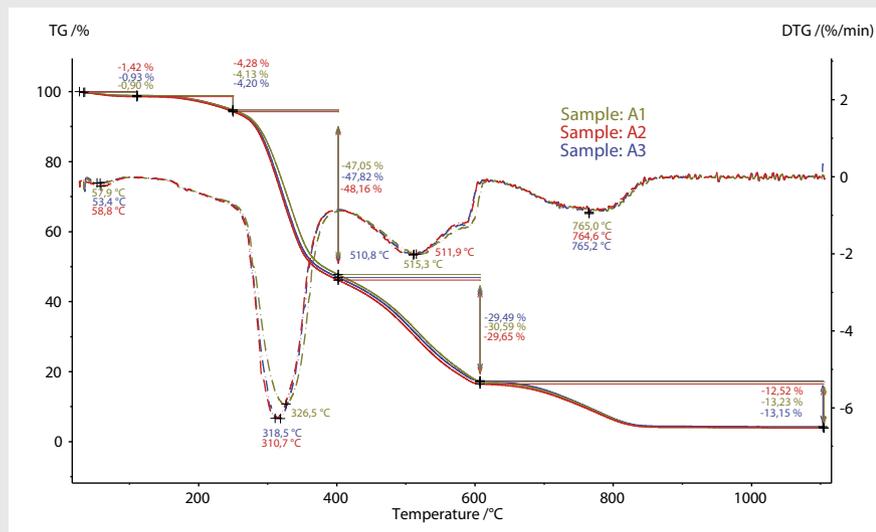
3 Using *Concavus* pans with ASC require re-adjustment of ASC gripper.

* TGA-DSC Sensor: HTP40000A**XX**.000-00 (**XX** = 63, 66, 73 or 74)

** DSC Sensor: DSC404F1A**XX**.000-00 (**XX** = 63, 66, 73 or 74)



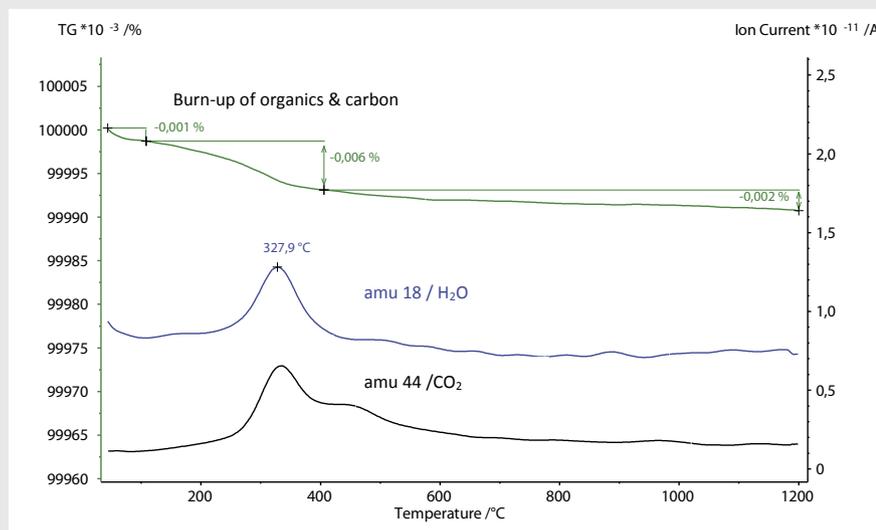
Applications – Special Crucibles for Large Samples, Oxygen-Sensitive Alloys



A ceramic mass (approx. 130 mg) was tested here in the alumina beaker crucible for TGA measurements (heating rate 10 K/min, synthetic air atmosphere)

Test on a Fire Insulating Material

For TGA measurements on inhomogeneous materials or ones with low densities (e.g., foams), very large crucibles are available. The example shows three samples of a heavily foaming fire insulation material. It can clearly be seen that these three samples exhibit identical TGA behavior. Therefore, no significant fluctuations in the composition of the lot are to be expected.



Quartz sand (4099.46 mg) was tested here in the alumina beaker crucible for TGA measurements

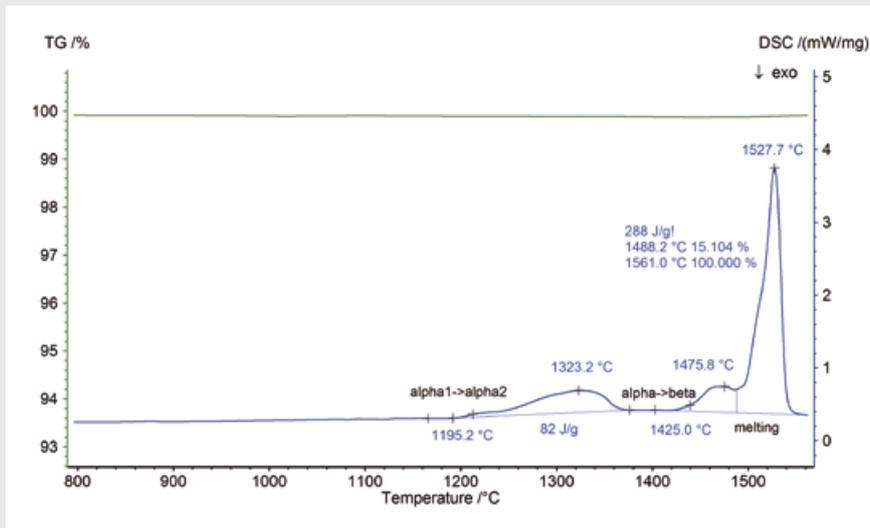
Burn-Up of Organics and Carbon

Resolution for the smallest of TGA steps can be achieved by increasing the sample mass. In this example, quartz sand was heated at a rate of 20 K/min in synthetic air. The results demonstrate that extremely small mass-loss steps can be resolved by using a sample mass of more than 4000 mg. The evolved gases in this example were detected using our QMS 403 *Aëolos*®. The second mass-loss step amounts to 0.006% and is mainly due to the burn-up of organics and carbon (CO₂: u 44 and water: u 18).

Please note:

In applications where even larger crucibles are required, we recommend our Multiple-Module Calorimeter, MMC 274 *Nexus*®. This tabletop instrument bridges the gap between large adiabatic reaction calorimeters and small differential scanning calorimeters. It allows for large sample volumes, while quickly and precisely analyzing properties such as heat capacities. It is also capable of quantitatively characterizing exothermic and endothermic effects.





Phase transitions in γ -TiAl. Only a pure oxygen-free atmosphere leads to well-defined phase transitions at higher temperatures because oxidation of the metal alloy is inhibited.

Phase Transformation of an Oxygen-Sensitive Material

The high-performance metal γ -TiAl is resistant to corrosion and high temperatures and has a low specific weight. However, it is highly sensitive to oxygen, so DSC measurements have to be carried out in a very pure atmosphere. Additionally, measurement results can be improved by using the right crucible. Metallic crucibles with high thermal conductivities and low time constants cannot be used for metals. In order to maintain the advantages of a Pt crucible and to prevent reaction between the sample and crucible material, a special Pt crucible with an Al_2O_3 liner is available. The DSC 404 **F3 Pegasus**[®], with its vacuum-tight design and various sensor and crucible types, completely fulfills all measurement requirements for highly oxygen-sensitive materials. In addition, an oxygen trap system (OTS[®]) is available to further reduce oxygen traces in the atmosphere.

The DSC curve of γ -TiAl shows an endothermic effect at a peak temperature of 1323°C. This is due to the $\alpha_2 \rightarrow \alpha_1$ transition. At 1476°C, the $\alpha \rightarrow \beta$ transition occurs and is followed by melting of the sample.



The crucible combination of PtRh/ Al_2O_3 (order no. 6.225.6-93.2.00) can be used for samples which may react with Pt up to 1700°C. In addition to the Al_2O_3 liner, MgO and Y_2O_3 liners are also available.

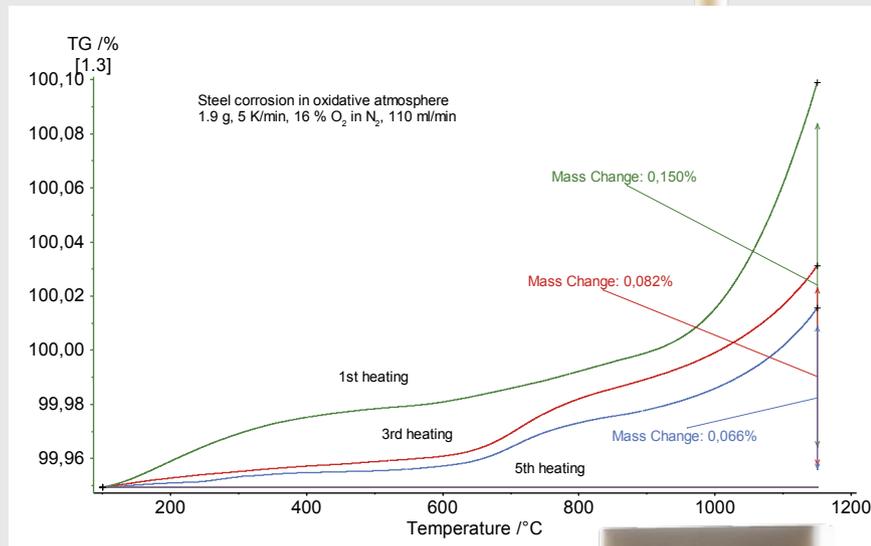
Furthermore, these crucible combinations are suitable for c_p determination on metals or other oxygen-sensitive materials up to 1400°C.

The reshaping tool for the PtRh crucible (order no. GB397964) can be used to reshape it after measurement, allowing it to be used multiple times.

Applications – Special Crucibles for High Temperatures

Oxidation and Corrosion Studies on Metals

For oxidation and corrosion studies, it is preferable to have a large sample surface in order to maximize the gas access. TGA and TGA-DTA sample holders are available which allow the sample to be attached in a hanging position (TGA: HTP40000A60.000-00; TGA-DTA: HTP40000A61.000-00). The samples can be a plate, mesh, or a compact body which can be encased in a platinum basket.

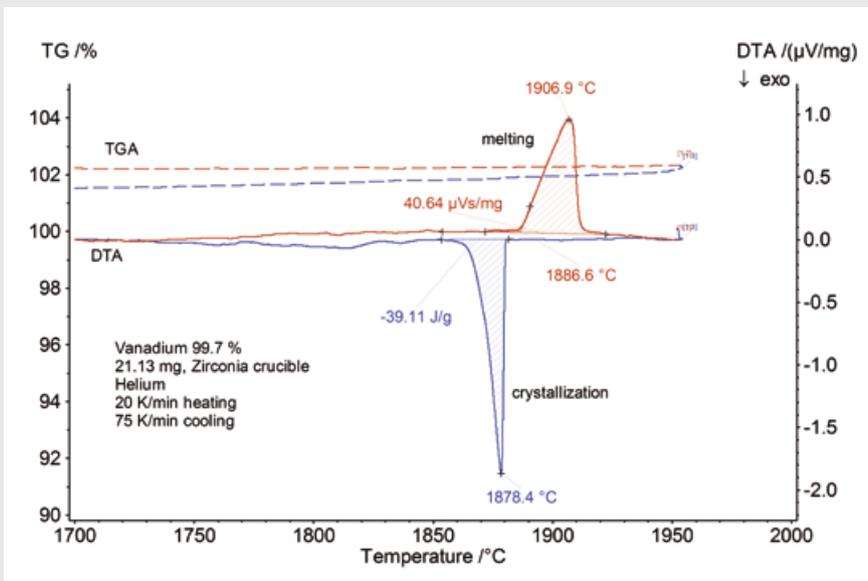


Increased sample surface for oxygen access improves oxidation tests



Steel Corrosion

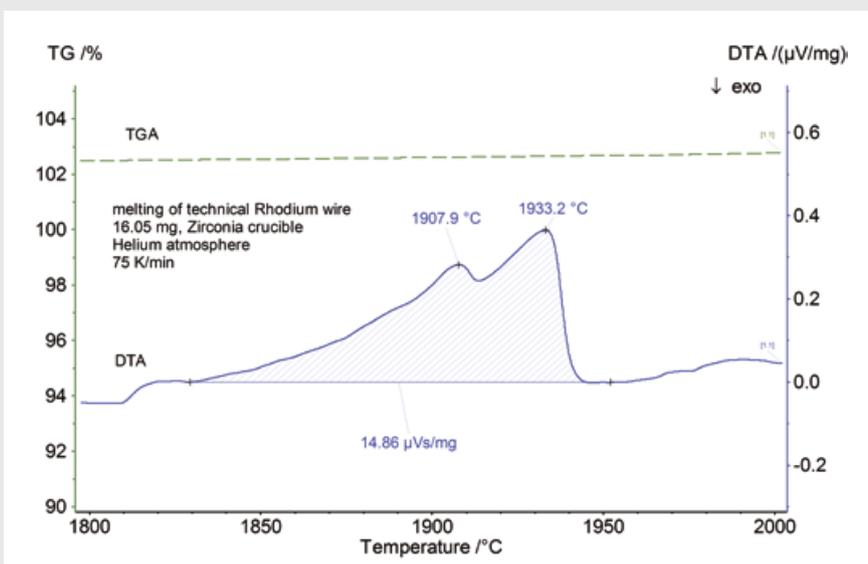
This TGA measurement shows several heating cycles on a hanging steel sample. The steel sheet was heated at a rate of 5 K/min in a nitrogen atmosphere with 16% oxygen. Oxidation (mass increase) decreases with each subsequent heating cycle. At the beginning of the test, oxidation of the sheet surface occurs. This can be observed in the early onset and rapid mass increase for the first heating. After a couple of heating cycles, inner oxidation occurs, which is indicated by a slower, diffusion-dependent mass increase. Hanging the sample in the special holder maximizes the accessible sample surface and therefore improves oxygen access to all sample sides. This is a prerequisite for certain analyses, such as kinetic studies of the oxidation behavior.



Vanadium melting point determination in ZrO₂ crucibles

Melting Point Determination on Vanadium

In this example, the melting point of a vanadium sample (99.7%) was determined with the STA 449 **F1 Jupiter**[®]. ZrO₂ crucibles were used. Melting occurs at an onset temperature of 1886°C. During cooling, the sample recrystallizes with only a small supercooling effect at 1878°C.



Double melting peak of a technical rhodium wire

Technical Rhodium Wire – Failure Analysis at High Temperatures

Here, a failure analysis on a rhodium wire was carried out with the STA 449 **F1 Jupiter**[®]. The wire material was measured in ZrO₂ crucibles up to 2000°C in a helium atmosphere. The broad double peak and temperatures (1908°C and 1933°C) confirm that the wire material is not made of pure rhodium.

Sensors and Sample Carriers



DSC, TGA-DSC Sensors and TGA Sample Carriers

Nearly all DSC, TGA and STA instruments allow measurements with exchangeable DSC and DTA or TGA, TGA-DTA and TGA-DSC sensors and sample carriers which cover different temperature ranges. Additionally, the instruments differ in the types of thermocouple which are compatible with them, as well as their ability to accommodate large crucibles. The type of thermocouple selected influences the sensitivity and resolution of a given DSC or STA system.

Choosing the right thermocouple can thus improve the measurement curve and also the evaluated test results, so this should be as carefully considered as the temperature range.

The following pages give an overview of the DTA, DSC, TGA, TGA-DSC and TGA-DTA sensors and sample carriers for these instruments:

- DSC 204 **F1 Phoenix**[®]
- DSC 404 **F1/F3 Pegasus**[®]
- STA 449 **F1/F3/F5 Jupiter**[®]
- TG 209 **F3 Tarsus**[®]
- TG 209 **F1 Libra**[®]



DSC 204 **F1** Phoenix[®] Sensors, TG 209 **F1** Iris[®]/Libra[®] and TG 209 **F3** Tarsus[®] Sample Carriers

DSC 204 F1 Phoenix[®]

| Sensor Type* | Temperature Range | Remarks | Order Number |
|----------------|-------------------|---|-------------------------|
| τ -sensor | -180°C to 700°C | Complete system, ready for installation → High-resolution sensor | 6.240.10-09.0.00 |
| μ -sensor | -150°C to 400°C | Complete system, ready for installation → High-sensitivity sensor | 6.240.10-11.0.00 |

* From instrument series no. 240 10 034 on

τ -sensor with low time constant and very good sensitivity for standard applications

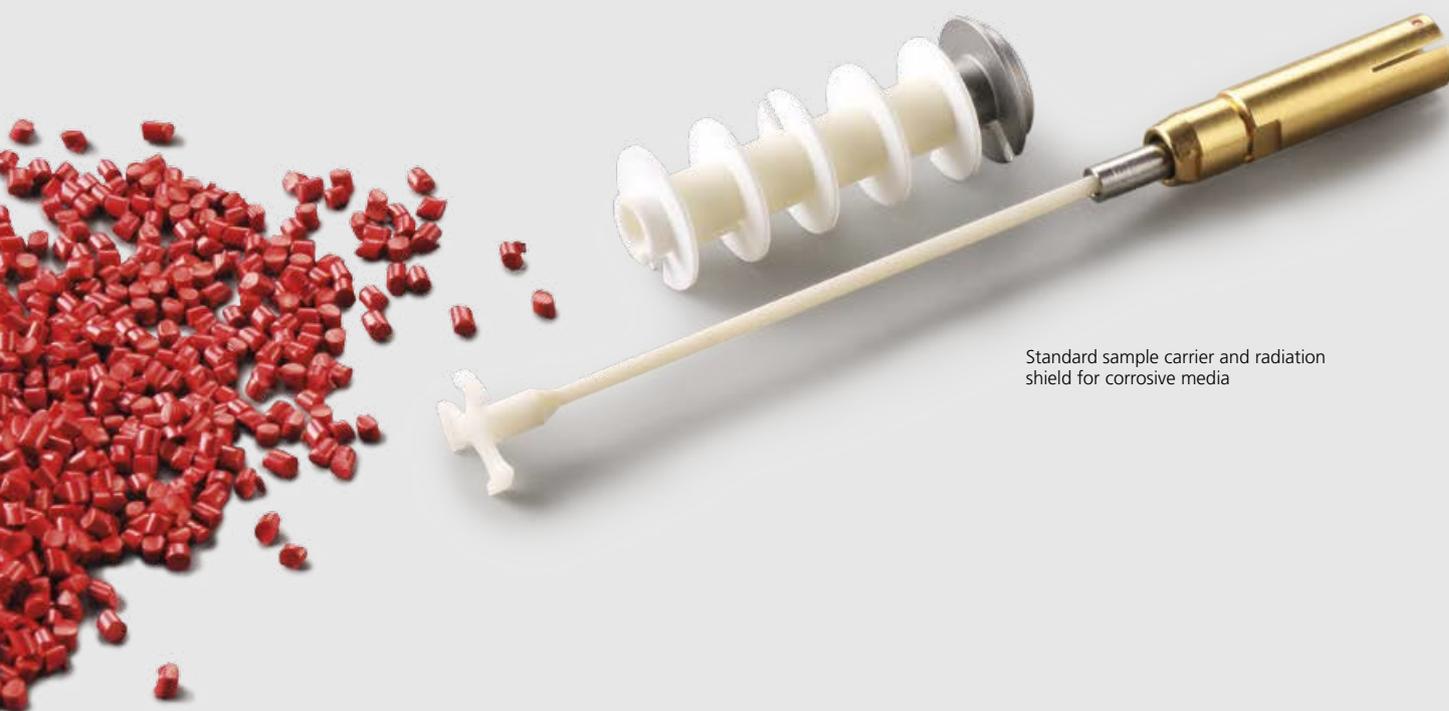


μ -sensor with extremely high sensitivity



TG 209 F1 Iris[®] – Up to Instrument number 220 10 76

| Sample Carrier Material | Temperature Sensor Type | Remarks | Order Number |
|--------------------------------|-------------------------|--|-------------------------|
| Al ₂ O ₃ | Type P | SiO ₂ -coated, up to instrument No. 220 10 031 | 6.220.10-57.1.00 |
| Al ₂ O ₃ | Type P | SiO ₂ -coated, from instrument No. 220 10 032 on | 6.220.10-57.5.00 |
| Al ₂ O ₃ | Type P | SiO ₂ -coated, for large samples, up to instrument No. 220 10 031 | 6.220.10-58.1.00 |
| Al ₂ O ₃ | Type P | SiO ₂ -coated, for large samples, from instrument No. 220 10 032 on | 6.220.10-58.5.00 |
| Radiation shield | | Made of Al ₂ O ₃ , complete with metallic flange | 6.220.10-04.4.00 |



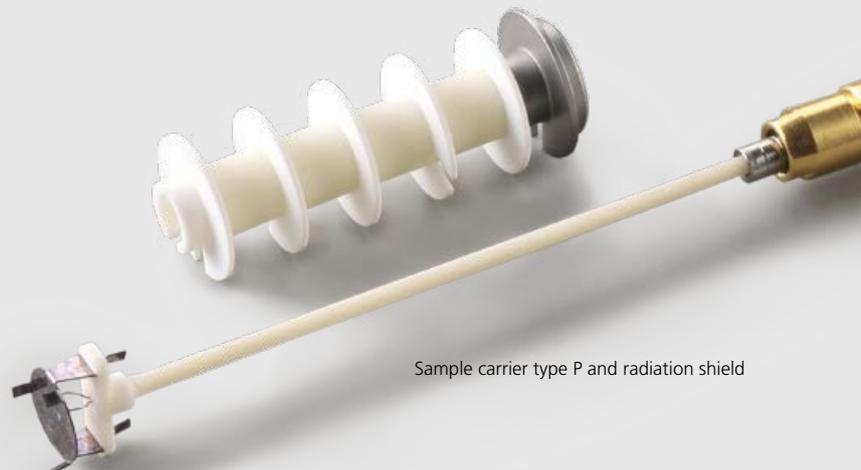
Standard sample carrier and radiation shield for corrosive media

TG 209 **F1 Iris**® (from Instrument Number ≥ 220 10 077*) / TG 209 **F1 Libra**® and TG 209 **F3 Tarsus**®**

| Sample Carrier Material | Temperature Sensor Type | Remarks | Order Number |
|--------------------------------|-------------------------|--|---------------------------|
| Al ₂ O ₃ | Type P | Higher sensitivity for c-DTA®, with SiO ₂ coating, including radiation shield | 6.220.10-60.0.00** |
| Al ₂ O ₃ | Type P | Higher sensitivity for c-DTA®, with SiO ₂ coating, without radiation shield | 6.220.10-60.1.00 |
| Al ₂ O ₃ | Type P | Higher sensitivity for c-DTA®, for large samples, including radiation shield | 6.220.10-61.0.00** |
| Al ₂ O ₃ | Type P | Higher sensitivity for c-DTA®, with SiO ₂ coating, without radiation shield | 6.220.10-61.1.00 |
| Al ₂ O ₃ | Type P | Sample support made of Al ₂ O ₃ , including radiation shield | 6.220.10-62.0.00** |
| Al ₂ O ₃ | Type P | Sample support made of Al ₂ O ₃ , without radiation shield | 6.220.10-62.1.00 |
| Al ₂ O ₃ | Type P | For large samples, sample supports made of Al ₂ O ₃ , radiation shield | 6.220.10-63.0.00 |
| Al ₂ O ₃ | Type P | For large samples, sample supports made of Al ₂ O ₃ , without radiation shield | 6.220.10-63.1.00 |
| Al ₂ O ₃ | Type P | For crucible up to an outer diameter of 9 mm, with protected thermocouple, particularly suitable for corrosive media, including radiation shield and crucible made of Al ₂ O ₃ with ø 8 mm (0.3 ml, order no. NGB803698) | 6.220.10-66.0.00** |
| Al ₂ O ₃ | Type P | For crucible up to an outer diameter of 9 mm, with protected thermocouple, particularly suitable for corrosive media | 6.220.10-66.1.00 |
| Al ₂ O ₃ | Type P | Standard sample carrier with closed stuck head plate, particularly suitable for corrosive media, including radiation shield | 6.220.10-65.0.00** |
| Radiation shield | | Made of Al ₂ O ₃ , for TG 209 F1 Libra ®, from instrument: TG 209 F1 Iris ® no. 77 up to 158, TG 209 F3 Tarsus ® no. 010 up to 048 | 6.220.10-60.2.00** |
| Radiation shield | | Made of Al ₂ O ₃ , for TG 209 F1 Libra ®, from instrument TG 209 F1 Iris ® no. 77 up to 159; for TG 209 F3 Tarsus ® from no. 049 onwards | 6.220.10-60.3.00** |

* Please contact us for sample carriers for instrument number 1 to 76.

** also for TG 209 **F3 Tarsus**®



Sample carrier type P and radiation shield

c-DTA[®] – More Than an Easy Calibration Routine

c-DTA[®] – Temperature Calibration and Determination of Caloric Effects in TGA Measurements

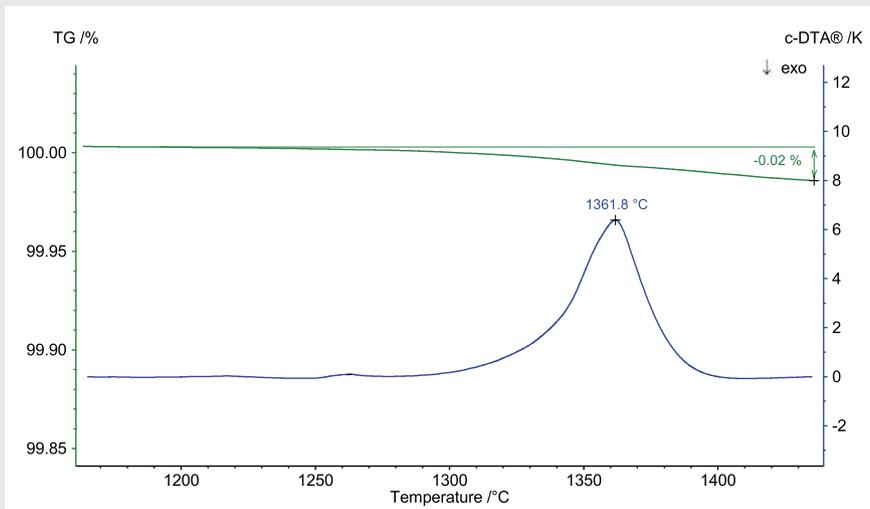
The calculated DTA/DSC-signal, c-DTA[®], is ideal for easy temperature calibration without the need for magnetic Curie point standards, which would often necessitate partial disassembling of the thermobalance. In addition, signals of mass change together with endo- and exothermal behaviors (e.g., vaporization with mass loss or melting without mass change) can be obtained without any hardware add-ons.

Thus, correlation of such results is not influenced by the hardware.

All TGA sample carriers (TG 209 **F1 Iris**[®]/**Libra**[®], TG 209 **F3 Tarsus**[®], STA 449 **F1/F3 Jupiter**[®]) allow for c-DTA[®] determination.

Ceramic and metallic crucibles are available to achieve optimum peak temperature results together with the caloric information on the tested sample.



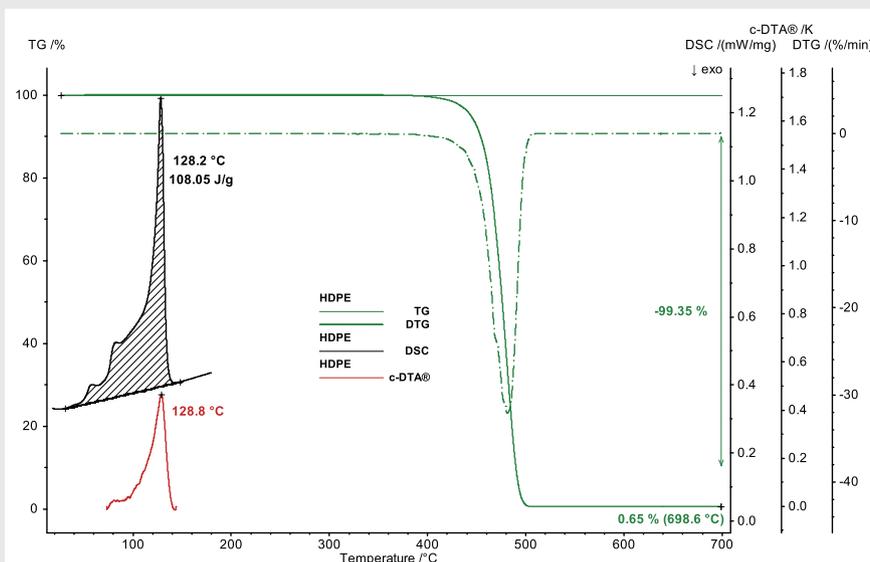


Measurements of large sample masses can be done with slip-on plates. Various dimensions of such plates are available.

Thermal Behavior of a Large Steel Sample

In this example, a large steel sample (1018.59 mg) was tested with the STA 449 **F1 Jupiter**® at a heating rate of 20 K/min in an argon atmosphere. A slip-on plate made of alumina (ø 17 mm) was used to carry the sample.

The c-DTA® curve (blue) shows an endothermic effect at 1362°C which corresponds to melting of the sample. In the same temperature range, the TGA curve (green) indicates some minor mass loss (0.02%) which is due to a small evaporation effect.



Very good correlation between calculated and measured melting peak temperature

Melting and Decomposition of HDPE with the TG 209 **F1 Libra**®

High density polyethylene (HDPE) was here measured with the TG 209 **F1 Libra**® at a heating rate of 10 K/min in an N₂ atmosphere. The sample mass amounted to 10.31 mg.

Before decomposition started at approx. 420°C (green), the c-DTA® curve (red) detected the melting peak at 128.8°C. Under the same conditions, the melting peak temperature of HDPE was determined with the DSC 200 **F3 Maia**® (black curve). As can be observed, the two sets of results are in perfect agreement.

DTA and DSC Sensors

DTA Sensors for the DSC 404 **F1/F3** Pegasus®

| Sensor Type | Temperature Range | Crucible/Volume/Order Number | Radiation Shield | Order Number |
|---------------------------------|-------------------|---|------------------|---------------------------|
| Type S | 25°C to 1650°C | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Integrated | DSC404F1A56.000-00 |
| Type S, protected thermocouples | 25°C to 1650°C | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Integrated | DSC404F1A68.000-00 |
| Type B | 25°C to 1750°C | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Integrated | DSC404F1A57.000-00 |
| W3%Re/W25%Re (exchangeable) | 25°C to 2000°C | 2x tungsten, GB453520 | Integrated | DSC404F1A53.000-00 |

DSC Sensors for the DSC 404 **F1/F3** Pegasus® – for Standard & c_p Measurements*

| Sensor Type | Temperature Range | Crucible/Volume/Order Number | Radiation Shield | Order Number |
|--------------------------|--|--|------------------|---------------------------|
| Type S, octagonal design | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A54.000-00 |
| Type P | -150°C to 1000°C | 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A59.000-00 |
| Type E | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A70.000-00 |
| Type K | -150°C to 800°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A71.000-00 |
| Type S | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A72.000-00 |
| Type B | 25°C to 1750°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A76.000-00 |
| Type K, octagonal | -150°C to 800°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A77.000-00 |
| Type S, octagonal | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A78.000-00 |
| Type E, octagonal | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A79.000-00 |

* c_p recommended up to 1400°C

DSC Sensors for the DSC 404 F1/F3 Pegasus® – for ASC Operation, Octagonal Design

| Sensor Type | c_p | Temperature Range | Crucible/Volume/Order Number | Radiation Shield | Order Number |
|-------------------------|-------|--|--|------------------|---------------------------|
| Type S, round design | Yes | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A73.000-00 |
| Type P | Yes | -150°C to 1000°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A75.000-00 |
| Type K | Yes | 150°C to 800°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Integrated | DSC404F1A74.000-00 |



DTA sensor
with protected
thermocouples



DSC sensor in round
design with Pt crucibles



DSC sensor



DSC sensor in
octagonal design
for ASC
operation

TGA Sample Carriers and TGA-DTA Sensors

TGA Sample Carriers for the STA 449 **F1/F3** Jupiter® – for Standard Measurements

| Material/Sensor Type | Temperature Range | Crucible/Volume/Order Number | Radiation Shield | Order Number |
|---|-------------------|---|------------------|---------------------------|
| Type P | -150°C to 1000°C | Al ₂ O ₃ , 3.4 ml, GB445213 | Included | HTP40000A55.000-00 |
| Type P | -150°C to 1000°C | - | Without | HTP40000A55.010-00 |
| Type S | 25°C to 1650°C | Al ₂ O ₃ , 3.4 ml, GB445213 | Included | HTP40000A58.000-00 |
| Type S | 25°C to 1650°C | - | Without | HTP40000A58.010-00 |
| Made of Al ₂ O ₃ , W3%Re/W25%Re | 25°C to 1650°C | Al ₂ O ₃ , 3.4 ml, GB445213 | Included | HTP40000A62.000-00 |
| Made of Al ₂ O ₃ , W3%Re/W25%Re | 25°C to 1650°C | - | Without | HTP40000A62.010-00 |
| Made of fused silica, type S | 25°C to 1100°C | Crucible required: Fused silica, 3.4 ml, NGB811507 | Included | HTP40000A64.000-00 |
| Made of fused silica, type S | 25°C to 1100°C | - | Without | HTP40000A64.010-00 |
| W3%Re/W25%Re | 25°C to 2400°C | Tungsten, NGB809001 | Included | HTP40000A52.000-00 |
| W3%Re/W25%Re | 25°C to 2400°C | - | Without | HTP40000A52.010-00 |
| Radiation shield from tungsten | 25°C to 2400°C | - | Complete | HTP40000A53.020-00 |



TGA type S sample carrier with different crucibles and plates, TGA type W sample carrier (W3%Re/W25%Re); for more crucibles and plates, see TGA crucibles table.

TGA Sample Carriers and TGA-DTA Sensors for the STA 449 F1/F3 Jupiter® – with Special Design

| Material/Sensor Type | Temperature Range | Crucible Type | Radiation Shield | Order Number |
|--|-------------------|--|---|---------------------------|
| TGA, type S, length 250 mm, for water vapor furnace HTP40000A88 | 25°C to 1650°C | Al ₂ O ₃ slip-on plate, ø 17 mm | Included | HTP40000A57.000-00 |
| TGA, type S, length 250 mm, for water vapor furnace HTP40000A88 | 25°C to 1650°C | - | Without | HTP40000A57.010-00 |
| TGA, made of Al ₂ O ₃ , type S, for hanging samples, sample size max. 13 x 20 mm | 25°C to 1650°C | Options: net-shaped cage made of Pt, order no. NGB813036 or order no. NGB813037 (see page 29) | Included | HTP40000A60.000-00 |
| TGA, made of Al ₂ O ₃ , type S, for hanging samples, sample size max. 13 x 20 mm | 25°C to 1650°C | | Without | HTP40000A60.010-00 |
| TGA, made of Al ₂ O ₃ , type S, for measurements in an immersion bath (e.g., salt solution), for hanging samples | 25°C to 1650°C | Special container made of Al ₂ O ₃ , 3 immersion container made of Pt and 5 immersion container made of BN | tube made of glass (single order no. NGB801706) | HTP40000B60.000-00 |
| TGA-DTA, type S, hanging samples | 25°C to 1650°C | | Included | HTP40000A61.000-00 |
| TGA-DTA, type S, hanging samples | 25°C to 1650°C | | Without | HTP40000A61.010-00 |



TGA-DTA sensor for hanging samples



TGA sample carrier for hanging samples



TGA sample carrier for measurements in an immersion bath

TGA-DTA and TGA-DSC Sensors

TGA-DTA Sensors for the STA 449 **F1/F3/F5** Jupiter® – for Standard Measurements

| Sensor Type | Temperature Range/ Atmosphere | Crucible/Volume/Order Number | Radiation Shield | Order Number |
|------------------------------------|--|---|------------------|---------------------------|
| Type E | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Included | HTP40000A50.000-00 |
| Type E | -150°C to 700°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A50.010-00 |
| Type K | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Included | HTP40000A51.000-00 |
| Type K | -150°C to 700°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A51.010-00 |
| Type P | 25°C to 1000°C | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Included | HTP40000B56.000-00 |
| Type S | 25°C to 1650°C | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Included | HTP40000A56.000-00 |
| Type S | 25°C to 1650°C | - | Without | HTP40000A56.010-00 |
| Type S, protected thermocouples | 25°C to 1650°C | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Included | HTP40000A68.000-00 |
| Type S, protected thermocouples | 25°C to 1650°C | - | Without | HTP40000A68.010-00 |
| Type W, protected thermocouples | 25°C to 1500°C | - | Included | HTP40000A94.000-00 |
| W3%Re/W25%Re | 25°C to 1650°C, not for oxidizing atmospheres! | 2x Al ₂ O ₃ , 0.2 ml, NGB810411 | Included | HTP40000A65.000-0 |
| W3%Re/W25%Re | 25°C to 1650°C, not for oxidizing atmospheres! | - | Without | HTP40000A65.010-0 |
| W3%Re/W25%Re | 25°C to 2000°C, not for oxidizing atmospheres! | 2x tungsten, NGB808999, wall thickness 0.2 mm; optional for STA 449 F3 Jupiter®: tungsten crucible, with wall thickness 0.3 mm, GB453520 | Included | HTP40000A53.000-00 |
| W3%Re/W25%Re | 25°C to 2000°C, not for oxidizing atmospheres! | - | Without | HTP40000A53.010-00 |



TGA-DTA sensors with different crucibles and plates; for more crucibles, please see TGA

TGA-DTA sensor Type W (W3%Re/W25%Re)

TGA-DTA sensor Type S, protected thermocouples

TGA-DSC Sensors for the STA 449 F1/F3/F5 Jupiter® – for Standard Measurements

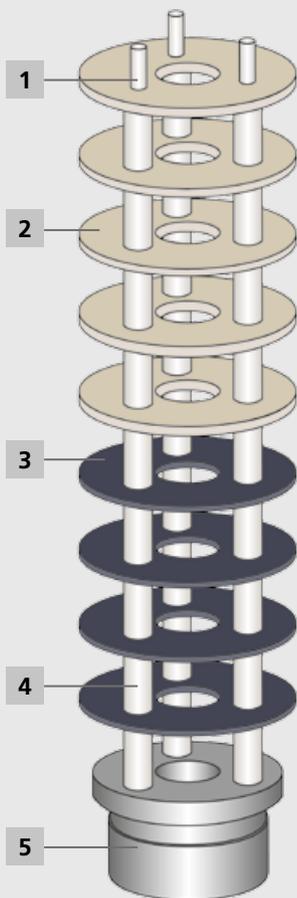
| Sensor Type | Temperature Range/ Atmosphere | Crucible/Volume/Order Number | Radiation Shield | Order Number |
|---------------------|--|---|------------------|---------------------------|
| Type P | -150°C to 1000°C | 2x Al ₂ O ₃ , 85 µl, crucible GB399972, lid GB399973 | Included | HTP40000A59.000-00 |
| Type P | -150°C to 1000°C | - | Without | HTP40000A59.010-00 |
| Type S | 25°C to 1650°C | 2x Al ₂ O ₃ , 85 µl, crucible GB399972, lid GB399973 | Included | HTP40000A69.000-00 |
| Type S | 25°C to 1650°C | - | Without | HTP40000A69.010-00 |
| Type E | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 85 µl, crucible GB399972, lid GB399973 | Included | HTP40000A70.000-00 |
| Type E | -150°C to 700°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A70.010-00 |
| Type E octagonal | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 85 µl, crucible GB399972, lid GB399973 | Included | HTP40000A79.000-00 |
| Type E octagonal | -150°C to 700°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A79.010-00 |
| Type K | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 85 µl, crucible GB399972, lid GB399973 | Included | HTP40000A71.000-00 |
| Type K | -150°C to 700°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A71.010-00 |
| Type K octagonal | -150°C to 800°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 85 µl, crucible GB399972, lid GB399973 | Included | HTP40000A77.000-00 |
| Type K octagonal | -150°C to 800°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A77.010-00 |



Different TGA-DSC Sensors

Radiation Shield for STA Systems

Radiation Shield with 3 Supporting Bars



Component parts

Radiation shield – Complete

- 1** Supporting bar made of Al_2O_3
- 2** Disk for radiation shield made of Al_2O_3
- 3** Disk for radiation shield made of nickel
- 4** Spacing tube made of Al_2O_3
- 5** Metal base for radiation shield

Order number

HTP40000A72.040-00

GB348216

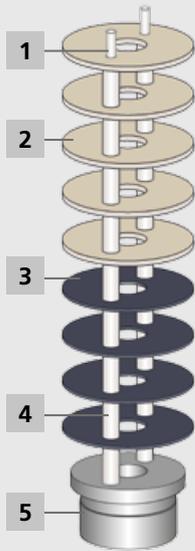
NGB809780

NGB809779

GB351383

NGB809778

Radiation Shield with 2 Supporting Bars



Component parts

- 1** Supporting bar made of Al_2O_3
- 2** Disk for radiation shield made of Al_2O_3
- 3** Disk for radiation shield made of nickel
- 4** Distance tube made of Al_2O_3
- 5** Metal base for radiation shield

Order number

- GB348216**
- NGB806979**
- NGB806987**
- GB351383**
- HTP40000A72.020-01**



TGA and TGA-DSC Sensors for c_p Determination and Operation with the Automatic Sample Changer (ASC)

TGA-DSC Sensors for the STA 449 F1/F3/F5 Jupiter® – for c_p Determination (Recommended up to 1400°C)

| Sensor Type | Temperature Range/ Atmosphere | Crucible/Volume/Single Order Number | Radiation Shield | Order Number |
|---|----------------------------------|--|---------------------|---------------------------|
| Type S, round design, especially for c_p | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Included | HTP40000A54.000-00 |
| Type S, round design, especially for c_p | 25°C to 1650°C | - | Without | HTP40000A54.010-00 |
| Type S | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Included | HTP40000A72.000-00 |
| Type S | 25°C to 1650°C | - | Without | HTP40000A72.010-00 |
| Type S octagonal version | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Included | HTP40000A78.000-00 |
| Type S octagonal version | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Without | HTP40000A78.010-00 |
| Type B | 25°C to 1750°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Included | HTP40000A76.000-00 |
| Type B | 25°C to 1750°C | - | Without | HTP40000A76.010-00 |

TGA-DSC sensors for c_p determination



TGA, TGA-DTA and TGA-DSC Sensors for the STA 449 F1/F3/F5 Jupiter® – for Operation with the Automatic Sample Changer (ASC)

| Sensor Type | Temperature Range/ Atmosphere | Crucible/Volume/Single Order Number | Radiation Shield | Order Number |
|---|--|---|---------------------|---------------------------|
| TGA-DSC, Type P | -150°C to 1000°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Included | HTP40000A63.000-00 |
| TGA-DSC, Type P | -150°C to 1000°C | - | Without | HTP40000A63.010-00 |
| TGA-DSC, Type E | -150°C to 700°C, inert 150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 85 µl, crucible GB399972, lid GB399973 | Included | HTP40000A66.000-00 |
| TGA-DSC, Type E | -150°C to 700°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A66.010-00 |
| TGA-DSC, Type S, for c _p | 25°C to 1650°C | 2x PtRh crucible, GB399205, lid GB399860 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Included | HTP40000A73.000-00 |
| TGA-DSC, Type S, for c _p | 25°C to 1650°C | - | Without | HTP40000A73.010-00 |
| TGA-DSC, Type K | -150°C to 700°C, inert -150°C to 500°C, oxidizing | 2x Al ₂ O ₃ , 85 µl crucible GB399972, lid GB399973 | Included | HTP40000A74.000-00 |
| TGA-DSC, Type K | -150°C to 800°C, inert -150°C to 500°C, oxidizing | - | Without | HTP40000A74.010-00 |
| TGA, type S | 25°C to 1650°C | Spare part: slip-on plate made of Al ₂ O ₃ , NGB809010 Crucible: Al ₂ O ₃ , 85 µl, GB399972 Lid: GB399973  | Included | HTP40000A75.000-00 |
| TGA, type S | 25°C to 1650°C | - | Without | HTP40000A75.010-00 |
| TGA-DTA, type S, with protected thermocouples and slip-on plates | 25°C to 1650°C | Spare part: slip-on plate made of Al ₂ O ₃ , NGB815222; 2x Al ₂ O ₃ crucible, GB399972, lid GB399973 | Included | HTP40000B68.000-00 |
| TGA, type P | -150°C to 1000°C | Spare part: slip-on plate made of Al ₂ O ₃ , NGB809010 Crucible: Al ₂ O ₃ , 85 µl, GB399972 | Included | HTP40000A67.000-00 |
| TGA, type P | -150°C to 1000°C | - | Without | HTP40000A67.010-00 |

TGA-DSC sensor for ASC operation



TGA-DTA sample carrier (with protected thermocouples) and slip-on plates for ASC operation



Different Sensor Types – The Right Sensor for Each Application

The STA 449 **F1/F3 Jupiter**® and DSC 404 **F1/F3 Pegasus**® systems are unique in their flexibility. One of their outstanding features is their capability for easy insertion of different sensor types and sample carriers in a matter

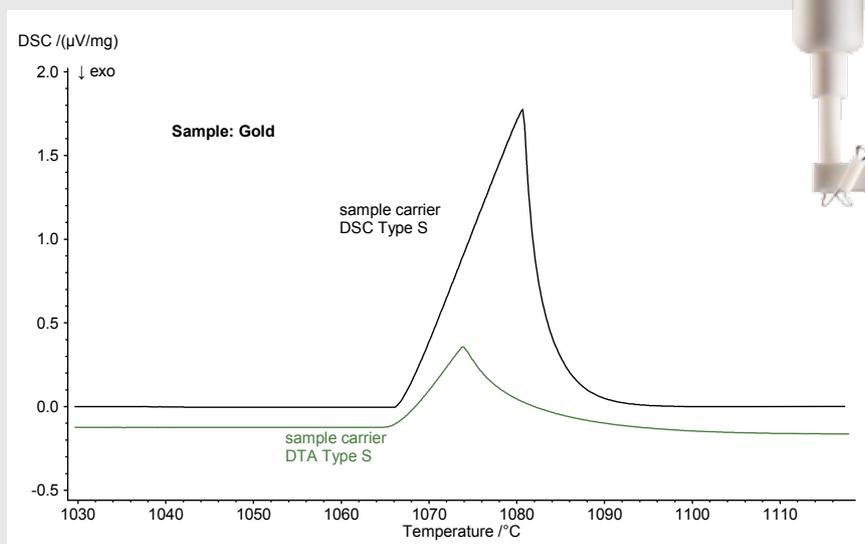
of only a few seconds. Selection of the right crucible, together with the most appropriate furnace out of nine options, allows a single instrument to cover an extremely broad application range.

Of course, the vacuum-tight design of these instruments further extends the measurement capabilities in the temperature range between -150°C and 2400°C.

Advantages of True DSC Measurements

True DSC measurements require special sensors with optimized design and thermocouple positioning. They are not the same as a calibrated DTA test.

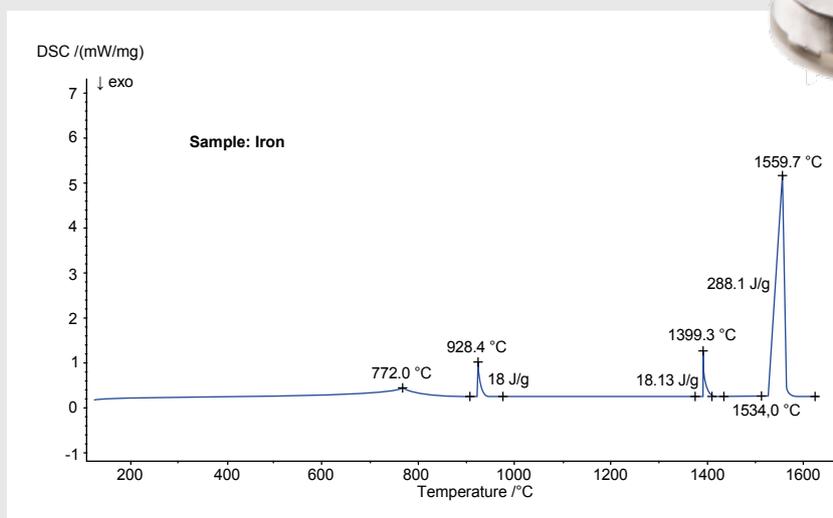
- DSC sensors offer a more stable baseline, a higher sensitivity, an improved detection limit, and a shorter time constant.
- Therefore, transition peaks are sharp, reliable and easy to separate from the baseline curvature.
- DSC sensors can detect weak transitions and glass transition steps and offer excellent reproducibility.
- DSC- c_p sensors are capable of measuring the specific heat (c_p).



DSC and DTA type S sensor comparison

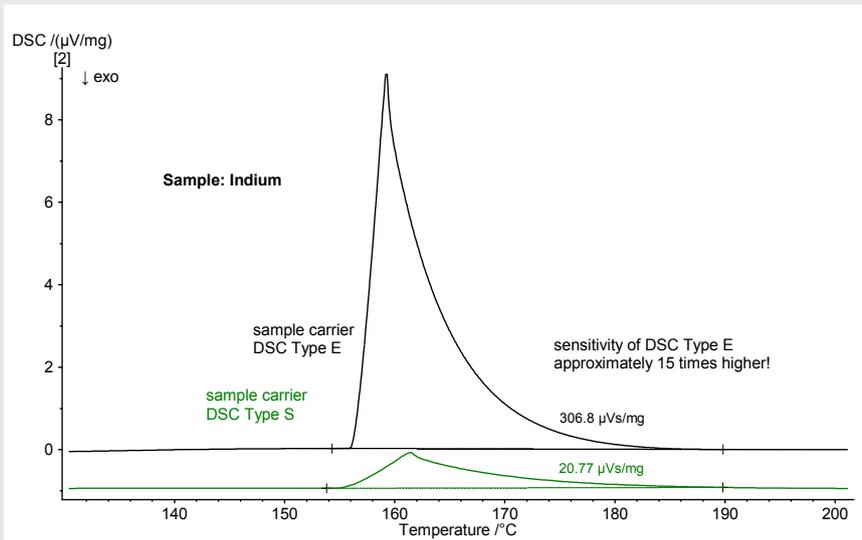
Detection of Weak Effects with the High-Temperature DSC Sensor

This example shows a DSC measurement of an iron disk between room temperature and 1650°C. The iron disk was placed in a Pt+Al₂O₃ liner closed with a Pt lid. The sample mass amounted to 129.68 mg and was heated in an argon atmosphere at a rate of 20 K/min. The DSC type S sensor is especially designed for c_p determination and exhibits outstanding sensitivity. Even the weakest of effects such as iron solid-solid transitions can clearly be detected. Melting occurs at 1534 °C (onset).



DSC measurement of an iron disk up to 1650°C





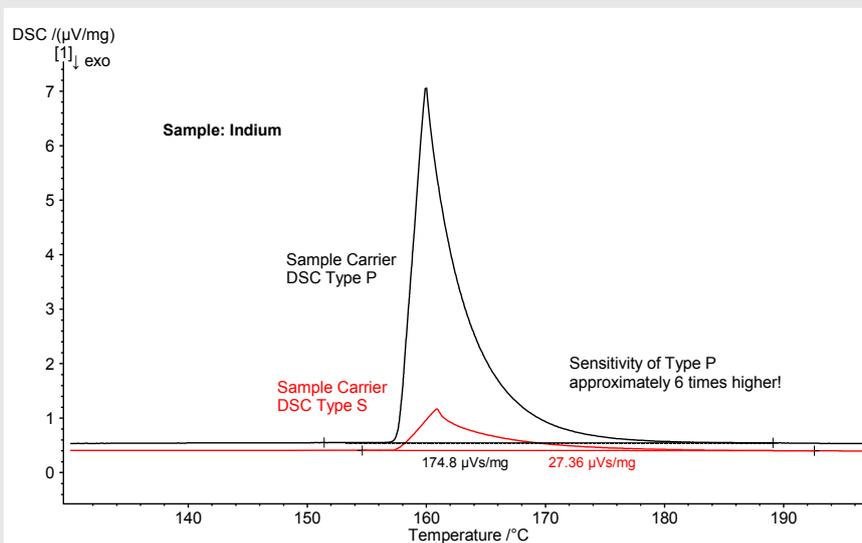
DSC sensor types E and S comparison

DSC Sensor Types E and S

DSC sensor types E, K and P allow measurements with extremely high sensitivity.

DSC type S and B sensors have the advantage of a wide temperature range and a short time constant.

The DSC signal in the lower temperature range is approx. 15 times higher for sensor type E than for type S. On the other hand, the resolution of type S is very high.



Comparison of DSC sensor types P and S

DSC Sensor Types P and S

In the lower temperature range up to 1000°C, the DSC sensor type P has excellent sensitivity and well-pronounced resolution compared to the high-temperature sensor type S. The latter is designed for measurements up to 1650°C. Its sensitivity increases with temperature.

This example shows that the DSC sensor type P is a good alternative to the DSC sensors type E and K (max. 700°C/800°C in an inert atmosphere) when temperatures up to 1000°C are required.

Tungsten for Highest Temperature Applications

Basic Requirements for Highest Temperature Applications

For applications at very high temperatures, critical hardware issues have to be resolved. Ceramic insulation materials become electrically conductive, thermocouple wires become very brittle, and for most materials, a potentially high level of vapor pressure needs to be taken into consideration. Additionally, metallic parts are sensitive to oxygen and the heat transfer is dominated by conduction and radiation. These problems have certain consequences for the design of the high-temperature instruments, their sample holders and crucibles by making it necessary to ensure the following:

- No insulation of thermocouples in the hot zone
- Possibility of repairing thermocouples on site
- Free access to the sample holder for loading sample and reference crucibles
- Good vertical upward purge gas flow to take away all vaporized sample products
- Vacuum-tight instrument construction
- Contact between crucible and sample carrier independent of the atmosphere, radiation and temperature condition
- Possibility of covering samples for unchanged radiation behavior

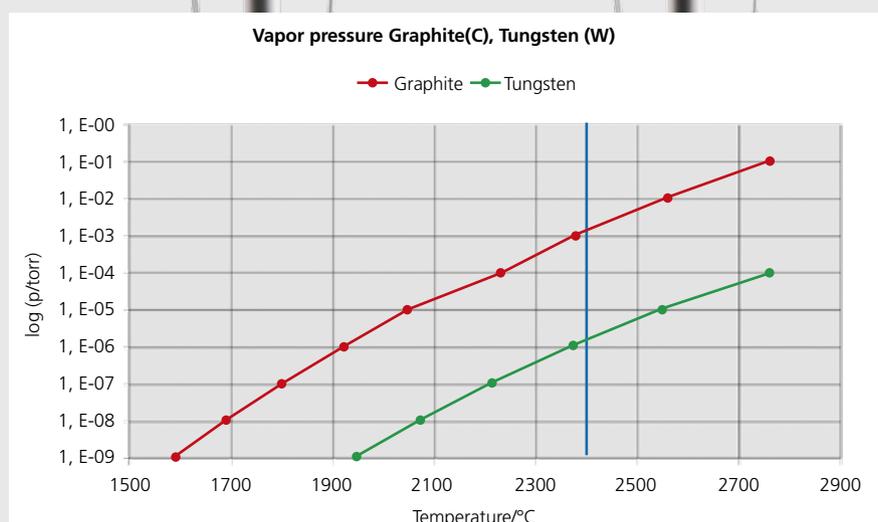
Low-Mass Sample Holder Made of Tungsten

The low-mass tungsten sample holder has well-defined heat flow paths and a split geometry for high calorimetric sensitivity. The conical sample crucible fits safely into the massive sample holder plate. The thermocouple connection is designed such that no welding is necessary. It is thus possible to measure the temperature and DTA signal directly and with high accuracy. Additionally, this sample holder allows for a “crucible-in-crucible” arrangement in order to handle critical samples.

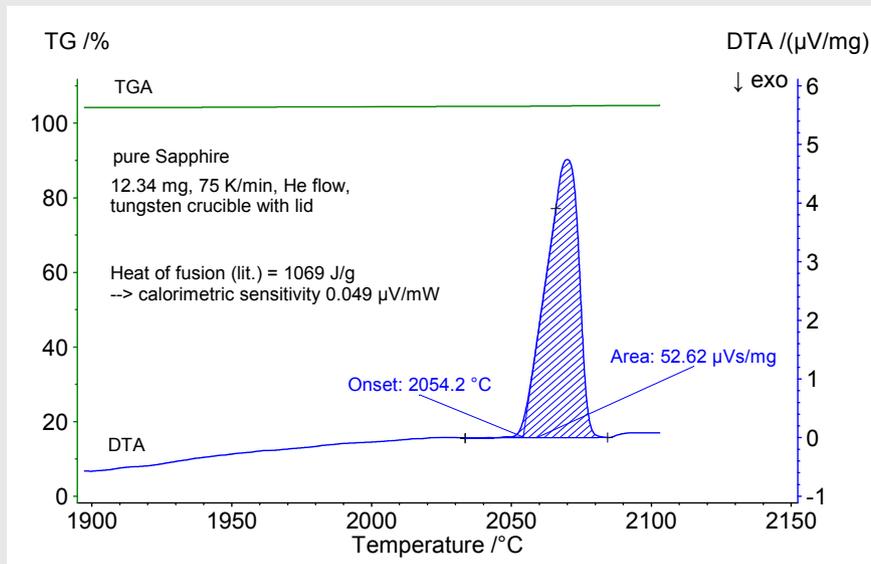


Tungsten – The Material of Choice for Highest-Temperature Measurements

At 2400°C, the vapor pressure of graphite is 10^{-3} Torr, therefore no high vacuum can be applied. The tungsten furnace of the STA 449 **F1 Jupiter**[®] allows measurements in high vacuum (10^{-5} Torr) up to 2400°C. This is essential for clean atmospheres and Knudsen cell tests.



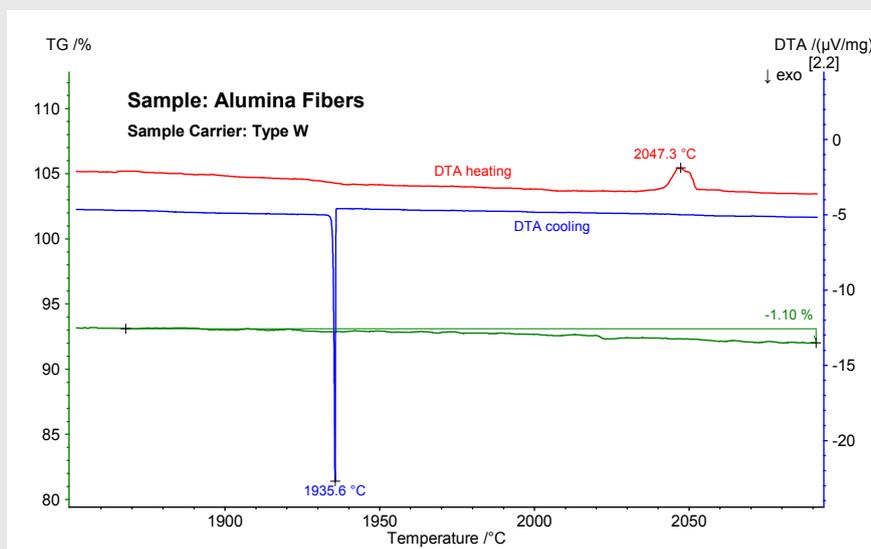
Pure Sapphire at the Highest Temperatures: Tungsten Furnace, Sensor, and Tungsten Crucibles



This TGA-DTA measurement on pure sapphire was performed in the tungsten furnace up to 2100°C. The crucibles were closed with lids, both made of tungsten. Melting of the pure material occurred at 2054°C (extrapolated onset). Determination of the calorimetric sensitivity of the TGA-DTA sensor Type W was based on this measurement in a helium atmosphere. The calorimetric sensitivity was determined to be 0.049 μ V/mW. As expected for the pure material, the TGA curve (green) shows no mass loss before and during melting.

Melting of a pure sapphire sample

Alumina Fibers at the Highest Temperatures: Tungsten Sensor and Tungsten Crucibles



Melting and recrystallization of alumina fibers observed in the DTA signal. Small mass loss indicates evaporation of the sample.

Tungsten has a lower vapor pressure compared to graphite and is therefore the material of choice when very high-end temperatures are required. This TGA-DTA measurement was performed with the tungsten furnace and sensor type W3%Re/W25%Re. Alumina fibers (6.8 mg) in the tungsten crucible were heated to 2100°C and then cooled in a helium atmosphere. Upon heating, an endothermic effect occurred at 2047°C (peak temperature) which can be attributed to melting of the alumina fiber sample. Upon cooling, recrystallization occurred at 1936°C (peak temperature). A slight mass loss was detected above 1900°C which was probably due to partial evaporation of the sample.

Special Sample Carriers for Special Applications – **OTS**®

Oxygen Trap System – **OTS**®

The **OTS**® oxygen trap system, for the STA 449 **F1/F3** Jupiter® and DSC 404 **F1/F3** Pegasus® systems removes traces of residual oxygen in the gas atmosphere inside the instrument. A residual oxygen content of <1ppm can be achieved.

A ceramic substrate bearing a getter ring is mounted on the sample carrier or in the sample carrier tube. This getter

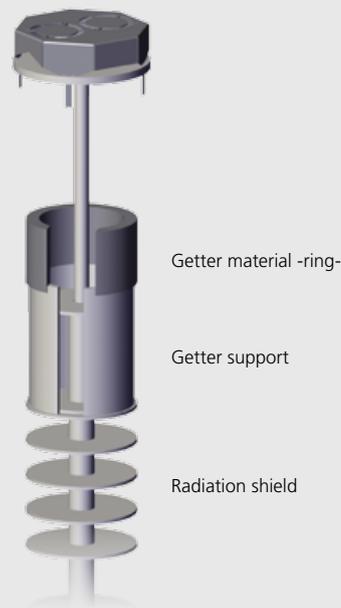
ring is capable of almost entirely eliminating the residual oxygen after evacuation.

Such low oxygen concentrations cannot be achieved unless the instrument is vacuum-tight and equipped with an evacuation system. Both of these requirements are fulfilled by the STA 449 **F1/F3** Jupiter® and DSC 404 **F1/F3** Pegasus® systems.

Residual O₂ level is a function of:

- Vacuum tightness of the instrument
- Desorption of O₂ from the walls
- Vacuum tightness of the gas supply
- Purity of the purge gas

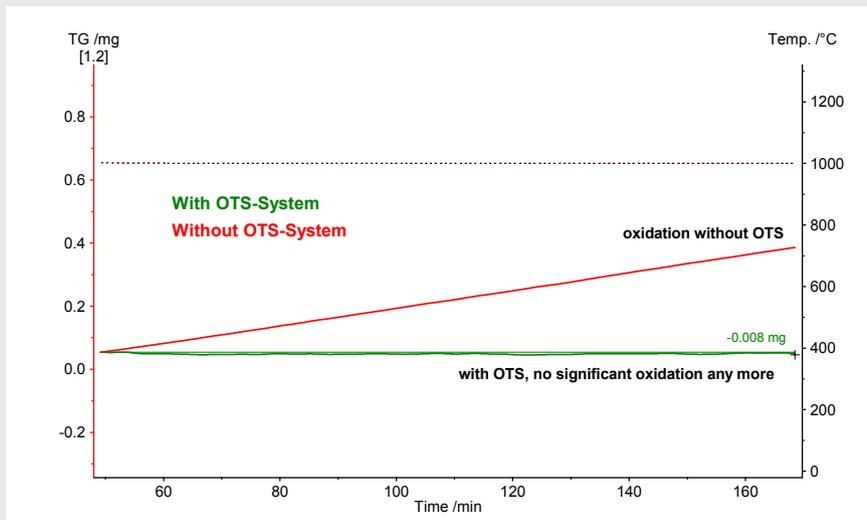
TGA-DSC Sensor



OTS® – for STA 449 F1/F3/F5 Jupiter® and DSC 404 F1/F3 Pegasus®

| Instrument | Order Number |
|---|--------------------|
| STA – OTS® getter support and ring | HTP40000A97.010-00 |
| DSC – OTS® getter support and ring | HTP40000A98.010-00 |
| STA/DSC – getter ring (for ordering separately) | NGB809270 |

Please note: The OTS® system can be used with all sensor types. Restrictions may occur with sensor type P when used with the steel and silver furnace. Please contact your sales & service representative for details.



STA measurement of zirconium under isothermal conditions

Oxidation of Zirconium

In this example, a zirconium sample (190.0 mg) was measured with the STA 449 **F1 Jupiter**® in a helium atmosphere under isothermal conditions (1000°C).

After three hours, oxidation of the Zr sample (red curve) led to a total mass increase of approx. 0.38 mg when no OTS® was used.

When the OTS® system was applied to the sample carrier for a measurement on the same sample mass, however, no significant oxidation occurred (green curve). The mass increase was determined to amount to only 0.008 mg.

Special Sample Carriers for Special Applications – Hanging Samples

Large Sample Surface – Excellent Atmospheric Access

The special TGA sample carrier and TGA-DTA sensor for hanging or suspended samples allow perfect access to all sample surfaces by the atmosphere.

These Al₂O₃ sample carriers and sensors with Al₂O₃ frame are available for the STA 449 **F1/F3/F5** Jupiter® systems (TGA order no. HTP40000A60.000-00; TGA-DTA order no. HTP40000A61.000-00).

The sample carrier is easy to handle and allows selection of the hanging wires according to the sample properties.

Advantages of the Sample Carrier for Hanging Samples

- Sample can be hung down directly
- Bendable thermocouple type S
- Improved contact between the sample and purge gas
- Sample dimensions up to 13 x 20 mm
- Very slow mass increase detectable (µg/hour range)
- Long-term signal stability, typical drift less than 0.1 µg/h
- Choice of atmosphere:
 - O₂
 - Corrosive
 - Controlled humidity

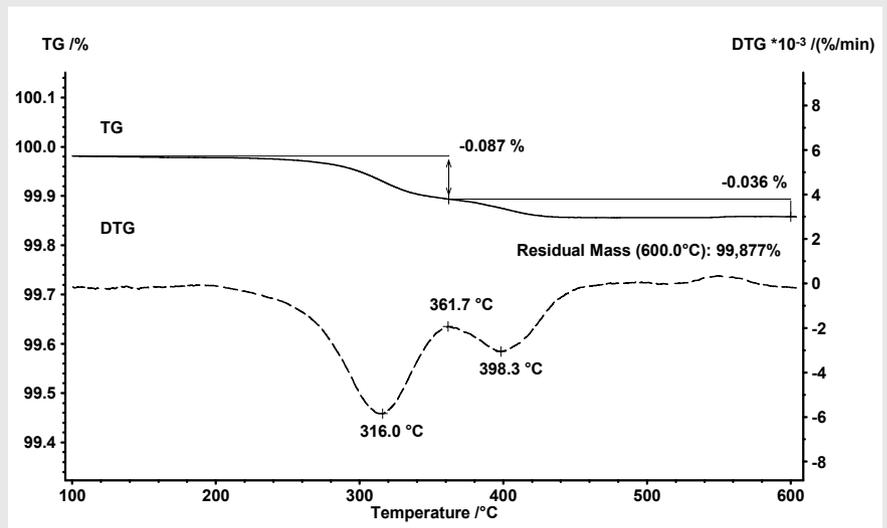


TGA-DTA sensor
for hanging
samples

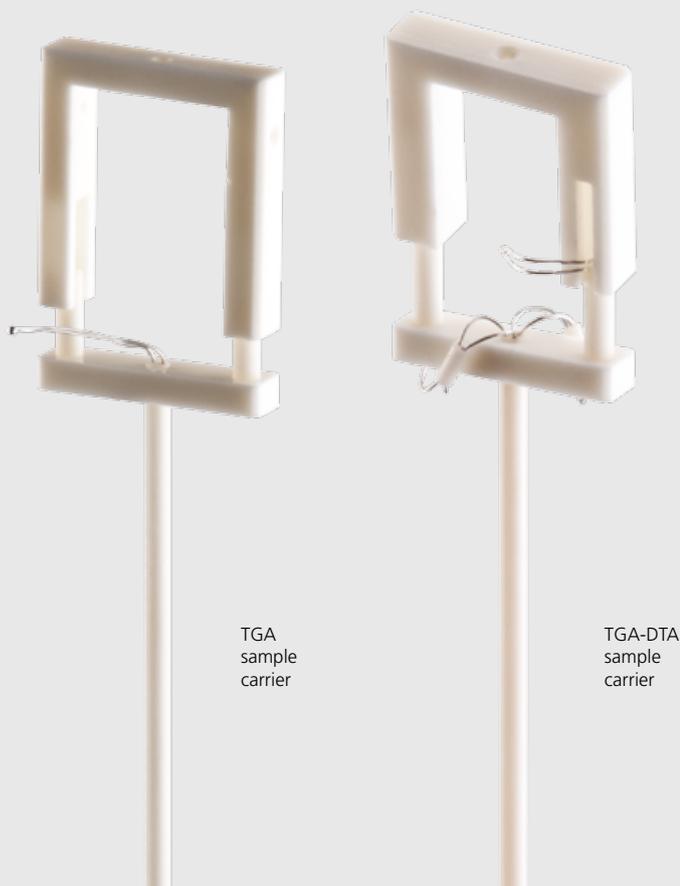
TGA Measurement of a Hanging Coated Glass Sample

In this example, two sheets of a coated glass sample were pierced and fixed into the special TGA sample holder for suspended samples. The total sample mass amounted to 274.99 mg. The two sheets were heated up to 600°C in synthetic air (heating rate 5 K/min).

The improved contact between the sample surface and purge gas resulted in the detection of very weak mass-loss steps at 316°C (0.087%) and 398°C (0.036%).



Mass-loss behavior of coated glass sheets



Calibration

Calibration is a fundamental requirement for thermoanalytical investigations. The knowledge of a quantitatively defined relationship between the value indicated by the measuring instrument and the correct value is essential.

Calibration of modern Differential Scanning Calorimeters (DSC) and Simultaneous Thermal Analyzers (STA) is achieved by quantification of the produced signal when a known quantity of energy is generated within the system.

The calibration procedure consists of measuring thermal properties of

standard materials which are well known.

Experimental conditions of calibration and measurement should be matched as closely as possible: not only the quantity of energy to be measured must be similar but the site and kinetics of the generation and temperature range should be as close as possible in both calibration and measurement experiments.

All the results of subsequent measurements depend on the validity of the calibration. Therefore, it is mandatory to carefully carry out all calibration procedures.

Thermal Conductivity of Crucible

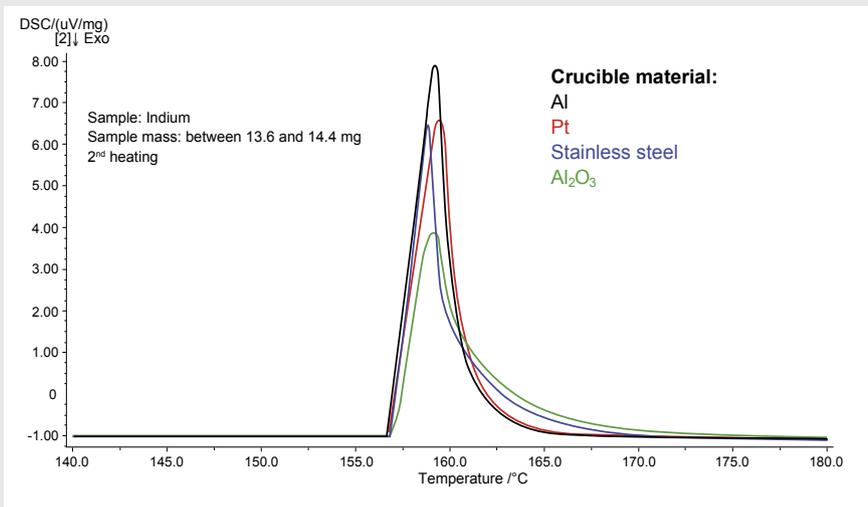
Type of Instrument

DSC/TGA

CALIBRATION INFLUENCING FACTORS

Kind & Dimension of Crucible

Mass of Sample



Indium measurements in different crucible materials

Crucible Selection

DSC and STA (TGA-DSC/DTA) measurements are strongly influenced by the crucible material. The measurements shown here on indium in Al, Pt, stainless steel and Al₂O₃ crucibles clearly demonstrate that both the thermal conductivity and the mass of the crucible have an effect on the DSC and DTA peak shape. The calibration procedures take these effects into consideration and eliminate the influence of the crucible material on the measurement results.

Heating & Cooling Rate

Type of Cooling System

Type & Flow Rate of Purge Gas

Calibration Materials and Kits

Calibration Materials

Reference materials must be homogeneous and stable, and the certified values must be accurate. Finding a material that can be used as a reference for more than one property is one of the goals of current research. Various calibration kits and individual standards are available for each thermoanalytical method presented (DSC, TGA and STA) allowing the instrument to be calibrated across its entire temperature range and allowing for the use of different crucible types. The table gives an overview of possible combinations of calibration and crucible materials.

| | | Crucible Materials | | | | | | | | | |
|---|---------------------------------|--------------------------------|---|------------------|----|----|----|-------------------------|----|---|---|
| | | Al ₂ O ₃ | C | SiO ₂ | Al | Ag | Au | Stain- less Steel | Pt | W | |
| Common Calibration Materials/Phase Transition Temperature | C ₁₀ H ₁₆ | -64.5°C | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ |
| | H ₂ O | 0.0°C | - | - | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | - |
| | Biphenyl | 69.2°C | ✓ | | | ✓ | | | | ✓ | |
| | RbNO ₃ | 164.2°C | ! | | | ✓ | ✓ | ✓ | | ✓ | |
| | Benzoic acid | 122.4°C | ✓ | | | ✓ | | | | ✓ | |
| | In | 156.6°C | ✓ | ✓ | ✓ | ✓ | - | ! | ✓ | ! | |
| | Sn | 231.9°C | ✓ | ✓ | ✓ | ✓ | - | - | ! | - | |
| | Bi | 271.4°C | ✓ | | | ✓ | - | - | ✓ | - | ✓ |
| | KClO ₄ | 300.8°C | ✓ | | | ✓ | ✓ | ✓ | | ✓ | |
| | Pb | 327.5°C | ✓ | ✓ | ✓ | ✓ | - | - | ✓ | - | ! |
| | Zn | 419.5°C | ✓ | ✓ | ✓ | ✓ | - | - | - | - | ✓ |
| | Ag ₂ SO ₄ | 426.4°C | ✓ | | | ✓ | | | - | ✓ | |
| | CsCl | 476.0°C | ✓ | | | ✓ | ✓ | ✓ | ✓ | ✓ | |
| | Li ₂ SO ₄ | 578.0°C | ✓ | ✓ | ✓ | ✓ | | ✓ | | ✓ | |
| | Al | 660.3°C | ✓ | ✓ | ! | - | - | - | - | - | ✓ |
| | K ₂ CrO ₄ | 668.0°C | ✓ | | | - | ✓ | | - | ✓ | |
| | BaCO ₃ | 808.0°C | ✓ | | - | - | - | | - | ✓ | |
| | Ag | 961.8°C | ✓ | ✓ | ✓ | - | - | - | - | - | ✓ |
| Au | 1064.2°C | ✓ | - | ✓ | - | - | - | - | - | ✓ | |
| Ni | 1455.0°C | ! | - | - | - | - | - | - | - | | |

- ✓ No solubility or effects on the melting temperature expected
- ! Corrosion reactions possible with negligible changes in the melting temperature
- Melt or transformation product reacts with the crucible material. Changes in the melting temperature can be expected.
- Compatibility unknown

Calibration Kits for DSC and DTA

Various calibration kits for different temperature ranges, methods, and crucible materials are available. The following tables show the common calibration kits for DSC and DTA measurements with their order numbers,

materials transition temperatures (e.g., melting point), lid perforation, and compatibility with crucible material. These kits are used for temperature and enthalpy calibration.

Mass calibration of the TGA systems can usually be done automatically with an internal weight. For temperature calibration via the *c-DTA*[®] feature, the DSC/DTA calibration kits can be used.

Reference Materials for the STA 2500 *Regulus*

| Transition (°C) | Material | | | Quantity | Single Order No. |
|-----------------|----------|--------------------------------|----------|----------|------------------|
| 156.6 | Indium | In | Granules | 5 g | J1560142 |
| 231.9 | Tin | Sn | Granules | 5 g | J1560143 |
| 327.5 | Lead | Pb | Granules | 5 g | J1560144 |
| 660.3 | Aluminum | Al | Wire | 0.1 g | J1560296 |
| 1064.2 | Gold | Au | Wire | 2.5 cm | J1560209 |
| 2072 | Alumina | Al ₂ O ₃ | Powder | 50 cc | J1560146 |

DSC Sets for C_p, Sensitivity and Enthalpy Calibration

| Material | Disc ø | Thickness | For crucible type | Order No. |
|----------|--------|-------------------------|--------------------------------|------------------------|
| Sapphire | 6 mm | 0.25, 0.5, 0.75, 1.0 mm | Metallic | 6.235.1-91.1.00 |
| Sapphire | 5.2 mm | 0.25, 0.5, 0.75, 1.0 mm | Al ₂ O ₃ | 6.235.1-91.2.00 |
| Sapphire | 4 mm | 0.25, 0.5, 0.75, 1.0 mm | Cold-weldable Al crucibles | 6.239.2-91.5.00 |

DSC/DTA Set* for the Use in Al₂O₃ Crucibles Individual Substances in Glass Vials

Order No.: 6.223.5-91.3.00

| Transition (°C) | Material | | Quantity | Single Order No. |
|-----------------|---------------|----|----------|------------------|
| 156.6 | Indium | In | 400 mg | 6.223.5-91.3.01 |
| 231.9 | Tin | Sn | 400 mg | 6.223.5-91.3.02 |
| 271.4 | Bismuth | Bi | 400 mg | 6.223.5-91.3.04 |
| 419.5 | Zinc | Zn | 400 mg | 6.223.5-91.3.03 |
| 660.3 | Aluminum wire | Al | 400 mg | 6.223.5-91.3.05 |
| 961.8 | Silver wire | Ag | 400 mg | 6.223.5-91.3.06 |
| 1064.2 | Gold wire | Au | 400 mg | 6.223.5-91.3.07 |
| 1455.0 | Nickel wire | Ni | 400 mg | 6.223.5-91.3.08 |

* Standard set can be used with high-pressure DSC

Calibration Materials and Kits

DSC Standard Set* – Substances in Sealed Al Crucibles (Volume 25 µl, sample mass minimum 10 mg)

Order No.: 6.239.2-91.3.00
For HP-DSC also

| Transition (°C) | Material | | Pierced lid | Single Order No. |
|-----------------|-----------------|---------------------------------|-------------|------------------|
| -64.5 | Adamantane** | C ₁₀ H ₁₆ | | n/a |
| 156.6 | Indium | In | x | n/a |
| 231.9 | Tin | Sn | x | n/a |
| 271.4 | Bismuth | Bi | x | n/a |
| 419.5 | Zinc | Zn | x | n/a |
| 476.0 | Cesium chloride | CsCl | x | n/a |

* Standard set can be used with high-pressure DSC

** HP-DSC: only atmospheric pressure

DSC Standard Set* – Substances in Sealed Al Concavus Crucibles Made of Al (sample mass approx. 10 mg)

Order No.
DSC21400B00.913-00

| Transition (°C) | Material | | Pierced lid | Single Order No. |
|-----------------|-----------------|---------------------------------|-------------|------------------|
| -64.5 | Adamantane** | C ₁₀ H ₁₆ | x | n/a |
| 156.6 | Indium | In | x | n/a |
| 231.9 | Tin | Sn | x | n/a |
| 271.4 | Bismuth | Bi | x | n/a |
| 419.5 | Zinc | Zn | x | n/a |
| 476.0 | Cesium chloride | CsCl | x | n/a |

* Standard set can be used with high-pressure DSC

** HP-DSC: only atmospheric pressure

DSC Low Pressure Set – Substances in Al Low-Pressure Crucibles (Volume 35 µl, max. 3 bar, sample mass minimum 10 mg)

Order No.: 6.240.10-90.3.00

| Transition (°C) | Material | | Pierced lid | Single Order No. |
|-----------------|-----------------|---------------------------------|-------------|------------------|
| -64.5 | Adamantane | C ₁₀ H ₁₆ | | n/a |
| 156.6 | Indium | In | x | n/a |
| 231.9 | Tin | Sn | x | n/a |
| 271.4 | Bismuth | Bi | x | n/a |
| 419.5 | Zinc | Zn | x | n/a |
| 476.0 | Cesium chloride | CsCl | x | n/a |

DSC High-Pressure Set – Substances in CrNi Steel Crucibles (Volume 27 µl, max. 100 bar, sample mass minimum 10 mg)

Order No.: 6.240.1-91.4.00

| Transition (°C) | Material | | Pierced lid | Single Order No. |
|-----------------|-----------------|---------------------------------|-------------|------------------|
| -64.5 | Adamantane | C ₁₀ H ₁₆ | | n/a |
| 156.6 | Indium | In | | n/a |
| 231.9 | Tin | Sn | | n/a |
| 271.4 | Bismuth | Bi | | n/a |
| 419.5 | Zinc | Zn | | n/a |
| 476.0 | Cesium chloride | CsCl | | n/a |

DSC High-Pressure Set – Substances in CrNi Steel Crucibles (Volume 100 µl, sample mass minimum 10 mg)

Order No.: 6.240.1-91.5.00

| Transition (°C) | Material | | Pierced lid | Single Order No. |
|-----------------|-----------------|---------------------------------|-------------|------------------|
| -64.5 | Adamantane | C ₁₀ H ₁₆ | | n/a |
| 156.6 | Indium | In | | n/a |
| 231.9 | Tin | Sn | | n/a |
| 271.4 | Bismuth | Bi | | n/a |
| 476.0 | Cesium chloride | CsCl | | n/a |

DSC Medium-Pressure Set – Substances in CrNi Steel Crucibles (Volume 120 µl, max. 20 bar, sample mass minimum 10 mg)

Order No.: 6.240.1-91.6.00

| Transition (°C) | Material | | Pierced lid | Single Order No. |
|-----------------|------------|---------------------------------|-------------|------------------|
| -64.5 | Adamantane | C ₁₀ H ₁₆ | | n/a |
| 0.0 | Water | H ₂ O | | n/a |
| 156.6 | Indium | In | | n/a |
| 231.9 | Tin | Sn | | n/a |

DSC µ-Sensor Set* – Substances in Al Crucibles (volume 25 µl)

Order No.: 6.240.3-91.3.00

| Transition (°C) | Material | | Pierced lid | Single Order No. |
|-----------------|------------|---------------------------------|-------------|------------------|
| -64.5 | Adamantane | C ₁₀ H ₁₆ | | n/a |
| 156.6 | Indium | In | x | n/a |
| 231.9 | Tin | Sn | x | n/a |
| 271.4 | Bismuth | Bi | x | n/a |

* Calibration set only for DSC **F1** with µ-sensor

DSC/DTA Set for the Use in Al₂O₃ Crucibles, Individual Substances in Glass Vials

Order No.: 6.223.5-91.1.00

| Transition (°C) | Material | | Quantity | Single Order No. |
|-----------------|---------------------|---------------------------------|---|------------------|
| -64.5 | Adamantane (Powder) | C ₁₀ H ₁₆ | 400 mg | 6.217.1-92.1.09 |
| 156.6 | Indium | In | 400 mg | 6.223.5-91.3.01 |
| 231.9 | Tin | Sn | 400 mg | 6.223.5-91.3.02 |
| 271.4 | Bismuth | Bi | 400 mg | 6.223.5-91.3.04 |
| 419.5 | Zinc | Zn | 400 mg | 6.223.5-91.3.03 |
| 476.0 | Cesium chloride | CsCl | 400 mg (for individual order, vial contains 500 mg) | 6.223.5-91.2.05 |
| 660.3 | Al wire | Al | 400 mg | 6.223.5-91.3.05 |

Calibration Materials and Kits

DSC/DTA Individual Substances in Glass Vials for Use in Pt Crucibles

| Transition (°C) | Material | | Quantity | Single Order No. |
|-----------------|------------------------|------------------------------------|----------|------------------|
| 69.2 | Biphenyl* | C ₁₂ H ₁₀ | 500 mg | 6.223.5-91.2.01 |
| 122.4 | Benzoic acid | C ₆ H ₅ COOH | 500 mg | 6.223.5-91.2.02 |
| 164.2 | Rubidium nitrate* | RbNO ₃ | 500 mg | 6.223.5-91.2.03 |
| 300.8 | Potassium perchlorate* | KClO ₄ | 500 mg | 6.223.5-91.2.04 |
| 426.2 | Silver sulfate | AgSO ₄ | 500 mg | 6.223.5-91.2.08 |
| 476.0 | Cesium chloride | CsCl | 500 mg | 6.223.5-91.2.05 |
| 668.0 | Potassium chromate* | K ₂ CrO ₄ | 500 mg | 6.223.5-91.2.06 |
| 808.0 | Barium carbonate | Ba CO ₃ | 500 mg | 6.223.5-91.2.07 |

* Hazardous material

Calibration Kits for TGA and STA

Temperature Calibration via Magnetic Transitions

| TG 209 F1 Libra®, TG 209 F3 Tarsus | | Order Number |
|--|--|--------------------|
| 6 Curie Standards | | 6.221.2-91.1.00 |
| Calibration supplement for magnetic transition | | 6.220.10-93.1.00 |
| STA 449 F1/F3 Jupiter® | | Order Number |
| 6 Curie Standards | | 6.221.2-91.1.00 |
| Calibration supplement for magnetic transition | | HTP40000A96.010-00 |

Verification of Mass Changes for STA 449 F1/F3/F5 Jupiter® and TG 209 F1 Libra® / F3 Tarsus® - Substance in Glass Vial

| Material | Quantity | Single Order No. | |
|-----------------------------|--|------------------|-----------------|
| Calcium oxalate monohydrate | C ₂ H ₂ CaO ₅ | 2 g | 6.223.5-91.2.10 |

c-DTA® Set (TGA) for the Use in Al₂O₃ Crucibles, Substances in Glass Vials

Order No.: 6.221.5-91.1.00

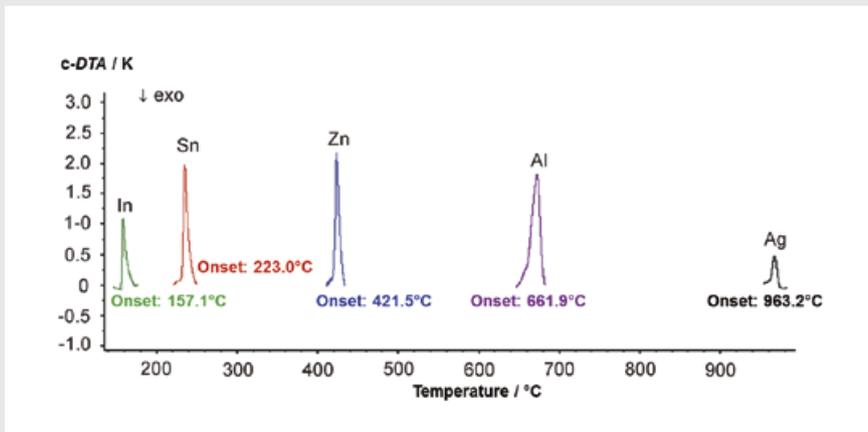
| Transition (°C) | Material | | Quantity | Single Order No. |
|-----------------|------------------------|----|----------|------------------|
| 156.6 | Indium | In | 400 mg | 6.223.5-91.3.01 |
| 231.9 | Tin | Sn | 400 mg | 6.223.5-91.3.02 |
| 271.4 | Bismuth | Bi | 400 mg | 6.223.5-91.3.04 |
| 419.5 | Zinc | Zn | 400 mg | 6.223.5-91.3.03 |
| 660.3 | Aluminum wire (ø 1 mm) | Al | 400 mg | 6.223.5-91.3.05 |
| 961.8 | Silver wire (ø 0.5 mm) | Ag | 400 mg | 6.223.5-91.3.06 |

c-DTA® Set* (TGA) for the Use in Al₂O₃ Crucibles, Substances in Glass Vials

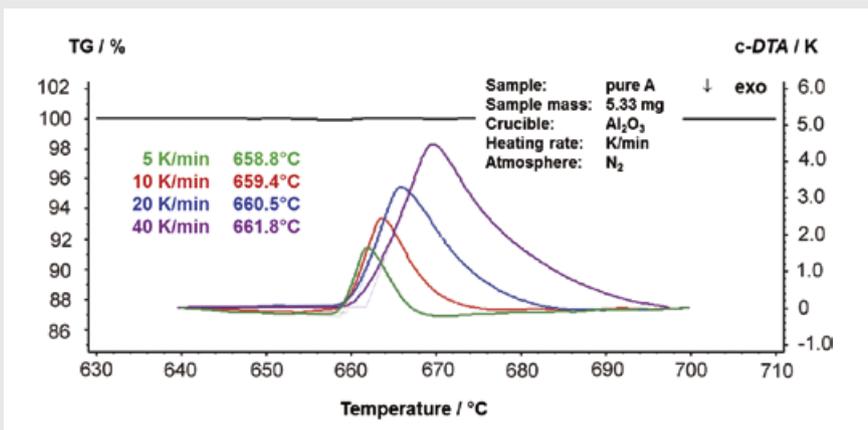
Order No.: TGA209F1D91.010-00

| Transition (°C) | Material | Quantity | Single Order No. |
|-----------------|------------------------|----------|------------------|
| 156.6 | Indium | 400 mg | 6.223.5-91.3.01 |
| 231.9 | Tin | 400 mg | 6.223.5-91.3.02 |
| 271.4 | Bismuth | 400 mg | 6.223.5-91.3.04 |
| 419.5 | Zinc | 400 mg | 6.223.5-91.3.03 |
| 660.3 | Aluminum wire (ø 1 mm) | 400 mg | 6.223.5-91.3.05 |
| 961.8 | Silver wire (ø 0.5 mm) | 400 mg | 6.223.5-91.3.06 |
| 1064.2 | Gold (ø 1.0 mm) | 400 mg | 6.223.5-91.3.07 |

* This calibration set is recommended for the TG 209 **F1** *Libra*®



There are various reference materials which can be used for c-DTA® calibration routines.



c-DTA® calibration in the TG 209 **F1** and **F3** systems. Excellent coupling of the Platinel sensor with the crucible guarantees that the influence of the heating rate will be small.

Chemical Behavior of Pt, Al₂O₃ and Graphite Crucibles and Sensors



DSC sample carrier type S (Pt-Pt10%Rh)

Environmental Effects: Chemical Behavior of Crucibles and Sensors Made of Pt, Al₂O₃ and C

In practice, the service life of sensors (thermocouples) and crucibles is most heavily impacted by interactions with the environment. Diffused impurities, released from the samples, change the thermal tension or may even cause initial cracking of the thermocouple wire and crucible wall.

The following tables give some details on the chemical compatibility of Pt, Al₂O₃ and C thermocouples and crucibles with other sample materials and gas atmospheres.

The following lists also serve to illustrate how important regular inspections and calibration measurements are.

These overviews are not exhaustive, but meant rather as a guideline for the user. The temperatures given are primarily literature values. Temperatures under test conditions might shift to lower values. It is always advisable to run preliminary tests in separate furnaces.

Chemical Behavior of Platinum (Pt)

| Critical | No resistance | Limited resistance |
|---|--|--|
| <ul style="list-style-type: none"> ■ Halogens (Cl₂, F₂, Br₂), aqua regia ■ Li₂CO₃, prior to emission of CO₂ ■ PbO, FeCl₂ ■ Be alloys (evaporation) ■ HCl with oxidants (e.g., chromic acid, manganates, iron (III) and molten salts); reducing atmospheres ■ Metals and metal vapors (e.g., B, Pb, Zn, Sn, Ag, Au, Li, Na, K, Sb, Bi, Ni, Fe, etc.; Se > 320°C (evaporation)) ■ Metals and metal oxides with reducing substances such as C, organic compounds or H₂ ■ Oxides in an inert gas atmosphere at higher temperatures (reduction) ■ Sulfur (roughening of the surface, embrittlement) ■ Alkali hydroxides, -carbonates, -sulfates, -cyanides and rhodanides at higher temperatures ■ KHSO₄ at higher temperatures ■ Carbon black or free carbon >1000°C ■ SiO₂ under reducing conditions ■ SiC and Si₃N₄ >1000°C (release of elementary Si) ■ HBr, KCl solution at high temperatures | <ul style="list-style-type: none"> ■ Mixtures of KNO₃ and NaOH at 700°C under exclusion of air ■ Mixtures of KOH and K₂S at 700°C under exclusion of air ■ LiCl at 600°C; MgCl₂, Ba(NO₃)₂ at 700°C ■ HBr, HI, H₂O₂ (30%); HNO₃ at 100°C ■ KCl decomposition products which form during melting at 768°C | <ul style="list-style-type: none"> ■ KHF₂, LiF₂, NaCl at 900°C ■ Mixtures of NaOH and NaNO₃ at 700°C under exclusion of air |

Chemical Behavior of Alumina (Al_2O_3)

Critical

- N_2 in the presence of carbon (formation of AlN ; not recommended to measure carbon black in N_2 atmosphere at elevated temperatures)
- F_2 : formation of AlF_3 and O_2
- Cl_2 : formation of AlCl_3 at $>700^\circ\text{C}$
- Sulfur: no reaction with liquid sulfur; formation of sulfides occur in the presence of carbon in the gas phase
- H_2S : when heated, formation of Al_2S_3
- C: formation of carbides and Al above 1400°C
- HF: reaction to AlF_3 and H_2O at higher temperatures
- CuSO_4 : above 1000°C , diffusion through the bottom of the crucible
- Compounds containing Si, e.g., MoSi_2 ; contamination of $\text{Al}_2\text{O}_3 > 1200^\circ\text{C}$
- Metal fluorides: formation of $[\text{AlF}_6]^{-3}$ anions and salts similar to cryolites
- SiO_2 glass: melt dissolves Al_2O_3
- Hydrogen sulfates of alkaline metals and alkaline-earth metals
- HCl: above 600°C , reactions in the presence of carbon
- B_2O_3 (Borax): melt dissolves Al_2O_3 ; formation of Al-borates and -borides
- Alkaline and alkaline-earth oxides and their salts with volatile anions; important for hydroxides, nitrides, nitrates, carbonates, peroxides, etc.
- CaC_2 : when heated, formation of Al_4C_2
- PbO : reaction above 700°C
- UO_3 : reaction starts at 450°C
- Me^nO : $\text{Me} = \text{Fe}^{2+}, \text{Cu}^{2+}, \text{Ni}^{2+}$, etc., formation of spinels, CaO formation above 1200°C
- Alkaline and alkaline-earth ferrites: melt dissolves Al_2O_3
- Ti-alloys (very high oxygen affinity)
- Reactions with Fe-Ni- and Mg-alloys possible at higher temperatures

Chemical Behavior of Graphite (C)

Critical

- O_2 : reaction above 400°C
- N_2 : reaction starting at 1700°C (formation of small amounts of cyanides)
- Oxides
- Water vapor
- F_2, Br_2 : reaction at room temperature
- Sulfur
- Si: formation of SiC at approx. 1400°C
- Chromic acid
- Chlorosulfonic acid ClSO_3H
- SiO_2 : formation of SiC: formation of SiC via intermediate product SiO (technical production of SiC above 1800°C ; however, reaction between SiO_2 and C begins at lower temperature).
- Nitrous gases (NO, NO_2)
- Sulfuric acid, H_2SO_4 : concentrated acid critical above 150°C , fuming acid already at room temperature
- Nitric acid, HNO_3 : diluted acid critical at 90°C , fuming acid already at room temperature
- SO_3 : critical above 100°C
- Danger of explosion with perchloric acid, HClO_4
- NaOCl : critical above 50°C



Material Compatibility – Sample in Crucible

Material Compatibility

The right crucible must always be chosen for the given experiment. Crucible materials should be specially selected depending on the application and sample materials in order to prevent reactions between the sample and crucible.

These tables serve to indicate which of the common crucible types can be used for each application, including ceramics, metals, inorganics and other more general applications.

General Applications

| Materials/ Crucible Types | Pt/Rh | Al ₂ O ₃ | Al | Pt+Al ₂ O ₃ liner | Al ₂ O ₃ crucible sprayed with Y ₂ O ₃ | Graphite |
|------------------------------|-------|--------------------------------|----|---|--|----------|
| Clays | ✓ | ✓* | ✓ | ✓* | ✓* | No |
| Minerals | ✓ | ✓* | ✓ | ✓* | ✓* | No |
| Oxide ceramics | ✓ | ✓* | ✓ | ✓* | ✓* | No |
| Salts | ✓ | No | ✓ | No | No | No |
| Glasses | ✓ | No | ✓ | No | No | ✓* |
| Metals | No** | ✓ | No | ✓ | ✓ | No |
| Polymers | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ |
| Carbon materials | ✓* | ✓* | ✓ | ✓* | ✓* | ✓ |
| Inorganics | ✓* | ✓* | ✓* | ✓* | ✓* | ✓* |

Ceramics

| Materials/ Crucible Types | Pt/Rh | Al ₂ O ₃ | Al | Pt+Al ₂ O ₃ liner | Al ₂ O ₃ crucible sprayed with Y ₂ O ₃ | Graphite |
|---|-------|--------------------------------|----|---|--|----------|
| Alumina (Al ₂ O ₃) | ✓ | ✓ | ✓ | ✓ | ✓ | ✓* |
| Zirconia (ZrO ₂) | ✓ | ✓ | ✓ | ✓ | ✓ | ✓* |
| Y ₂ O ₃ or MgO | ✓ | ✓ | ✓ | ✓ | ✓ | ✓* |
| Silicon dioxide (SiO ₂) | ✓ | No | ✓ | No | No | No |
| Silicon nitride (Si ₃ N ₄) | No | ✓* | ✓ | ✓* | ✓* | ✓* |
| Aluminum nitride (AlN) | ✓* | ✓* | ✓* | ✓* | ✓* | ✓* |
| Boron nitride (BN) | ✓* | ✓* | ✓* | ✓* | ✓* | ✓* |
| Silicon carbide (SiC) | No | ✓* | | ✓* | ✓* | ✓* |
| Titanium oxide (TiO ₂) | ✓ | ✓ | ✓ | ✓ | ✓ | ✓* |

Metals

| Materials/ Crucible Types | Pt/Rh | Al ₂ O ₃ | Al | Pt+Al ₂ O ₃ liner | Al ₂ O ₃ crucible sprayed with Y ₂ O ₃ | Graphite |
|------------------------------|-------|--------------------------------|----|---|--|----------|
| Al and Al-alloys | No** | ✓ | No | ✓ | ✓ | ✓ |
| Mg and Mg-alloys | No** | ✓* | No | ✓* | ✓* | ✓ |
| Cu and Cu-alloys | No** | ✓ | No | ✓ | ✓* | ✓ |
| Fe and Fe-alloys | No** | ✓* | No | ✓* | ✓ | No |
| Ni and Ni-alloys | No** | ✓* | No | ✓* | ✓ | No |
| Ti and Ti-alloys | No** | ✓* | No | ✓* | ✓ | No |
| Sn and Sn-alloys | No** | ✓ | No | ✓ | ✓ | ✓ |
| Au- and Ag-alloys | No** | ✓ | No | ✓ | ✓ | ✓* |
| Cr-, Mo-, Co-alloys | No** | ✓* | No | ✓* | ✓ | No |

Inorganics

| Materials/ Crucible Types | Pt/Rh | Al ₂ O ₃ | Al | Pt+Al ₂ O ₃ liner | Al ₂ O ₃ crucible sprayed with Y ₂ O ₃ | Graphite |
|------------------------------|-------|--------------------------------|----|---|--|----------------|
| Silicon | No | No | ✓ | No | No | ✓* |
| Iron oxide | ✓ | No | ✓ | No | No | No |
| Lead oxide | No | ✓* | ✓* | ✓* | ✓* | No |
| Magnesium fluoride | ✓ | No | ✓ | No | No | No information |
| Calcium fluoride | ✓ | No | ✓ | No | No | ✓ |
| Copper oxide | ✓ | No | ✓ | No | No | No |
| Graphites | ✓* | ✓* | ✓* | ✓* | ✓* | ✓ |
| Carbonates | ✓ | ✓* | ✓ | ✓* | ✓* | No |
| Sulphates | ✓ | ✓* | ✓ | ✓* | ✓* | No |

✓ No reaction expected

✓* Reactions possible at high temperatures

No** Not recommended; reactions may occur prior to or during melting.

This could lead to damage to the crucible and/or sensor.

Extreme caution advised.

Recommendations for Cleaning Al₂O₃ and Pt Crucibles

Recommendations for Cleaning Al₂O₃ and Pt Crucibles

In most cases, the majority of ceramic and metallic crucibles and their lids can be reused many times. In order to ensure a long lifetime of proper use, the

following cleaning recommendations are given for the most common crucibles, made of alumina (Al₂O₃) and platinum (Pt).



Acids must be handled very carefully under a fume hood. Use protective glasses, gloves and apron and read the MSD sheets. Acids should only ever be handled by a person experienced in the handling of chemicals.

All acids (especially HF) are very dangerous and can cause extremely serious injuries or death if they come in contact with the skin or are inhaled.

Work with acids is at the user's own risk. NETZSCH can assume no liability for damage or injury resulting from the use of acids.

Al₂O₃ Crucibles

■ Contamination with organics (polymers, organic pyrolysis products, carbon black, etc.)

Heat crucibles in air or oxygen to approx. 900°C to burn off the organics.

If there are oxide fillers present, be careful with the end temperature, as there might be a reaction with alumina (lower the temperature and work in oxygen). Use a separate furnace, if available.

■ Contamination with metals and alloys

Clean with HCl acid (concentration 25% or higher). If there is no reaction with the contaminant, heat the beaker with the acid. If HCl alone does not work, use a mixture of HCl/HNO₃ (1:1) (both concentrated). This may be heated as well.

■ Oxides and other salts

Some salts are water soluble. Boil the crucibles in distilled water.

Use HCl or a mixture of HCl/HNO₃ (1:1) (both concentrated).

Oxides are often very stable or have already reacted with the alumina.

It could easily be the case that it is impossible to remove the deposits.

In that case, the crucible must be discarded. HF dissolves oxides, but also the alumina.

Following these cleaning procedures, rinse the crucibles several times with distilled water and let them dry at room temperature. Then heat them in a separate furnace in air to 1500°C.

Pt Crucibles

■ Contamination with organics (polymers, etc.)

Heat crucibles in air or oxygen to approx. 900°C to burn off the organics. If oxide fillers are present, attempt to remove them mechanically or use HF (see next procedure).

■ Contamination with metals and alloys

There is often no way of removing metals, as Pt will alloy with most metals at higher temperatures.

However, make an attempt with HCl acid (concentration 25% or higher).

If there is no reaction with the contaminant, try heating the beaker with the acid.

Do not use a mixture of HCl/HNO₃ (1:1). This would also dissolve the Pt!

■ Oxides and other salts

Some salts are water soluble. Boil the crucibles in distilled water.

Most oxides can be dissolved in HF.

In some cases, it is helpful to warm the acid.

Some salts can be dissolved in HCl.

The NETZSCH Group is a mid-sized, family-owned German company engaging in the manufacture of machinery and instrumentation with worldwide production, sales, and service branches.

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When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction) and the determination of Thermophysical Properties, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

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