

DAB-2 / DAB-3

Pressure Digestion System



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Subject to technical changes.

Trademark

Digestec® is a registered trademark of Berghof Products + Instruments GmbH.

This Manual

- describes the pressure digestion system Digestec from Berghof Products + Instruments GmbH. It contains product specific information, current as of the date of publication of this manual.
- guides the user in the safe and proper use of the pressure Digestec system. Familiarity with the relevant chapters of this handbook is required for safe and intended use of the equipment.
- should read the user carefully prior operating the equipment. Failure to follow these instructions could result in damage to the instrument as well as personal injury.
- does not include repair instructions. Should repairs be required, please contact your representative or Berghof Products + Instruments GmbH.

After unpacking, carefully check the equipment for mechanical damage and missing parts. Should you find any damage incurred during shipment, contact the manufacturer immediately and do not operate the equipment.

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1. General Notes

Please read this instruction manual carefully before operating your high pressure digestion system. Failure to follow these instructions could result in damage to the equipment as well as personal injury.

The operator is responsible for working being carried out in a safety-conscious manner avoiding risks.

Safety instructions are described in section 'Danger Categories and Signal Words'. These safety notes must be heeded in all cases!

1.1. Danger Categories and Signal Words

The signal words described below are used in connection with warnings throughout this manual. For your own safety and to avoid property damage, you must strictly heed these warnings!

The safety signals are printed in boldface or are otherwise marked throughout this manual and have the following meanings:



Means that failure to follow the safety instructions **is likely to cause** severe personal injury, death or severe property damage.



Means that failure to follow the safety instructions **may cause** severe personal injury, death or severe property damage.



Means that failure to follow the safety instructions **may cause** personal injury or some property damage.



This symbol indicates that this is important information regarding the product or refers to a part of the manual, which requires particular emphasis.

1.2. Qualified Users

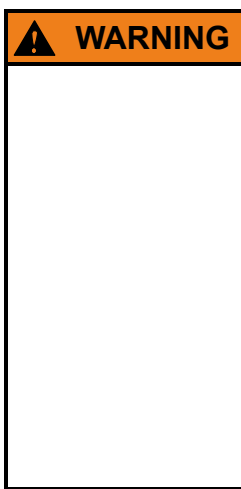
Only Qualified Users should operate this equipment. Qualified Users with respect to this manual are relevantly schooled and trained specialists with a basic knowledge of chemistry and technology. They should be trained in the relevant legal regulations for handling of chemicals.

1.3. Proper Use



Explosion hazard!

The unit is not designed for the use with substances / solutions, gas- / liquid mixtures or similar, which can form explosive mixtures and substances under operating conditions. (E.g., perchloric acid, solvent / air mixtures in certain concentrations, etc.)



Observe all operating instructions!

Always exercise particular care when handling pressurized vessels. Always comply with the operating instructions and all legal requirements as well as general safety regulations related to handling the individual chemical substances involved.

The operator may only permit knowledgeable and properly trained personnel to operate the pressure digestion system and its accessory equipment and must ensure that other employees or third parties are not endangered.

The operator must prepare appropriate operating instructions in order to ensure that personnel operating the pressure digestion system or equipment receive proper instruction.

All locally applicable regulations governing the operation of the pressure vessels must be complied with.



Observe all operating instructions!

Since sample composition is generally not precisely defined, a certain residual risk exists when performing pressure digestion processes. Careful working practices and conscious compliance with safety regulations can only minimize this risk, not totally eliminate it. Due to different degrees of mixing, even identical samples can exhibit totally different behaviors during the course of the reaction!

Please strictly comply with the operating instructions, particularly when handling the pressure vessels, that is, when removing and opening the vessels. Incorrect handling of the hot, pressurized vessels can result in injuries from escaping acid vapors!

→ Application area

The Digestec pressure digestion system has been designed to perform chemical digestion procedures under extreme pressure and temperature conditions in chemical laboratories. Digestion is understood to mean the decomposition of a solid material by means of a suitable digestion reagent at increased temperature in a vessel.

Digestion reagents used include nitric acid (65%), hydrochloric acid, hydrofluoric acid, phosphoric acid, and sulfuric acid, as well as mixtures of these acids. The maximum initial weight for organic compounds depends on the digestion vessel being used and the sample's carbon content, must always be observed.

By heating in the pressure vessel results in digestion temperatures in excess of 200 °C (392°F). The Digestec pressure digestion system has been specially developed for sample preparation for AAS and ICP spectroscopy procedures. Very temperature spikes >280 °C (>536°F) such as can occasionally occur during the course of exothermic reactions can result in irreversible damage to the PTFE-Inserts. Since such scorching cannot always be avoided, even with optimum heating regulation, damage of this kind is excluded from the warranty! The system may only be used within the context of data and applications specified in this documentation and the associated user manuals.

The pressure laboratory reactor may only be used:

- Properly
- In a technically flawless condition
- Without unauthorized alterations or modifications
- By qualified users

Please also observe all regulations published by professional/trade associations, the TÜV, VDE regulations, or corresponding national regulations.

1.4. General Safety Information

Using the pressure digestion system involves taking into account various safety precautions which go beyond those for general laboratory practices. The following general safety information should therefore be read carefully prior to using the system, and should be observed at all times.

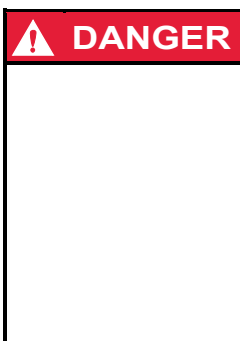
We can assume no liability for damages resulting from improper handling of a failure to comply with this information.



Explosion hazard !

Do not use perchloric acid.

Highly reactive substances which spontaneously react at higher temperatures or in the presence of oxidizing agents or which may be nitrated and are potentially explosive may not be digested. Examples of such substances include: explosives (TNT, etc.), perchlorates, ether, pyrophoric substances, etc.



Improper use!

Should a directly hazardous situation develop while the digestion system is in operation, e.g., as the result of an unexpected reaction or dangerous effects from the outside, all requisite countermeasures must be immediately initiated and, if necessary, the pressure vessels must be taken out of service.

A complete and detailed inspection of the pressure vessel must be performed if the operational fault resulted in the permissible pressure being exceeded by more than 10%, or if the permissible operating temperature was significantly exceeded, or if the pressure vessel or its fittings were damaged.

! DANGER**Pressure vessels!**

Use only digestion vessels and spare parts authorized by Berghof Products + Instruments GmbH for use with the Digestec. These can be obtained either from your local representative or directly from Berghof Products + Instruments GmbH (Refer to the "Repairs / Customer Service" section for contact addresses).

Always wear your personal protective equipment when working with the pressure vessels!

When using hydrofluoric acid especially appropriate safety equipment should be worn.

Stainless steel is not resistant against aqua regia. Due to diffusion of the acid through the PTFE-Insert the lifetime of the vessels will be reduced dramatically using aqua regia in the vessels DAB-2 or DAB-3.

Pressure vessel to be closed with torque wrench, using the stated locking force.

Note any changes in the appearance of the pressure vessel which could indicate possible material fatigue, e.g., deformation of the vessel, difficulty screwing on lids, etc. even when the vessel is cold and not under pressure. All such vessels should be immediately returned to Berghof Products + Instruments GmbH for inspection.

Never attempt to use force to open the vessels.

The pressure vessels are equipped with a rupture disc which produces a spontaneous pressure release upwards through the screw-on tube connections in case of excessive vessel pressure. In some cases, such a spontaneous pressure release may occur after the vessel has been removed from the heater.

The area above the pressure vessels should be marked as a danger zone, due to the possibility of the eruption of the bursting disc and the consequent release of hot acidic vapors.

The unit may never be employed without proper over-pressure protection (e.g., rupture disc). Corroded rupture discs shall be replaced in time.

Pressure vessel may be heated up to 250°C only! (Heating only with over-temperature safety device).

Open pressure vessel only in cold condition. Lid must be released and slacked (safety lock) before open quick-lock device.

Hot gases and vapors may be released from the digestion vessel when it is opened! Always make sure the vent opening on pressure vessels faces away from both yourself and other individuals!

Only open the digestion vessel under a fume hood or other exhaust system. Particular care is required when working with hydrofluoric acid (HF). Observe all relevant data sheets and safety instructions!

Never use more than one rupture disc per vessel!

The bayonet lock can only be securely locked after a 90° turn to the stop. Improperly locked bayonets can become unfastened during use and especially while opening.



Max. operating temperature and operating pressure!

The maximum interior temperature and maximum operating pressure may not be exceeded (refer to the 'Technical Specifications' section). Exceeding the maximum temperature will result both in a reduction of the vessel pressure resistance as well as possibly damaging the seals.

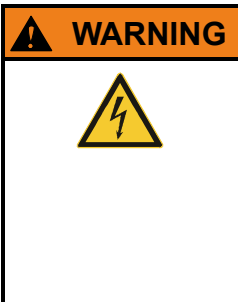


Accessories!

The Digestec pressure digestion system may only be used in conjunction with the specified heating and stirring systems.

Heating of the vessels may be done in Berghof heating blocks only. Never heat the pressure vessels in a drying oven; the pressure wave resulting from the eruption of the rupture disc may lead to damage of the oven.

Never allow liquids, particularly toxic compounds, to spill on hot parts.



Accessories use low voltage 230V!

Unit must always be carefully connected to a ground conductor. Provided the wall outlet used is equipped with a ground connection, the supplied power cord will ensure proper connection. If it is necessary to use an extension cord to connect the system to the power supply, only a three-wire cord with a ground connection may be employed.

Repairs and service may only be performed by properly trained and qualified personnel.



Only use approved supply cords!

Do not exchange the detachable mains supply cord with a cord of inadequate rating. Use only approved and undamaged supply cords.



Ventilation

Users are responsible for assuring that appropriate ventilation of toxic gases is assured.

Make sure that all ventilation procedures conform to applicable laws.

1.5. Legal Requirements



The user is responsible for ensuring that the current regulations are respected. The set of rules in this chapter is only an overview.

Within the European Union, 2014/68/EU (Pressure Equipment Directive) and 2014/29/EU (Simple Pressure Vessels Directive) bring the various national technical requirements into line. In the German Code, these have been included in the 6th and 14th Equipment and Product Safety Act. This enacted harmonized characteristics requirements. However, harmonized technical rules or adapted national construction requirements (e.g. AD-2000 in Germany) may also apply.

Manufacturers of pressure equipment are free to select among these regulations based on their countries!

1.5.1. Recurring inspection, Responsibility for technical inspection, maximum inspection periods according BetrSichV 2015

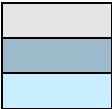
The laboratory reactor can be operated either as a pressure vessel or as an autoclave. The classification must be carried out by the operator.

The following applies to pressure vessels:

Vessel	Test-group	V, [Liter]	PS, [bar]	PS*V, [bar*Liter]	§15 Examination for commis-sioning	§16 Recurring inspections		
						External Inspection	Internal Inspection	Strength Test
DAB-2		0,05	200	10				
DAB-3		0,2 0,42	200	40 84				

Table 1: Pressure vessels

Legend:



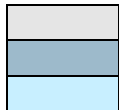
No test group according to BetrSichV
Person qualified for examination
Approved body according to §37 ProdSG

The following applies to experimental autoclaves:

Vessel	Test-group	V, [Liter]	PS, [bar]	PS*V, [bar*Liter]	§15 Examination for commissioning	§16 Recurring inspections		
						External Inspection	Internal Inspection	Strength Test
DAB-2		0,05	200	10				
DAB-3		0,2 0,42	200	40 84				

Table 2: Autoclaves

Legend:



No test group according to BetrSichV
Competent person
Approved body according to §37 ProdSG

After **each** use, the experimental autoclave must be examined by a competent person.

The special risk involved with pressure vessels has led to them being classified in the German Equipment and Product Safety Act (ProdSG) under “systems requiring monitoring”.

Under Section 3 of the BetrSichV 2015, ‘Additional requirements for systems requiring monitoring’:

The operator is to ensure that the result of the inspection and testing are documented according to §§ 15 and 16. If the inspection and testing is done by an approved monitoring office, a certificate of inspection for the result of the inspection is to be requested. At minimum, records and inspection certificates must give information about:

- System ID
- Inspection and testing date
- Type of inspection and testing
- Basis of inspection and testing
- Scope of inspection and testing
- Effectiveness and functioning of the protective measures
- Result of the inspection and testing
- Date until next required inspection and testing according to § 16 paragraph 2.

If the pressure vessel was manufactured in accordance with the national regulations of the countries listed below, the following inspection periods apply.

Recurring inspection								
	D	F	B	I	L	NL	GB	S
In years	5*	1,5-3	1-3	1-2	5	4	2,17	3

See Table 1 and 2

Operation of pressure equipment is regulated by the European Union's individual member states. In Germany, this is regulated by the Ordinance on Industrial Safety and Health, supplemented by a secondary body of technical rules.

The operator must comply with all locally applicable operating regulations. With regard to the recurring inspections, operators within the European Union may choose to abide by either their national requirements or by the German requirements. This is possible because the reactor has been manufactured in accordance with the AD-2000 body of rules and these rules are recognized throughout the EU. Therefore, an inspection period of 5 years applies to all Berghof pressure laboratory reactors employed within the European Union.

Regarding competent persons

The technical regulations for operating safety, TRBS 1203, specify the requirements for the necessary expertise needed by a person qualified under § 2, Abs. 7 BetrSichV.

Vocational training:

For the purposes of this Ordinance, a competent person means a person who has the technical knowledge necessary for inspecting the work equipment because of his/her vocational training, professional experience and current activity on the job.

Professional experience:

Professional experience requires that the qualified person has used the equipment to be tested and inspected in practice for a proven length of time in a professional environment and knows the functions and operation of it to the necessary extent. Furthermore, there were sufficient occasions to actually undertake the inspections and testing, e.g., as a result of a risk assessment and from daily observation.

Participation in inspections and testing of work equipment has led to an accumulation of experience concerning the performance of the inspections or similar tests, and the necessary knowledge for handling the testing instruments as well as evaluating the test results has been acquired.

Professional experience includes the ability to evaluate whether a recommended testing procedure is suitable for inspecting and testing the work equipment. This also includes detecting hazards involved through the testing activity and the work equipment to be tested.

Recent professional activity:

Recent professional activity in the sense of § 2 Abs. 7 BetrSichV includes activity in the context of the forthcoming inspection and testing as well as adequate training.

The execution of multiple inspections and testing per year (keeping testing skills up-to-date) belongs to recent professional activity.

In the event of lengthier interruptions of the inspection and testing activity, experience in inspection and testing should be renewed and necessary technical skills refreshed by participating in inspection and testing done by other parties.

The competent person must have knowledge about the state of the art regarding the work equipment to be tested and the hazards which might occur and keep this knowledge up-to-date. Familiarity with the operational safety regulations and their technical regulations and other statutory occupational safety regulations (e.g., ArbSchG, GefStoffV) and the associated technical regulations as well as regulations with requirements relating to the properties (e.g., ProdSG, relevant ProdSV), with provisions of the accident insurance carrier and other regulations (e.g., standards, recognized test principles) is required so that the safe state of the work equipment can be properly evaluated.

Additional qualifications for inspecting pressure systems that require monitoring according to Annex 2, Sec. 4, No. 3 BetrSichV:

- Relevant technical vocational training.
- At least one year experience with manufacturing, assembling, operating or maintaining the equipment or system components to be inspected and tested.
- Current state of knowledge about pressure hazards by participation in a training or instruction.

1.5.2. Categories (Test group)

The pressure equipment referred to in Article 4 (1) of PED 2014/68/EU shall be classified by category in accordance with Annex II to the increasing risk potential.

The reason for this classification lies in the energy stored in the pressure vessel. This energy can be expressed as the pressure content product.

- Pressure content product: $P \times V = [\text{bar} \times \text{l}]$; (1 bar x l for gases, this corresponds to approx. 100 Joules)
- Charging medium (fluid): Gases store a greater amount of energy than liquids. However, liquids heated beyond their boiling point store additional energy in the form of heat.

Pressure equipment is subdivided into the following categories on the basis of this pressure content product and the charging media (fluid):

Application of the categories of danger according to the CLP regulation

Assignment of the substance properties to fluid groups:

Fluid group 1:

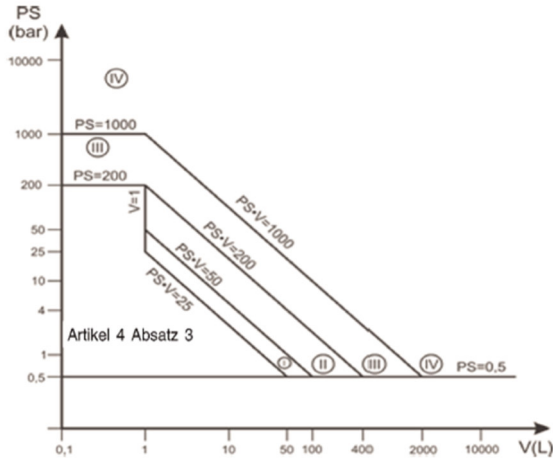
- explosive substances / mixtures according to Annex I, No. 2.1.
- flammable gases according to Annex I, No. 2.2.
- oxidizing gases according to Annex I, No. 2.4.
- flammable fluids according to Annex I, No. 2.6 (category 3 fluids only if $TB > TFl$ and $TFl < 55^\circ \text{C}$).
- pyrophoric fluids according to Annex I, No. 2.9.
- oxidizing fluids according to Annex I, No. 2.13.
- Acute toxic substances according to Annex I, No. 3.1.2 Categories 1 and 2.

Fluid group 2: all substances which do not belong to fluid group 1.

Because the pressure laboratory reactors described in this manual are intended for use in chemical laboratories, they generally fall under Group 1 – Hazardous Fluids!

As the following diagram illustrates, the Berghof pressure laboratory reactors with a volume smaller than 1000 ml and a max. operating pressure of 200bar cannot be assigned to any specific category. These must be designed, manufactured, and tested in accordance with Art. 4 Para. 3 (PED 2014/68/EU). Such reactors do not require CE certification and may not carry a CE symbol.

→ [Conformity Assessment Diagram](#)



1.5.3. TÜV/CE Mark

The minimum requirements for the conformity evaluation procedure can be deduced from this category.

Berghof Products + Instruments GmbH applies the following process for this purpose:

- EC type examination test: By the TÜV as an authorized testing center
- Conformity with design: Pressure test of every reactor by the TÜV as an authorized testing center

This methodology ensures that all reactors are designed, manufactured, and tested in accordance with the directive 2014/68/EU and the AD 2000 body of rules. This is confirmed by the TÜV seal, the conformity declaration and, to the extent that the reactors may be so designated, by the CE symbol.

1.5.4. Operation



If the reaction progression is not precisely known or if unanticipated pressure increases or exothermic reactions may occur (test autoclave), the unit must be set up in a special reactor chamber.

→ Operators Responsibilities

Based on the classification into the above-cited categories, the following obligations arise for the operator
§ 15 Inspection prior to putting into service, § 16 Recurrent inspections:

- The recurring inspections of test autoclaves must be performed by an approved body if the product of the maximum permissible pressure, P, and the relevant volume, V, is greater than 100 bar x liter.
- The recurring inspections must be performed at least every 5 years.

This test includes:

- Check the documentation (manufacturing documents, test book)
- Internal inspection (the determination of the actual state, the comparison of the actual state with the desired state, and the evaluation of the deviation of the actual state from the desired state)
- Functionality
- Pressure test

Special test regulations according to BetrSichV Annex 2, Section 4, No. 6.19:

In the case of pressure vessel and connected pipelines for flammable gases and gas mixtures in liquid state, which are applied to the walls of the vessels and pipelines

- which have corrosive effect, external tests must be carried out every two years by an approved body
- which have no corrosive effect, external tests must be carried out every two years by a competent person.

Experimental autoclaves for which the expected pressures and temperatures during experiments are not known with certainty are to be set up in specially protected chambers or behind safety walls so that they are protected from outside exposure and so that employees and others are safe in the event that the experimental autoclaves fail. There are safety precautions in this pressure equipment for experimental purposes which do not help in the event that pressure and temperature becomes excessive. Watching the safety and measurement equipment and operating the experimental autoclaves must be done in a safe place.

Measures for protecting persons have been undertaken to avoid extreme heating by the heat load which could result in failure of the pressure-loaded wall sections, equipment parts relevant for safety and/or supporting components of pressure equipment or its parts.

Protective measures are, e.g.:

- Safety distance
- Safety wall
- Ground cover
- Fire protection insulation
- Fixed fire extinguishing systems
- Sprinkler systems

The recurring inspections of experimental autoclaves must be performed by an approved body if the product of the maximum permissible pressure, P , and the relevant volume, V , is greater than 100 bar x liter.

1.6. Operating regulations and Characteristic Requirements

1.6.1. National Regulations, Laws, Ordinance, Technical Rules

- Ordinance on Industrial Safety and Health concerning the protection of safety and health in the provision of work equipment and its use at work (Ordinance on Industrial Safety and Health - Betriebssicherheitsverordnung – BetrSichV)
- Hazardous Substances Ordinance (Gefahrstoffverordnung – GefStoffV)
- Act on making products available on the market (Product Safety Act - Produktsicherheitsgesetz – ProdSG)
- Fourteenth Ordinance on the Product Safety Act (Pressure Equipment Regulation - 14. ProdSV)
- TRBS 1201 Part 2: Tests for hazards caused by steam and pressure
- TRBS 1203: Competent persons
- TRBS 2141: Risk posed by steam and pressure - General requirements
- TRBS 2141 Part 3: Risk posed by steam and pressure upon release of media

1.6.2. International regulations

- Directive 2014/68/EU of the European Parliament and of the Council of 15 May 2014 on the harmonisation of the laws of the Member States relating to the making available on the market of pressure equipment
- Directive 2014/29/EU of the European Parliament and of the Council of 26 February 2014 on the harmonisation of the laws of the Member States relating to the making available on the market of simple pressure vessels
- Corresponding national provisions

1.6.3. Applicable Documents

AD 2000 code, in particular:

- Datasheet HP 30: Execution of pressure test
- Datasheet HP 512 R: Construction regulations - Design examination, final testing and pressure testing of piping

1.7. Safety

Every Berghof pressure digestion system has been tested at least 1.6 times the maximum operating pressure, with a corresponding certificate included in the delivery.

In the following situations, the unit must be disconnected from the mains power supply and be locked out to prevent accidental use:

- If the unit evidences visible signs of damage
- If unit parts or components are found to be loose
- If the unit fails to operate
- If the unit is to be stored for extended periods of time under unfavorable conditions (e.g., outdoors, in very moist or humid environments)



The housing cover may only be removed by authorized trained personnel!
The instrument must be disconnected from the power outlet before being serviced.

1.7.1. Safety information on the unit



Warning of dangerous electrical voltage!

This label is located on the bottom of the heating block (DAH)

1.8. Information on Disposal

Disposal within
the EU

European Community

Electrical and electronic products should not be mixed with general household waste. Berghof Products and Instruments GmbH accept its electrical and electronic instruments from on a free of charge basis for proper treatment, recovery and recycling from their business users. If you wish to discard electrical and electronic products, please contact your local representative or Berghof Products and Instruments GmbH (laboratorytechnology@berghof.com). They will provide further information. Disposing of this product correctly will help to protect our environment.

→ Disposal outside
the EU

Countries outside the European Union

This regulation is only valid in the European Union

 NOTICE

Products of Berghof Products + Instruments GmbH are **not** used in private households.

1.9. Warranty / Limitation of Warranty

Limited Warranty

Each product manufactured by Berghof Products + Instruments GmbH is warranted to conform to Berghof Products + Instruments GmbH's applicable specifications on the date on shipment. The warranty period is twelve (12) months after the date of delivery, unless another period is specified. The warranty does not extend to damages due to improper installation, improper maintenance, abuse, accident, negligence, alteration, misuse, ordinary wear and tear, or the like. Claims for replacement of parts caused by ordinary wear and tear are excluded, as are claims for replacement of parts meant to be expended during the operation of the equipment.

Remedies

Under the limited warranty, Berghof Products + Instruments GmbH repairs or replaces any products which Berghof Products + Instruments GmbH determines to be defective and covered by this limited warranty. This is the sole and exclusive remedy. If Berghof Products + Instruments GmbH determines that repair or replacement fails its essential purpose, the purchaser will, at its option, be entitled to a refund of the purchase price for the products in question or a credit therefore.

All warranty claims must be accompanied by a description of the claim, which description must be attached to the equipment claimed to be defective. Claim descriptions must include the claimants name, address, department (if applicable) and telephone number.

The equipment should be returned in its original packaging, to the extent possible. We regret that Berghof Products + Instruments GmbH cannot be held responsible for damage caused during shipping due to improper packaging.

Limitation of warranty

Except as expressly set forth above, there are no other warranties hereunder, whether expressed or implied, arising by operation of law or otherwise, including without limitation, the warranties of merchantability, fitness for a particular purpose and any warranties arising under course of performance, course of dealing or usage of trade.

Under no circumstances will Berghof Products + Instruments GmbH be liable for any consequential, exemplary, incidental, indirect or special damages, or lost profits, expenses or losses arising out of or related to the sale or use of any of its products regardless of whether the liability resulted from any general or particular requirement or need which Berghof Products + Instruments GmbH knew or should have known of.

1.9.1. Caveat emptor

Berghof Products + Instruments GmbH does not accept any warranty for wearing parts (e.g. PTFE-Insert, Rupture disc, Belleville spring), for the improper usage, storage and handling.

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2. Device Description

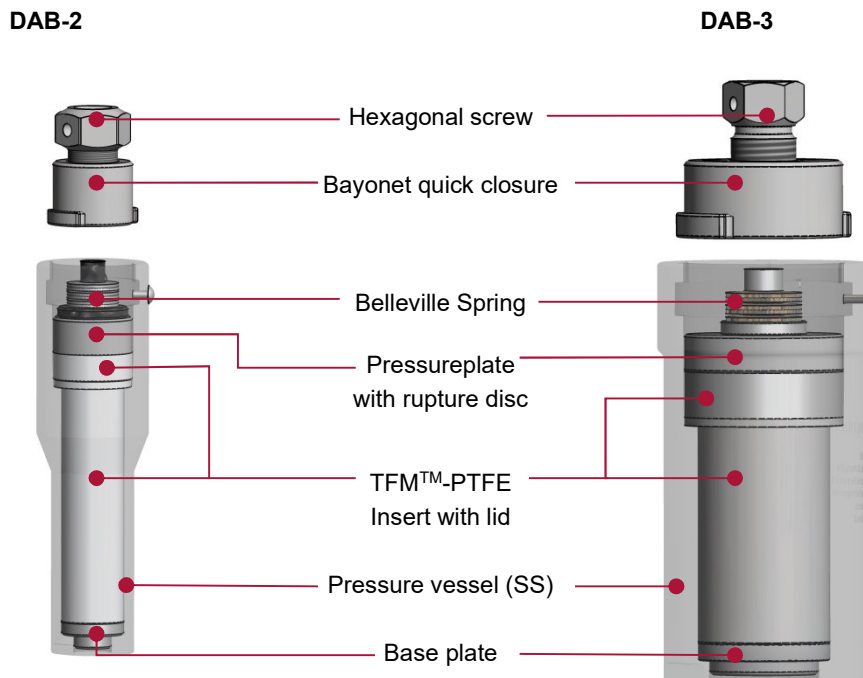
2.1. Basic Instrument

The pressure vessel consists of a cylindrical base body closed at the bottom with a movable base plate and above with a pressure plate with rupture disc as well as a bayonet-like quick-lock device. All metal parts are made of an alloyed CrNiMo stainless steel. Inside the pressure vessel is an exchangeable PTFE-liner with lid, forming a dense system. This liner is manufactured of a pure and high-quality TFM™-PTFE by means of a special pressing process. This PTFE material impresses as it is almost non-porous, universal chemical resistant and has an equal heat expansion.

Inside this system the weight-in sample and the added digestion acid come only in touch with the PTFE-material. By the outer pressure vessel the form of the PTFE-Insert is kept even also at high pressure (max. 200 bar) and high temperature (max. 250° C). The pressure vessel is secured against too high pressure loading by means of a rupture disc. This rupture disc is installed in the pressure plate and lays upon the set-point of fracture of the PTFE -lid.

The quick-lock device system consists of a bayonet-like head piece through which is leading a hollow hexagonal screw, pressing by means of Belleville spring and pin upon the pressing plate and thus also upon the PTFE lid.

For simple and safety closing and opening of the autoclave serves a mounting tool (see also chapter 2.3.1 Mounting Tools). With the aid of the torque wrench the pressure vessel is closed by means of tighten the hollow hexagonal screw until the adjusted locking pressure is reached.



2.1.1. Technical Specification

Digestion vessel					
Parameter	DAB-2			DAB-3	
Material	SS 1.4571 (SS316 TI), TFM™-PTFE			SS 1.4571 (SS316 TI) TFM™-PTFE	
Height	189 mm			194 mm	275 mm
Exterior dimensions Ø	54 mm			98 mm	
Weight	1,31 kg			5,18 kg	7,67kg
max. temperature	250 °C (482°F)			250 °C (482°F)	
max. pressure	200 bar (2900psi)			200 bar (2900psi)	
Closing force	25 Nm			25 Nm	
Overpressure protection	Rupture disc			Rupture disc	
Inliner (Seal)	V	V	L	V	L
Volume	25 ml	49 ml	53 ml	210 ml	420 ml
max filling volume	max. 80 % of volume				
max. sample weight (organic)	< 800 mg			< 2000 mg	

2.2. Safety Equipment

Rupture discs

All vessels are equipped with a rupture disc which produces a spontaneous pressure release upwards through the screw-on tube connections in case of excessive vessel pressure. In some cases, such a spontaneous pressure release may occur after the vessel has been removed from the heater.

 WARNING	Rupture discs as overpressure protection! Use only original Berghof rupture discs and protective cover sheets. These can be obtained from your local, authorized representative or Berghof Products + Instruments GmbH (Refer to the “Repairs / Customer Service” section for contact addresses). The area above the pressure vessels should be marked as a danger zone, due to the possibility of the eruption of the bursting disc and the consequent release of hot acidic vapors. The unit may never be employed without proper over-pressure protection (e.g., rupture disc). Corroded rupture discs shall be replaced in time. Never use more than one rupture disc per vessel!

2.3. Accessories

2.3.1. Mounting Tools

For simple and safety closing and opening of the autoclave serves a mounting tool. It consists of a mounting base plate and a torque wrench with plug-in nut (hexagonal). The mounting base plate serves to fix the pressure vessel by means of the two milled surfaces and the plug lifts the mobile base plate for ejection of the PTFE -Insert.



No.	Description
1	Socket
2	Torque wrench
3	PTFE-Paste
4	Brush
5	Spring replacement mandrel
6	Face spanner
7	Base plate for DAB-2 und DAB-3

2.3.2. Heating Block and Hot Plate Stirrer

Thermo-insulated heater block mount made of aluminum with stainless steel safety hood.

The heater block mount is intended for use in conjunction with the BLH-800 laboratory heating plate. The temperature can be controlled either by means of the thermal regulator integrated in the heating plate (external temperature regulation), or by measurement of the interior temperature in conjunction with the BTC-3000 temperature regulator. A special connecting cable is provided to allow the BTC-3000 to control the laboratory heating plate.

A magnetic stirrer has also been integrated into the laboratory heating plate to permit the reactor contents to be stirred using a conventional magnetic stirring rod.

Technical Data

Heating block			
Parameter	DAH-3	DAH-4	
Material	Aluminum with stainless steel protective cover		
Dimension B x L x H	180 x 180 x 140 mm 7,1 x 7,1 x 5,5 inch		
Appropriate heating plate Ø	145 mm / 5,7 inch		
Weight without digestion vessel	4, 0 kg	4,9 kg	
Reception of vessel	1 x DAB-3	4 x DAB-2	

Hot plate stirrer		
	BLH-650	

See attached original manufacturer's operating instruction



2.3.3. Electric Heating block

Electric heating block with a heating ring and coated stainless steel safety hood as well as an integrated over-heat protection, adjustable between 50 – 300°C. The heating block exterior is insulated and clad.

Technical Data

Heating block for DAB-2			
Parameter	DAH-406	DAH-412	
Heating power	1000 W	2000 W	
Designed for digestion vessel	6 x DAB-2	12 x DAB-2	
Operating temperature	0 - 350° C (32 – 662 °F)		
Overtemperature protection	+50 - +320°C		
Temperature sensor	NiCr/Ni DIN 43710		
Connection to heater	to control unit 230 V / 50 Hz via 3-pin plug connection		

Heating block for DAB-3			
Parameter	DAH-902	DAH-904	
Heating power	1000 W	2000 W	
Designed for digestion vessel	2 x DAB-3	4 x DAB-3	
Operating temperature	0 - 350° C (32 – 662 °F)		
Over temperature protection	+50 - +320°C		
Temperature sensor	NiCr/Ni DIN 43710		
Connection to heating	to control unit 230 V / 50 Hz via 3-pin plug connection		

2.3.4. Temperature control with BTC-1000 or BTC-3000

The temperature is controlled by measurement of the interior temperature of the electric heating block in conjunction with a BTC-1000 or BTC-3000 temperature regulator. A special connecting cable is provided to supply power to the heating block through the BTC-1000 or BTC-3000.
(see chapter Setup and Commissioning)

BTC-1000



BTC-3000



Detailed description see manual BTC-1000 or BTC-3000.

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3. Setup and Commissioning

3.1. Standard Delivery

Because of the modular nature of the Digestec pressure digestion system, there is no generally applicable standard delivery. Therefore, please check the shipping documentation with regard to the completeness of your specific unit.

3.2. Transport and Storage

The Digestec pressure digestion system may only be moved once all pressure has been released. Please observe the ambient conditions described below.

3.3. Ambient Conditions

Pressure equipment must be set up in such a way that neither operators nor others are endangered. All safety zones mandated by national ordinances in the country of use must be complied with.

The pressure laboratory reactor must be set up in such a way that:

- It is accessible and can be viewed from all sides for any required visual inspections
- The seal (nameplate) is clearly visible
- The pressure reactor and its accessories can be operated from a safe and secure position
- It is protected against mechanical influences from the outside and that damage to the unit cannot be anticipated
- Access by unauthorized individuals is not possible

The pressure digestion system is appropriately installed in a separate room, possible to be ventilated, or under an outlet. The zone above the pressure vessel is endangered during pressure wave (with particles and digestion acid) is up warding. The pressure vessel should therefore be installed in such way, so as nothing can be damaged in case of a rupture.



If the reaction progression is not precisely known or if unanticipated pressure increases or exothermic reactions may occur (test autoclave), the unit must be set up in a special reactor chamber.

The setup location must meet the following requirements:

Ambient conditions	
Ambient temperature	+10°C (50°F) to +40°C (104°F) during operation -40°C (-40°F) and +70°C (158°F) during shipping and storage
Max. rel. humidity	85%
Load bearing capacity	please refer to the shipping documents
Electrical connection	90- 240 V; 50/ 60 Hz

3.4. Unpacking and Inspection

Open the shipping packaging and carefully remove it. Please save all packaging in order to be able to return the unit to the manufacturer in its original packaging, should service be required.
 Also remove all other components and accessories and inspect the delivery to make sure it is complete.
 Inspect the system for shipping damage such as cracks, scratches, dents, etc.

The Berghof DAH heating blocks are subjected to a quality control before delivery. During the necessary heating process, the inner metal block becomes discolored. This color change is technically unavoidable and is not a quality defect.


WARNING

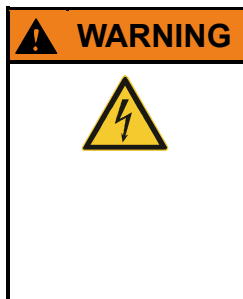
Visible damage!

For safety reasons, never operate the unit if it exhibits visible signs of damage. Contact your local, authorized representative or Berghof Products + Instruments GmbH to obtain the required service (Refer to the “Repairs / Customer Service” section for contact addresses).


NOTICE

Should any components be missing or damages be detected, contact your local, authorized representative or Berghof Products + Instruments GmbH directly (Refer to the “Repairs / Customer Service” section for contact addresses).

3.5. Installation



Accessories use low voltage 230V!

The heating and stirring systems must always be carefully connected to a ground conductor. This is ensured by the included power supply unit, provided that the wall outlet employed is connected to a corresponding ground circuit. If an extension cord must be used to connect the system to the mains power supply, make sure it is a 3-wire cord with a ground wire.

Repairs and service may only be performed by properly trained and qualified personnel.

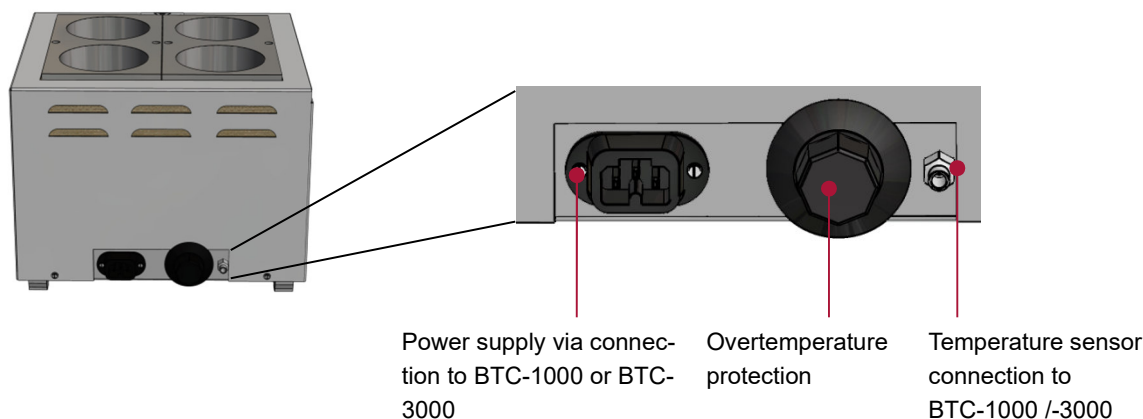
Positioning

First, position the accessories, particularly the heating system, if these items were part of your delivery. Install the digestion system in the heater and position the complete system on the intended work surface. The work surface must be adequately, statically dimensioned for the weight of the pressure digestion system (refer to the `Ambient Conditions` section).

Installation

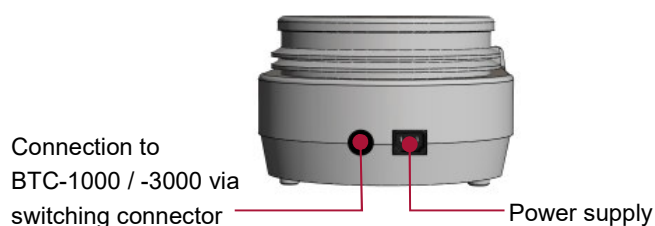
Using the connecting cables provided, connect the accessories as illustrated. As a final step connect all devices to the power supply.

Heating with heating block DAH-406, 412, 902 or 904

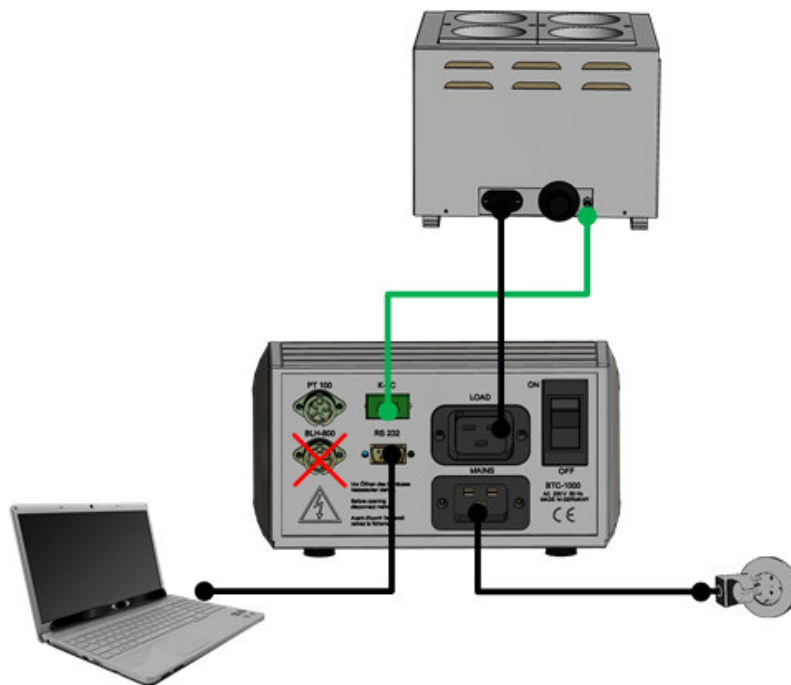


OR

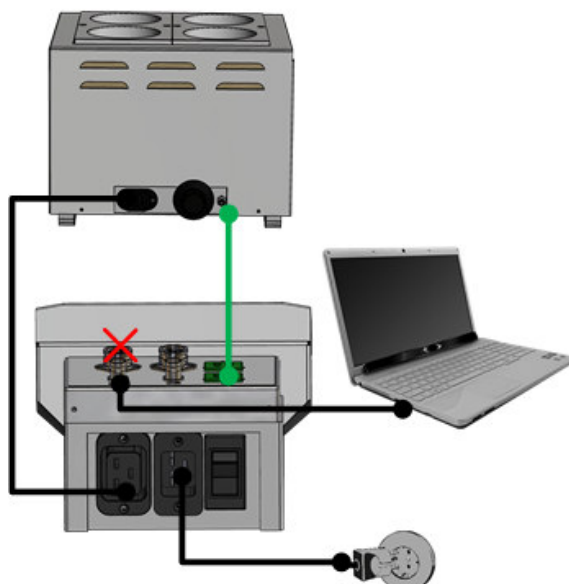
Heating with Hot plate stirrer BLH-650



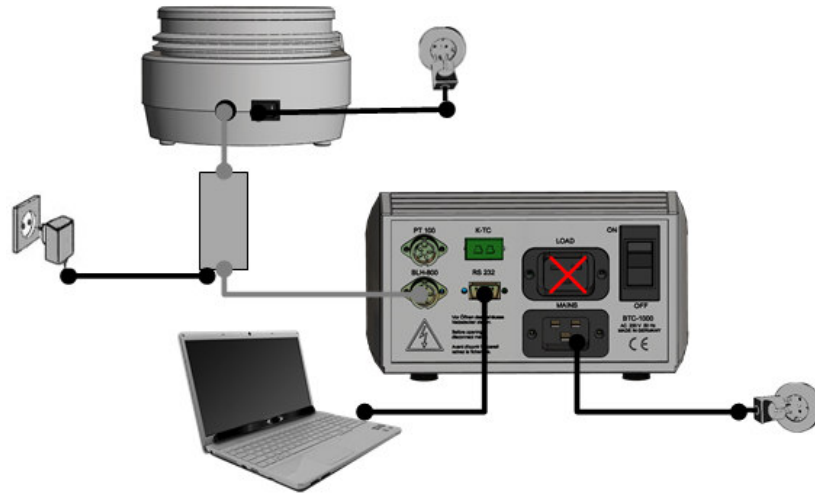
a) Installation with Heating block and BTC-1000



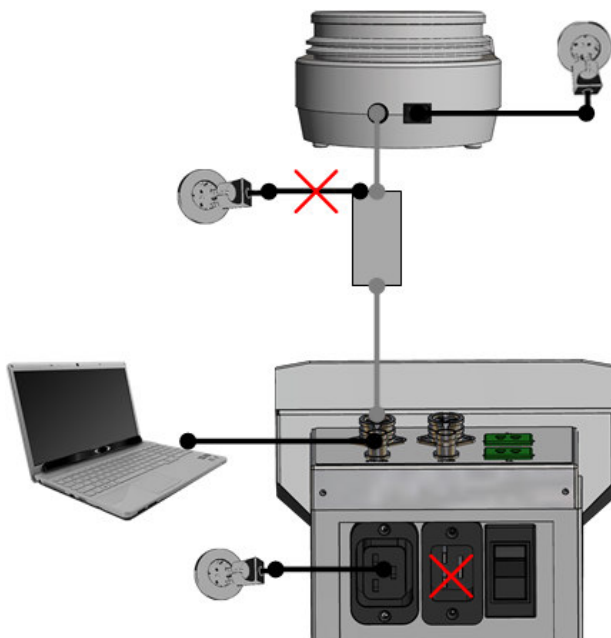
b) Installation with Heating block and BTC-3000



c) Installation with Hotplate stirrer and BTC-1000



d) Installation with Hotplate and BTC-3000



i NOTICE

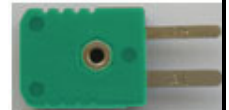
Connection BLH-650

Power supply of BLH-650 must always be connected to mains. Never connect the power supply of BLH-650 with power outlet of BTC-3000 to prevent any damages on BLH-650. See installation version d).

i NOTICE

Control via BTC-3000

If T2 is not connected to a Temp. sensor, T2 must be short-circuited with the supplied short circuiting-plug (see picture)



i NOTICE

Control via BDL-3000

In case that BLH-650 is controlled by BDL-3000 or BTC-3000 the temperature switch setting of BLH-650 functions as over temperature protection. Therefore this temperature switch should not be set to higher values than necessary to achieve additional safety. Please select temperature settings on BLH-650 which are approx. 50°C above requested operational temperature.

Mounting tool

The mounting tools should be used for a safe opening and closing of the pressure vessel. For that purpose the mounting base plate can be firmly screwed on a working table. (see chapter “Operation”)

The system is now ready for use

4. Operation

4.1. Opening and Closing

4.1.1. Information Regarding Digestion

Heating the acid mixture, results in the creation of vapor pressure. The degree of this vapor pressure depends greatly on the temperature. All digestion vessels have a maximum permissible pressure. If this maximum pressure is exceeded, the rupture disc in the pressure vessel activates, providing a rapid pressure release. In addition to the vapor pressure, reaction gases (primarily carbon dioxide generated by samples containing carbon) also produce pressure. This pressure is therefore dependent on the sample amount, the vessel volume, as well as the carbon content of the sample. → [Influence of the sample on the vapor pressure](#)

The weight-in quantity of the sample to be digested depends on the volume of the TFM™-PTFE -Insert and the kind of sample matrix as well as on the quantity of the digestion acid needed. Table 1 is assisting you hereto. As a rough guide can be stated: 1 g sample and 10 ml digestion acid per 100 ml volume of the vessel, for smaller PTFE-Insert eventually somewhat more, for larger volumes somewhat less. There can also be more weighted-in of inorganic and wet matrices than of dry and organic sample materials. Especially when working with samples tending to exothermal reactions with the digestion agent (plastic, fat, oil) there should rather be too less weighted-in. For inorganic samples, which cannot totally be decomposed, an increased acid addition can eventually be of use. Generally, the sample is weighted-in in grinded condition (large surface). Oily substances can be attached also on filter paper.

There are no limits set to the composition of the digestion acid mixture. Generally, it is tried to achieve a completed decomposition with pure HNO_3 (65 %). In case that HNO_3 is not sufficient enough (remainders in the digestion solution), there can also be added HF to the solution of mineral shares or H_2SO_4 (abstraction of water).

For digestion of inorganic samples, which must not be oxidized, HF can be used pore or together with HCl and H_2SO_4 . However, before using HClO_4 please note that it must be dissuaded because of is danger. When preparing organic samples it must be regarded that the oxidizing decomposition agent is available in surplus. The selection of the digestion acid or the combination of acid mixtures should also be made in respect to the chemistry of sample contents and the difficulty of subsequent elementary analysis methods.



Explosion hazard!

The unit is not designed for the use with substances decomposing / solutions, gas- / liquid mixtures or similar, which can form explosive mixtures and substances under operating conditions. (E.g., perchloric acid, solvent / air mixtures in certain concentrations, etc.)



Reaching the maximum pressure!

Please note that the reaction pressure is only reached during the course of the reaction, in other words, several minutes after the maximum temperature has been reached. Therefore, an activation of the rupture discs may be anticipated even after the temperature has reached a constant level.



Rupture disc corrosion!

Depending on the acid being employed and the digestion parameters, the rupture discs are subject to more-or-less extreme wear. Replace the discs regularly to prevent the discs from rupturing prematurely.

4.1.2. Developing a new application

Digestion of new sample material, that is, of samples for which you have no previous experience, requires a special, systematic procedure. We recommend the following process when developing such an application:

- Check the “Appendix” to determine whether the application list already contains work instructions for the sample material in question or for a similar material then, if necessary, request a copy of the original process instructions from your local, authorized representative or from Products + Instruments GmbH.
- Should you not be able to find your sample material, search through the relevant technical literature for a corresponding process instruction. You can also request assistance from your local, authorized representative or from:
Products + Instruments GmbH.
- For safety reasons, initially reduce the specified sample amount by 30-50 %, while still using the specified acid quantity and digesting with the defined temperature program.
- Optimize the acid mixture and the temperature profile until you obtain a clear solution.

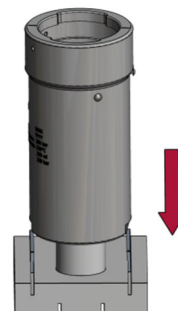
In the final step you can gradually increase the sample amount until the higher pressure causes the rupture disc to activate. The maximum sample quantity to be used should be approx. 30 % of this amount.

4.2. Feeding Digestion Vessel

DANGER	Pressure vessels! Use only digestion vessels and spare parts authorized by Products + Instruments GmbH for use with the Digestec. These can be obtained either from your local representative or directly from Products + Instruments GmbH (Refer to the “Repairs / Customer Service” section for contact addresses). Always wear your personal protective equipment when working with the pressure vessels! Stainless steel is not resistant against aqua-regia. Due to diffusion of the acid through the PTFE-Inserts the lifetime of the vessels will be reduced dramatically using aqua regia in the vessels DAB-2 or DAB-3. Pressure vessel to be closed with torque wrench, using the stated locking force. Note any changes in the appearance of the pressure vessel which could indicate possible material fatigue, e.g., deformation of the vessel, difficulty screwing on lids, etc. even when the vessel is cold and not under pressure. All such vessels should be immediately returned to Products + Instruments GmbH for inspection. Never attempt to use force to open the vessels. The pressure vessels are equipped with a rupture disc which produces a spontaneous pressure release upwards through the screw-on tube connections in case of excessive vessel pressure. In some cases, such a spontaneous pressure release may occur after the vessel has been removed from the heater. The area above the pressure vessels should be marked as a danger zone, due to the possibility of the eruption of the bursting disc and the consequent release of hot acidic vapors. The unit may never be employed without proper over-pressure protection (e.g., rupture disc). Corroded rupture discs shall be replaced in time. Pressure vessel may be heated up to 250°C only! (Heating only with over-temperature safety device). Open pressure vessel only in cold condition. Lid must be released and slacked (safety lock) before open quick-lock device. Hot gases and vapors may be released from the digestion vessel when it is opened! Always make sure the vent opening on pressure vessels faces away from both yourself and other individuals!! Only open the digestion vessel under a fume hood or other exhaust system. Particular care is required when working with hydrofluoric acid (HF). Observe all relevant data sheets and safety instructions! Never use more than one rupture disc per vessel! The bayonet closure can only be securely locked after a 90° turn to the stop. Improperly locked bayonet closures can become unfastened during use and especially while opening.
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4.2.1. Removal of the PTFE-Insert

For feeding purpose the PTFE-Insert is taken out of the pressure vessel by means of putting the opened vessel on the pin of the mounting base plate.



This pin is pressing the inlet up warding by means of the movable base plate.



4.2.2. Sample and acid addition

Weigh the sample material into the vessels, and then add the acid.



The maximum sample quantity depends on the vessels employed and must be strictly observed (refer to the "Technical Specifications" section).



Minimum fill amount!

Depending on the pressure vessel, at least approx. 5ml of liquid should be employed.

4.2.3. Closing the pressure vessel

All of the following steps apply to DAB-2 as well as to DAB-3:

After the PTFE-Insert has been fed with sample material and digestion acid, the PTFE-Insert is inserted together with the lid into the pressure vessel.



An aluminium foil is put on the lid in order to protect the thin rupture disc against aggressive acid fumes.



The Rupture disc holder is now inserted into the pressure vessel.

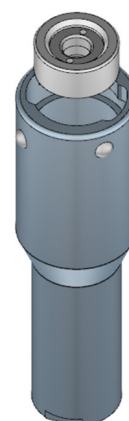
Rupture disc holder:



Please note that the rupture disc sits at the bottom with the bulge facing up.

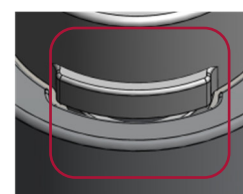
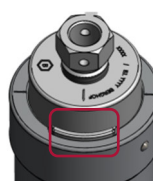


DAB-3



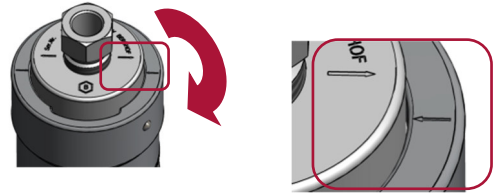
DAB-2

Bayonet quick closure is inserted and sealed in the pressure vessel next.



Turn the Bayonet quick closure clockwise, so that the arrow on the closure shows to the arrow on the top of the pressure vessel.

In case that the quick lock device cannot be revolved and locked between the pins, the hexagonal screw must be turned somewhat back (counter-clockwise) so that the pin is lifted

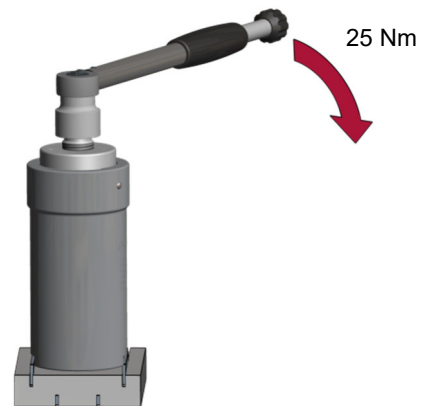


By screwing-in the hexagonal screw, the pressure plate is pressed on the PTFE-Lid.

By means of the torque wrench the hexagonal screw is tightened until the adjusted locking force of 25Nm is reached. This is indicated by a clearly-audible crack of the torque wrench.

The pressure vessel prepared as described can now be preferably heated in a heating block.

The thread of the quick closure must be regularly greased with PTFE paste.
(see chapter Maintenance)



4.3. Performing a Pressure Digestion



Explosion hazard!

The unit may never be employed without proper over-pressure protection (e.g., rupture disc). Corroded rupture discs shall be replaced in time.

The unit is not designed for the use with substances decomposing exothermically or forming explosive vapors upon heating (e.g. perchloric acid., Alcohols, Ether etc.).

Highly reactive substances which spontaneously react at higher temperatures or in the presence of oxidizing agents or which may be nitrated and are potentially explosive may not be digested. Examples of such substances include: explosives (TNT, etc.), Perchlorates, ether, pyrophoric substances, etc.

Heating of the vessels may be done in heating blocks only. Never heat the pressure vessels in a drying oven; the pressure wave resulting from the eruption of the rupture disc may lead to damage of the oven.



Always wear eye protection and protective gloves!

Always wear your personal protective equipment during work which may involve contact with chemicals, that is, when working with acids, pressure vessels, during cleaning, etc.!

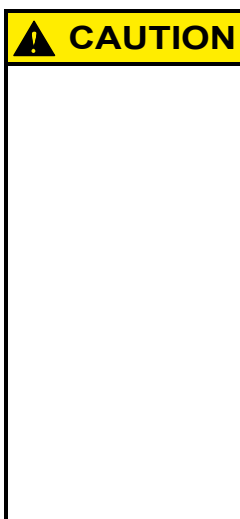


Heaters!

The Digestec pressure digestion system may only be used in conjunction with the specified heating systems.

Heating of the vessels may be done in Berghof heating blocks only. Never heat the pressure vessels in a drying oven; the pressure wave resulting from the eruption of the rupture disc may lead to damage of the oven.

Never allow liquids, particularly toxic compounds, to spill on hot parts.



Emissions!

The area above the pressure vessels should be marked as a danger zone, due to the possibility of the eruption of the bursting disc and the consequent release of hot acidic vapors.

Pressure digestions should be executed in a separate room or under an outlet. In case of a rupture disc break there should be nothing in the danger zone above the vessel.

Hot gases and vapors may be released from the digestion vessel when it is opened! Always make sure the vent opening on pressure vessels faces away from both yourself and other individuals!!

Should the over-pressure protection (e.g., rupture disc) activate, gases which may be hazardous to individuals will be suddenly released into the atmosphere. Always make sure the opening of the over-pressure protection is never directed towards individuals.

4.3.1. Heating

→ Temperature determination

According to the digestion difficulty, the temperature is adjusted from 140 – 190°C, or in utmost cases up to 250°C. Generally, the higher the temperature is adjusted, the quicker and more completed will be the sample decomposition, due to higher reacting kinetics and decomposition processes. However, it must be considered that due to high temperature the TFM™-PTFE -Insert is subjected to higher abrasion and the danger of overpressure is increased due to spontaneous reactions. For these reasons the temperature should be adjusted only as high as necessary, which can be ascertained by advantageous for certain samples (grass, leaves, plastic) to raise temperature slowly up to the final temperature. It must be considered that the internal active temperature is retarded during heating through the thick steel mantle and low heat conductivity of PTFE-material.

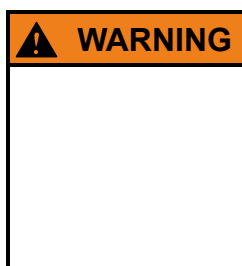
On the other side, the internal temperature can be for short higher than the external temperature due to exothermal reactions of the digestion mixture.

Due to the poor thermal conductivity of the TFM-material there is a certain delay between the heating of the sample and the heating of the heating block. For a final temperature of 200° C, a 30 minute delay is to be expected. In addition, there is always a small temperature difference between the measured temperature of the heating block and the interior temperature of the digestion vessel (to achieve an interior temperature of 200 °C, the heating block should be set at 210 °C), since the pressure vessel is not fully immersed in the heating block.

Generally when heating the pressure vessel in the aluminium heating block the adjusted set value at the temperature controller is reached after 1 hour to 88 % after 2 hours to 93 % and after 3 hours to 95 %. Simple matrices (food, corn, minerals) are usually digested after 1,5 – 2 hours and more difficult matrices (meat, dried milk, ceramics) after 2-4 hours. Complicated matrices (synthetic materials and coal) can take in certain cases up to 8 hours until being completely decomposed. Therefore it is advisable the digestion to be made over night. In special cases the optimum digestion parameters (weight-in, digestion acid, temperature and duration) have to be found out for the respective matrix-problem. Generally valid statements cannot be made, because no mineral, coal and no-biological web are similar to each other. Therefore it cannot be reckoned with the same digestion relations.

4.3.2. Cooling

After switching-off the heater the pressure vessel shall cool-down inside of the used heating system.



General operating instructions!

Cooling of the pressure vessel must take place exclusively in the heating block. Transport of the hot, pressurized vessel is not permitted for safety reasons. Even after complete cooling a certain residual pressure remains after digestion of organic samples, which means that even completely cooled down vessels need to be handled with caution. Especially the catch opening must never be directed towards any person as long as a vessel may still be under pressure.

Wait until the pressure vessel has cooled to room temperature.

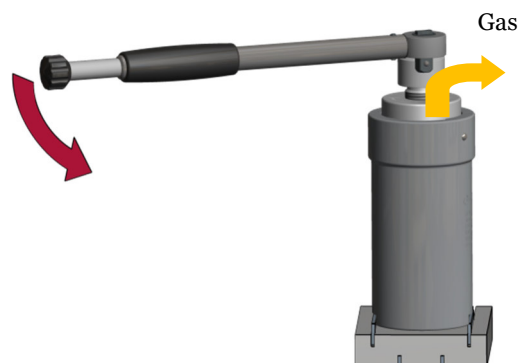
4.4. Opening

 DANGER	Pressure vessels! Use only digestion vessels and spare parts authorized by Berghof Products + Instruments GmbH for use with the Digestec. These can be obtained either from your local representative or directly from Berghof Products + Instruments GmbH (Refer to the "Repairs / Customer Service" section for contact addresses). Always wear your personal protective equipment when working with the pressure vessels! When using hydrofluoric acid especially appropriate safety equipment should be worn. Stainless steel is not resistant against aqua-regia. Due to diffusion of the acid through the PTFE-Insert the lifetime of the vessels will be reduced dramatically using aqua regia in the vessels DAB-2 or DAB-3. Pressure vessel to be closed with torque wrench, using the stated locking force. Note any changes in the appearance of the pressure vessel which could indicate possible material fatigue, e.g., deformation of the vessel, difficulty screwing on lids, etc. even when the vessel is cold and not under pressure. All such vessels should be immediately returned to Berghof Products + Instruments GmbH for inspection. Never attempt to use force to open the vessels. The pressure vessels are equipped with a rupture disc which produces a spontaneous pressure release upwards through the screw-on tube connections in case of excessive vessel pressure. In some cases, such a spontaneous pressure release may occur after the vessel has been removed from the heater. The area above the pressure vessels should be marked as a danger zone, due to the possibility of the eruption of the bursting disc and the consequent release of hot acidic vapors. The unit may never be employed without proper over-pressure protection (e.g., rupture disc). Corroded rupture discs shall be replaced in time. Pressure vessel may be heated up to 250°C only! (Heating only with over-temperature safety device). Open pressure vessel only in cold condition. Lid must be released and slacked (safety lock) before open quick-lock device. Hot gases and vapors may be released from the digestion vessel when it is opened! Always make sure the vent opening on pressure vessels faces away from both yourself and other individuals!! Only open the digestion vessel under a fume hood or other exhaust system. Particular care is required when working with hydrofluoric acid (HF). Observe all relevant data sheets and safety instructions! Never use more than one rupture disc per vessel! The bayonet can only be securely locked after a 90° turn to the stop. Improperly locked bayonets can become unfastened during use and especially while opening.
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4.4.1. Venting and Opening

Wait until the pressure vessel has cooled to room temperature.

To open the pressure vessel, use torque-wrench. Fix pressure vessels securely to the mounting plate. Apply torque-wrench and slowly turn in a counter-clockwise direction. As soon as gases start to escape, stop, or even briefly re-close.



4.4.2. Sample transfer

Carefully open the pressure vessel. Remove PTFE-Insert and transfer solution into a flask. Rinse the insert with bidest. water.

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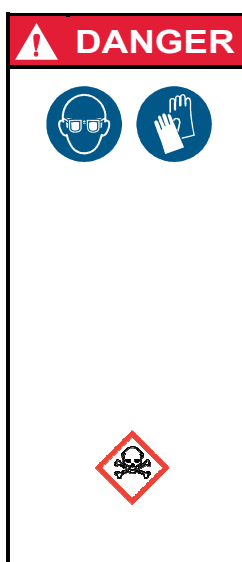
5. Maintenance, Troubleshooting and Service

All required inspection, maintenance, and repair work must be performed in compliance with the local ordinances.

No damaged components such as stripped, cracked, or broken screws, bent or broken nuts, bent clips or straps, damaged seals, etc. may be reused and must be replaced.

Any repair work which might compromise the safety of the pressure vessel such as welding, hot or cold forming that alter the reactor material properties may only be carried out in compliance with local ordinances.

5.1. Cleaning



Pressure vessels!

Always wear your personal protective equipment during work which may involve contact with chemicals, that is, when working with acids, pressure vessels, during cleaning, etc.!

Pressure vessel to be closed with torque wrench, using the stated locking force.

Never attempt to use force to open the vessels.

Open pressure vessel only in cold condition. Lid must be released and slacked (safety lock) before open quick-lock device.

Hot gases and vapors may be released from the digestion vessel when it is opened! Always make sure the vent opening on pressure vessels faces away from both yourself and other individuals!!

Only open the digestion vessel under a fume hood or other exhaust system. Particular care is required when working with hydrofluoric acid (HF). Observe all relevant data sheets and safety instructions!



Always wear personal protective equipment!

Always wear your personal protective equipment during work which may involve contact with chemicals, that is, when working with acids, pressure vessels, during cleaning, etc.!

In order to ensure the long-term, faultless operation of the digestion system and its accessories, various maintenance and inspection tasks should be performed at regular intervals.



Despite the fact that all the pressure vessel components are manufactured from high-grade stainless steel and the vessel and lid may be lined with PTFE, indications of wear and corrosion resulting from aggressive reaction materials and their vapors cannot be ruled out.

PTFE-Insert

Major contaminations, which may occur during incomplete digestions, can be removed initially using a brush and water. Afterwards rinse the vessels at least with bi-distilled water. A significantly improved cleaning effect is achieved by steaming with nitric acid in a special steaming device.

The exterior of the PTFE vessel insert should also be carefully cleaned because, for example, the chloride content of oils and sweat from user's hands promote corrosion on stainless steel (1.4571). The pressure vessel and fittings should also be rinsed off from time-to-time. After rinsing, make sure all components are completely dry before reinstalling them. In particular, there must be no moisture between the PTFE vessel insert and the pressure vessel in order to prevent the formation of gas blankets during subsequent operation.

The PTFE -Insert can be cleaned in hot acid by means of boiling or blank digestion. By blank digestion in HNO_3 or HCl , PTFE is additionally preconditioned for subsequent usage in trace analysis. Thereby it is obtained that interactions of PTFE-material with the digestion sample (adsorption and desorption) are avoided to a large extent. A yellow colouring of the PTFE-material after usage with HNO_3 follows as a consequence of nitrous fumes (diffusing) and is unobjectionable.

By heating the PTFE Inserts in an oven for 3-4 hours at 200-220 ° C (390 ° F 430) can be decolorized the inserts again. (see also "Tips and Tricks"). Likewise for technical reason is the some-shaped deformation in the middle of the PTFE-lid (rated break point), which is due to the form of the rupture disc in which the PTFE-lid is pressed in when being under pressure.

Stainless steel vessel

Although the pressure vessel is made of a high quality stainless steel it is not possible to avoid discolouring and in utmost cases corrosion (after digestions with HNO_3/HCl and HF). The fumes of digestion acid are diffusing under high pressure and temperature into the PTFE and are also given off externally to a certain degree, coming into contact with the hot stainless steel. According to the strain it is therefore very important to clean the stainless steel vessel with a scouring agent or to treat it with concentrated H_3PO_4 2 to 3 hours.

Hexagonal screw in bayonet closure

The thread of the hollow hexagonal screw must be equipped with a high-temperature PTFE paste so at the high digestion temperatures and mechanical stress no seizure of the thread (cold welding, thread galling) occurs.

See also chapter "maintenance and inspection"

Sealing surface

In order to ensure a proper sealing, the seal surfaces in the locking area must be kept clean and smooth.

5.2. Maintenance and Inspection

 WARNING	Use only specified spare parts! Only rupture discs with a bursting pressure equal to or less than the high-pressure laboratory reactor's maximum operating pressure may be installed in the holder. Only OEM rupture discs specified by Products + Instruments GmbH for your specific reactor may be used. These can be obtained from your local representative or directly from Products + Instruments GmbH (refer to the "Repair / Customer Service" section for the contact address).
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The digestion vessels are to be regularly serviced by competent personnel in order to ensure that it remains hermetically sealed in the long-term under the intended operating conditions and anticipated mechanical, chemical, and thermal loads. Based on the operating instructions and experience, the operator should note the required maintenance and inspection measures in the operating instructions themselves.

In particular, monitoring and ensuring the following are an essential part of the required inspection and maintenance tasks:

- Pressure tightness
- Ambient conditions and safety zones
- Identification and other labels
- Proper operation of all safety and alarm equipment
- Wall thickness where the unit is subject to corrosion

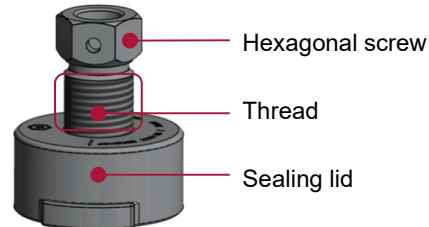
After repeated usage:

- Aluminium-protection foil to be replaced and high-temperature lubricant to be sprayed on locking screw!
- Clean pressure vessel with concentrated phosphoric acid in case of corrosion! (see chapter "Cleaning")
- Rupture discs and Belleville spring to be replaced after 1- 2 years of usage or in case of corrosion! The disk may fatigue after several very high pressure loads and thereby alter its response pressure value.

5.2.1. Apply PTFE-paste

As far as possible remove the hexagonal screw and apply PTFE-paste on the thread by means of a small brush.

The screw must be tightened and loosened by hand smoothly and easily.



NOTICE

Cold welding

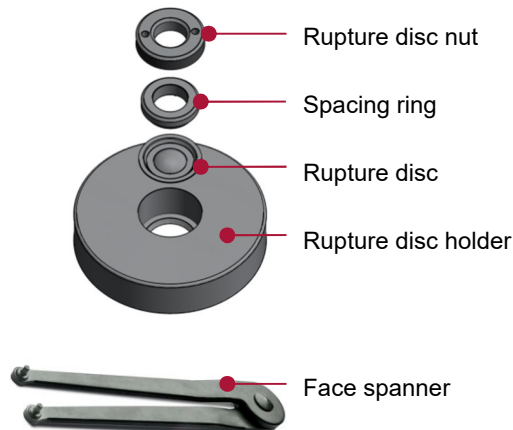
To avoid cold welding, the thread of the hexagon screw must be provided with sufficient thread lubricant.

5.2.2. Rupture disc replacement

After longer application (1-2 years) or when corrosion appears, the rupture disc should be replaced. For that purpose the rupture disc holder is opened with a face spanner. For this purpose the rupture disc holder is vertically clamped in a vice. The thread of the pressure disc holder and the pressure disc nut must be provided with the PTFE-paste.

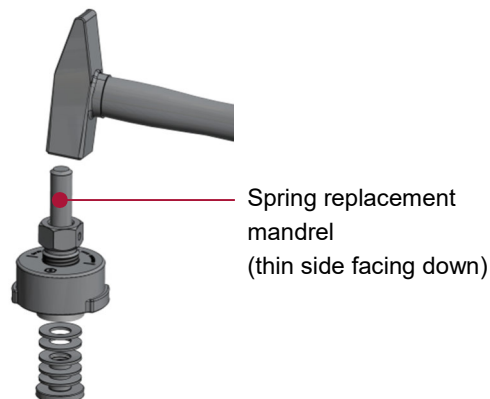
Reassemble in reverse order, the curvature of the rupture disc must face upwards.

Tighten the rupture disc nut with the face spanner only hand tight.

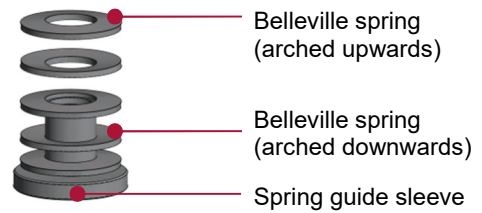


Belleville springs replacement

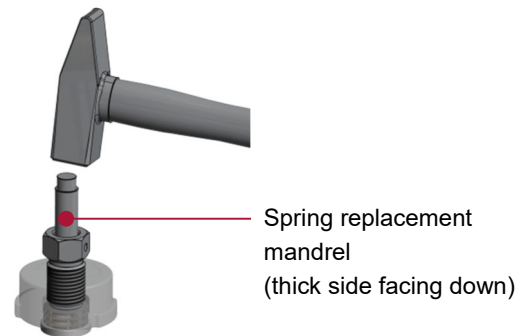
The belleville springs should also be replaced after longer usage, as they loose, particularly on high temperature loadings, their elastic force or get useless due to tension cracks. For replacement of the belleville springs the quick-lock closure is vertically clamped in a vise. With the **thin side** of the spring replacement mandrel the spring guide sleeve of the quick-lock device is loosened through the hollow hexagonal screw, by means of a hammer.



The new belleville springs must be rearranged on the spring guide sleeve alternately downwards and upwards.
(6 pieces for DAB-2 and 5 pieces for DAB-3)



The spring guide sleeve is reinserted along with the disc springs into the hexagonal hollow screw and placed on a hard base plate. By means of a hammer with the **thick side** of the spring replacement mandrel the beading is then restored.



5.3. Recurring Inspection

See chapter 1.5 Legal Requirements

5.4. Inspection

After each use, a visual inspection of the reactor's exterior and interior must be performed. No damaged components such as stripped, cracked, or broken screws, bent or broken nuts, bent clips or straps, damaged seals, etc. may be reused and must be replaced.

If due its purpose, the reactor in question falls under the category "test autoclave", after **each** use the experimental autoclave must be examined by a competent person.

See chapter 1.5 Legal Requirements

5.5. Repairs / Customer Service

You can obtain technical support from your regional Berghof representative or directly from Berghof Products + Instruments GmbH by contacting:

Berghof Products + Instruments GmbH
 Arbachtalstraße 26
 72800 Eningen
 Germany
 T +49.7121.894-170
 F +49.7121.894-300
 E-Mail: service.bpi@berghof.com
www.berghof-instruments.com

→ [Berghof Service](#)

Please provide your unit's serial number with all repair questions or repair orders. The serial number is located on the nameplate.

5.5.1. Reshipment

NOTICE

Cleaning and decontamination methods

Before using any cleaning or decontamination methods except those specified by Berghof Products + Instruments, users should check with Berghof Products + Instruments or your regional Berghof representative that the proposed method will not damage the equipment.

Customers wishing to return instrumentation and/or associated materials to Berghof Products + Instruments GmbH or regional representative for repair, maintenance, warranty, inspection or trade-in purposes are advised that all returned goods must be certified as clean and free from contamination.

→ [Safety and Health Declaration](#)

The customer's responsible body is required to fill in and sign the form "Safety and Health Declaration" and to display this declaration on the exterior of the shipping container.

This document is available at:

Berghof Service (see contact data above)

Outside the Federal Republic of Germany goods may only be returned to us via our authorized representative. Before returning the goods, please contact our service department.

5.5.2. Safety and Health Declaration

Return shipments of instruments and equipment not having decontamination certificates display on the exterior of the shipping container will not be processed and will remain unopened, in a sealed condition, until a determination of decontamination is received.

5.6. Tips and Tricks

5.6.1. PTFE Insert

PTFE –Insert regeneration

PTFE inserts deform after repeated use of pressure and temperature stress. However, it can be returned in the original shape by heating them in an oven for 3-4 hours at 300-320 ° C (572- 608°F).

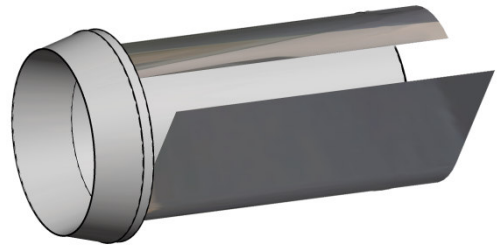
Distaining the PTFE -Insert

By heating the PTFE inserts in an oven at 200-220 ° C (390 ° F 430) for 3-4 hours, the inserts can be decolorized again.

Protection of PTFE -Insert

To protect the pressure vessel from corrosion, the PTFE-Insert can be wrapped with an aluminium foil.

Fix the remaining foil on the bottom of the vessel and press firmly.



5.7. Troubleshooting

Fault	Possible cause	Corrective action
Pressure loss	Deformed PTFE insert or lid	Replace damaged parts. Regenerate the PTFE insert (refer to the 'Maintenance' section)
	Closure force too low	Use correct torque with torque wrench
Rupture disc triggers at low pressure	Corroded rupture disc	Replace rupture disc
Screws cannot be removed	Too high residual pressure in the pressure vessel	Release pressure
	Used too little PTFE-Paste	Contact service

You can obtain technical support from your regional Berghof representative or directly from Berghof Products + Instruments GmbH by contacting:

Berghof Products + Instruments GmbH
 Arbachtalstraße 26
 72800 Eningen / Germany
 Tel: +49/(0)7121/894-170
 Fax: +49/(0)7121/894-300
 E-Mail: service@berghof-instruments.de
www.berghof-instruments.com

Please provide your unit's serial number with all repair questions or repair orders. The serial number is located on the nameplate.

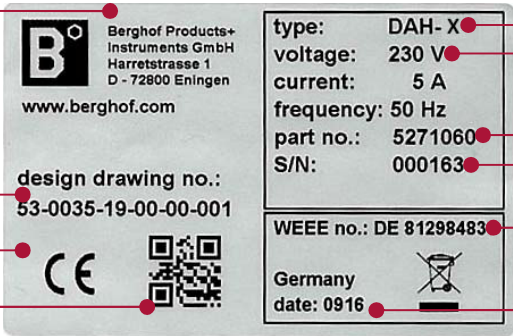
5.8. Stamping / Nameplate

5.8.1. Stamp marking on the pressure vessel

Manufacturer symbol		
Serial number	1234	
Year of manufacture	20XY	
Permitted operated pressure	max.200 bar	
Permitted operated temperature	max.250 °C	
Volume	50 ml / 250 ml	
Test pressure	330 bar	

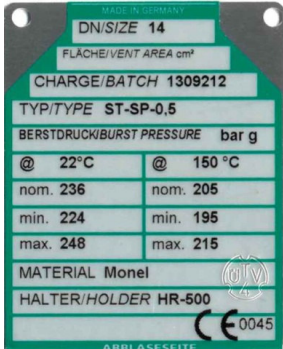
5.8.2. Name plate

Electrical Heating Block (DAH)

Manufacturer and adress			
Berghof	type: DAH- X	Modell-Typ	
design drawing number	voltage: 230 V	Supply voltage	
CE-Designation	current: 5 A		
QR Barcode	frequency: 50 Hz		
	part no.: 5271060	Part number	
	S/N: 000163	Serial number	
	WEEE no.: DE 81298483	WEEE Registration	
	Germany date: 0916	Manufacturing date	

The name plates of the heating blocks (DAH) are located on the bottom of the devices.

Rupture disc



This nameplate is attached on the rupture disc holder.

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6. Appendix

6.1. Conversion Table

Conversation of units		
Dimension	Unit	Conversion
Temperature	°C =	(°F – 32°) / 1.8
	°F =	1.8 * °C + 32°
Length	1 cm =	0.3937 inch
	1 inch =	2.540 cm
Volume	1 ml =	0.06102 inch ³ = 2.642*10 ⁻⁴ gallon
	1 inch ³ =	16.387 ml = 43.29*10 ⁻⁴ gallon
	1 gallon =	3785 ml = 231 inch ³
Pressure	1 bar =	14.504 psi = 0.1 Mpa
	1 psi =	0.06895 bar = 0.0068948 Mpa
	1 Mpa =	10 bar = 145.04 psi
Weight	1 kg =	2.2046 lb.
	1 lb. =	0.4536 kg

6.2. Abbreviation

Plastics	
Abbreviation	Technical term
PE	Polyethylene
PFA	Perfluoralkoxy
PP	Polypropylene
PTFE	Polytetrafluorethylene
TFM™-PTFE	Modified Polytetrafluorethylene
Kalrez® (FFKM)	DuPont™ Perfluoroelastomer
Viton® (FPM)	DuPont™ Fluoroelastomer

Metals	
Abbreviation	Technical term
SS	Stainless steel with the corresponding material number
HC-4	Hastelloy C4

6.3. Application

Application DAB System							
Sample	Digestion system	Weight	Vessel	Acid	Temp.	Time	File
Environment Geology							
Columbite	DAB	100 mg	DAB-2	H ₂ SO ₄ :HF	280°C	570 min	DAB-Columbite
Effluents Mercury	DAB	40 mL	DAB-2	HNO ₃	120°C	60 min – 90 min	DAB-Effluents-Hg
Effluents Phosphorus	DAB	40 mL	DAB-3	H ₂ SO ₄ :K ₂ S ₂ O ₈	120°C	120 min	DAB-Effluents-P
Galvanic ash	DAB	250 mg	DAB-2	HNO ₃ :H ₂ SO ₄ :HF	250°C	960 min	DAB-Galvanic-Ash
Resin	DAB	500 mg	DAB-3	HNO ₃	220°C	360 min	DAB-Resin
Rock	DAB	1000 mg	DAB-3	HNO ₃ :HCl:HF	220°C	120 min	DAB-Rock
Food Pharma Cosmetic							
Buckwheat	DAB	500 mg	DAB-3	HNO ₃ :H ₂ O ₂	200°C	240 min	DAB-Buckwheat
Cereals	DAB	1000 mg	DAB-3	HNO ₃	170°C	120 min	DAB-Cereal
Fat	DAB	1000 mg	DAB-3	HNO ₃	200°C	180 min	DAB-Fat
Flour	DAB	500 mg	DAB-3	HNO ₃	200°C	180 min	DAB-Flour
Flour	DAB	1000 mg	DAB-3	HNO ₃	180°C	120 min	DAB-Flour_A
Grass	DAB	1000 mg – 2000 mg	DAB-3	HNO ₃	200°C	180 min	DAB-Grass

Leaves	DAB	1000 mg	DAB-3	HNO3	170°C	120 min	DAB-Leaves
Line Seed	DAB	500 mg	DAB-3	HNO3	200°C	240 min	DAB-Line-Seed
Liver	DAB	1000 mg	DAB-3	HNO3	180°C	180 min	DAB-Liver
Multi vitamin tablet	DAB	300 mg	DAB-3	HNO3:H2O2:H2O	220°C	10 h	DAB-Multi-Vitamin
Multi vitamin tablet	DAB	300 mg – 1500 mg	DAB-3	HNO3:H2O2:H2O	220°C	10 h	DAB-Multi-Vitamin_A
Oil	DAB	1000 mg	DAB-3	HNO3	200°C	240 min	DAB-Oil
Poppy	DAB	500 mg	DAB-3	HNO3:H2O2	200°C	120 min	DAB-Poppy
Pumpkin seed	DAB	500 mg	DAB-3	HNO3:H2O2	200°C	240 min	DAB-Pumpkin
Sesame	DAB	500 mg	DAB-3	HNO3	200°C	240 min	DAB-Sesame
Starch	DAB	1000 mg	DAB-3	HNO3	160°C	120 min	DAB-Starch
Sugar	DAB	500 mg	DAB-3	HNO3	200°C	120 min	DAB-Sugar
Sunflower seed	DAB	500 mg	DAB-3	HNO3	200°C	240 min	DAB-Sunflower-Seed
Tissue	DAB	1000 mg	DAB-3	HNO3	180°C	120 min	DAB-Tissue
Tobacco	DAB	1000 mg – 2000 mg	DAB-3	HNO3	200°C	240 min	DAB-Tobacco
Tomato leaves	DAB	500 mg	DAB-3	HNO3:H2O2	200°C	240 min	DAB-Tomato-Leaves
Tomato leaves	DAB	1000 mg – 2000 mg	DAB-3	HNO3	200°C	240 min	DAB-Tomato-Leaves_A
Vegetables	DAB	1000 mg – 2000 mg	DAB-3	HNO3	200°C	240 min	DAB-Vegetables

Technology							
Alumina	DAB	200 mg	DAB-3	HCl:H ₂ O	260°C	720 min	DAB-Alumina
Ceramics	DAB	400 mg	DAB-3	HCl:HF	230°C	>120 min	DAB-Ceramics
CeZr	DAB	100 mg	DAB-2	H ₂ SO ₄ :HNO ₃ :HCl	280°C	520 min	DAB-CeZr
Coal	DAB	500 mg	DAB-3	HCl	220°C	360 min	DAB-Coal
Cu-alloy	DAB	500 mg	DAB-3	HNO ₃ :HF	220°C	120 min	DAB-Copper-Alloy
Graphite	DAB	500 mg	DAB-2	HNO ₃ :H ₂ SO ₄	250°C	48 h – 72 h	DAB-Graphite
Lubricant oil	DAB	1500 mg	DAB-3	HNO ₃ :HCl	200°C	240 min	DAB-Lubricant-Oil
Methanolysis of PET	DAB	50 mg - 250 mg	DAB-2	Me-OH Zn-acetate solut.	200°C	18 h	DAB-Methanolysis
Oil	DAB	500 mg	DAB-3	HNO ₃	180°C	480 min	DAB-Oil
Paint	DAB	800 mg	DAB-3	HNO ₃ :H ₂ O ₂	200°C	180 min	DAB-Paint
Paper	DAB	1000 mg	DAB-3	HNO ₃	150°C	120 min	DAB-Paper
PE	DAB	500 mg	DAB-3	HNO ₃ :H ₂ O ₂	200°C	180 min	DAB-PE
Platinum group elements	DAB	100 mg	DAB-2	HNO ₃ :HCl	280°C	520 min	DAB-PGM
Pigment gel	DAB	200 mg	DAB-3	HNO ₃ :H ₂ O ₂	200°C	180 min	DAB-Pigment-Gel
Plastic	DAB	500 mg	DAB-3	HNO ₃ :H ₂ O ₂	200°C	180 min	DAB-Plastic
Plastic	DAB	500 mg	DAB-3	HNO ₃ :HCl	200°C	180 min	DAB-Plastic_A

Plastic	DAB	500 mg	DAB-3	HNO3:H2SO4	200°C	180 min	DAB-Plastic_B
Porcelainite	DAB	200 mg	DAB-2	H3PO4:HF	270°C	450 min	DAB-Porcelainite
PP	DAB	200 mg	DAB-2	HNO3	250°C	180 min	DAB-PP
Quartz	DAB	500 mg	DAB-2	HNO3:HF	250°C	180 min	DAB-Quartz-Sand
SiC	DAB	250 mg	DAB-2	HNO3:HF:H2SO4	250°C	12 h – 72 h	DAB-SiC
Silicon oil	DAB	500 mg	DAB-3	HNO3:HF	180°C	8 h	DAB-Silicon-Oil
SiN	DAB	250 mg	DAB-2	HNO3:HF:H2SO4	250°C	12 h	DAB-SiN
Steel	DAB	500 mg	DAB-3	HNO3:HCl	200°C	120 min	DAB-Steel
Talcum	DAB	50 mg	DAB-3	HNO3:HF	200°C	10 h	DAB-Talcum
Textiles	DAB	500 mg	DAB-3	HNO3:H2O2	200°C	180 min	DAB-Textiles
Textiles	DAB	500 mg	DAB-3	HNO3:HCl	200°C	180 min	DAB-Textiles-A
Wax	DAB	1500 mg	DAB-3	HNO3	240°C	180 min	DAB-Wax
Weld dust	DAB	1 filter	DAB-3	HNO3:HF	200°C	240 min	DAB-Weld-Dust