

# IKA-Calorimeter system C 5000 control C 5000 duo-control



Abb.: C 5000 duo-control



*OPERATING INSTRUCTIONS*

*GB /  
USA*

**CE – KONFORMITÄTSERKLÄRUNG****D**

Wir erklären in alleiniger Verantwortung, daß dieses Produkt den Bestimmungen der Richtlinien 89 / 336 EWG; 89 / 392 EWG und 73 / 23 EWG entspricht und mit folgenden Normen und normativen Dokumenten übereinstimmt:  
EN 61 010; EN 50 082; EN 55 014; EN 60 555.

**CE – DECLARATION OF CONFIRMITY****GB**

We declare under our sole responsibility that this product corresponds to the regulations 89 / 336 EEC; 89 / 392 EEC and 73 / 23 EEC and conforms with the standards or standardized documents:  
EN 61 010; EN 50 082; EN 55 014; EN 60 555.

**DÉCLARATION DE CONFORMITÉ CE****F**

Nous déclarons sous notre responsabilité que ce produit est conforme aux réglementations 89 / 336 CEE; 89 / 392 CEE et 73 / 23 CEE et en conformité avec les normes ou documents normalisés suivants :  
EN 61 010; EN 50 082; EN 55 014; EN 60 555.

**DECLARACION DE CONFORMIDAD DE CE****E**

Declaramos por nuestra responsabilidad propia que este producto corresponde a las directrices 89 / 336 CEE; 89 / 392 CEE y 73 / 23 CEE y que cumple las normas o documentos normativos siguientes:  
EN 61 010; EN 50 082; EN 55 014; EN 60 555.

**CE – DICHIARAZIONE DI CONFORMITÀ****I**

Dichiariamo, assumendone la piena responsabilità, che il prodotto è conforme alle seguenti direttive CCE 89 / 336 ; CCE 89 / 392 e CCE 73 / 23, in accordo ai seguenti regolamenti e documenti:  
EN 61 010; EN 50 082; EN 55 014; EN 60 555.

IKA-LABORTECHNIK Janke & Kunkel GmbH & Co. KG

Staufen, February 18 1998



Wolfgang Buchmann  
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Quality Assurance

## Explanation of icons



This icon identifies **information that is absolutely essential to ensure your health and safety**. Failure to observe this information may result in injury or may adversely affect your health.



This icon identifies **information that is significant for operating the equipment in a technically correct manner**. Failure to observe this information may result in damage to the calorimeter system.



This icon identifies information that refers you to information that is important to operate calorimetric measurements properly and to work with the calorimeter system. Failure to observe this information may lead to imprecise results in measurements.

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# 1 For your safety



The C 5000 Calorimeter system has been developed and manufactured according to the most modern safety requirements. According to the stipulations, we must draw your attention to the following points:

## Application purpose

The C 5000 calorimeter system may only be used to determine the gross calorific values of solid and liquid substances.

Many materials have a tendency to combust in an explosive manner (because of peroxide formation, for example), which could result in the decomposition vessel bursting.

## Explosive materials

**It is absolutely essential to use a special high-pressure decomposition vessel for receiving fuel sample when performing research with fuel samples capable of exploding. The standard decomposition vessels C5010 and C5012 must not be used for this purpose.**

## Combustion samples, residues and auxiliary materials

In addition, it is also possible that toxic combustion residues in the form of gasses, ashes or precipitates may form on the inner wall of the decomposition vessel.

When working with combustion samples, residues of combustion and auxiliary materials, the safety precautions appropriate for each one must be observed. Hazards may be present, for example, in substances with any of the following characteristics

- corrosive
- easily flammable
- capable of exploding
- contaminated with bacteria
- toxic
- radioactive

When using crucibles made of stainless steel, you should check their condition carefully after the experiment. After a maximum of 25 combustions, the crucibles may no longer be used for reasons of safety.

## Qualifications of the user

The unit may only be operated by a professional or a person who has received instruction. Among other qualifications, the user must be familiar with combustion processes and products of combustion that arise during the process.

## Decomposition vessel

The decomposition vessel corresponds to the requirements of the German regulation DK 621.642-986 on pressure containers (Group II). A manufacturer's certificate is included with the accompanying papers. This should be kept, since it must be presented upon the request of safety authorities.



**According to the regulation on pressure containers, the operator is responsible for the safety of the decomposition vessel.**

Pressure tests and service work on the decomposition vessel may only be performed by authorized personnel.



**We urgently recommend that you regularly send the decomposition vessel in to our factory to be checked or repaired (see maintenance contract).**

For detailed references, please read the Operating Instructions for the decomposition vessels C5010/C5012.

**Regulation on  
pressure  
containers**

German regulation DK 621.642-986 on pressure containers (excerpt):

**§8 Categorization into test groups**

Pressure containers are categorized according to the permissible operating pressure  $p$  in bars, the capacity of the pressure area  $l$  in liters and the pressure content product  $p \cdot l$ . If there are several pressure areas that are separated from each other, the product for each pressure area is determined separately. The following categories are distinguished:

**Group II:**

Pressure containers with a permissible operating pressure  $p$  of more than 0.1 bar, but not more than 1 bar, and pressure containers with a permissible operating pressure  $p$  of more than 1 bar, for which the pressure content product  $p \cdot l$  is not more than 200.

**§ 10 Recurring tests**

(2) A pressure container of Group I, II, III, IV and VI must be subject to recurring tests by professionally competent personnel. The timing of the tests will be determined by the operator according to experience with the work procedure and the type of coating.

**§ 13 Operating pressure containers**

(1) Anyone who operates a pressure container must maintain the same in proper operating condition, must operate it properly, must monitor it, must perform necessary maintenance and repair work without delay, and must take necessary safety measures corresponding to circumstances.

**Parts that conduct  
electricity**

(3) A pressure container must not be operated if it has any defect that endangers employees or a third party.

The calorimeter system may only be opened by a maintenance or customer service location.

We recommend you refer to our customer service department for your service needs.



## 2 User notes



Working  
through  
Sections 1 ... 9

Performing  
experiments

### 2.1 Notes on using the operating instructions

In this section you will learn how to work through these Operating Instructions in the most effective manner to be able to work safely with the calorimeter system.

**The instructions in Section 1 “For your Safety” must be followed.**

You should work through sections 1 through 9 in order, one after the other. In Section 3 “Calorimetric measurements,” you will find helpful information about determining gross calorific values with calorimeters. Section 4 “Features of the system” provides you with information about standards to which the system conforms, measurement ranges of the system and the reference states into which the gross calorific value can be converted. Section 5 “Transportation, storage and setup location” is of relevance for the reliability of the system and for ensuring a high degree of reliability in measurements.

In addition to the description of system components, Section 7 contains technical data on individual components.

The calorimeter system is ready for a measurement after you have performed the procedures in Section 8 “Setting up and placing in service” and Section 9 “System calibration”. The following determinations of gross calorific values should be performed according to Section 10 “Determining gross calorific values” and Section 11 “Evaluating experiments”.

### 2.2 Guarantee

You have purchased an Original IKA-WERKE device, which conforms to the highest standards of technology and quality. The guarantee is for 12 months, according to the IKA guarantee conditions. If you need to use the guarantee, please refer to the appropriate dealership or supplier. You can also send the unit directly to the IKA factory, including with it the invoice from the supplier and stating the reasons for returning it, and telling us who the contact person is. Shipping costs are paid by the sender.

### 2.3 Warrantee and liability

Please read through these Operating Instructions attentively. IKA WERKE considers itself responsible for the safety, reliability and performance of the device only:

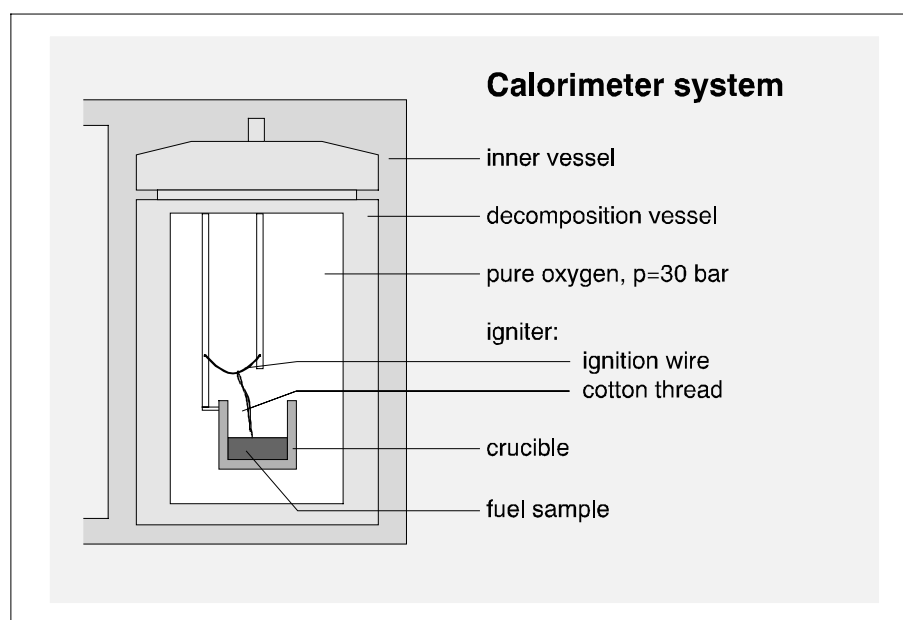
- If the unit has been used in accordance with the operating instructions
- If only persons authorized by the manufacturer perform maintenance on or make repairs to the unit, and

- If only original parts and original accessories are used for repairs.

We also direct your attention to the appropriate safety requirements and accident prevention specifications.

IKA WERKE is not responsible for damages or costs resulting from accident, misuse of the unit or unauthorized modifications, repairs or innovations.

## 3 Calorimetric measurements



### 3.1 Determining the gross calorific value

#### Calorimeter system

In a calorimeter, combustion processes take place under precisely defined conditions. For this purpose, the decomposition vessel is charged with a weighed in fuel sample, the fuel sample is ignited, and the increase in temperature in the calorimeter system is measured. The specific gross calorific value of the sample is calculated from:

- the weight of the fuel sample
- the heat capacity (C value) of the calorimeter system
- the increase in temperature of the water in the inner vessel of the measurement cell

#### Experiment conditions

To optimize the combustion process, the decomposition vessel is filled with pure oxygen (99.95%). The pressure of the oxygen atmosphere in the decomposition vessel is 30 bar.

The exact determination of the gross calorific value of a substance is based on the requirement that the combustion proceeds under precisely defined conditions. The applicable standards are based on the following assumptions:

- The temperature of the substance to undergo combustion is 22°C before combustion.
- The water contained in substance and the water formed during combustion of compounds in the substance containing hydrogen are present after combustion in liquid state.
- No oxidation of atmospheric nitrogen takes place.

- The gaseous products of combustion consist of oxygen, nitrogen, carbon dioxide and sulfur dioxide.
- Solid ash is formed.

Often, however, the products of combustion assumed by the standards are not the only ones that are formed. In such cases, analyses must be performed on the fuel sample and the combustion products that yield data for a correction calculation. The standard gross calorific value is then determined from the measured gross calorific value and the analysis data.

**Ho gross  
calorific value**

The Ho gross calorific value is formed from the quotient of the quantity of heat liberated during complete combustion of a solid or liquid combustible substance and the weight of the fuel sample. In this calculation, the water formed before the combustion of compounds of the combustible substance must be present in a liquid state after the combustion.

Reference temperature 22°C

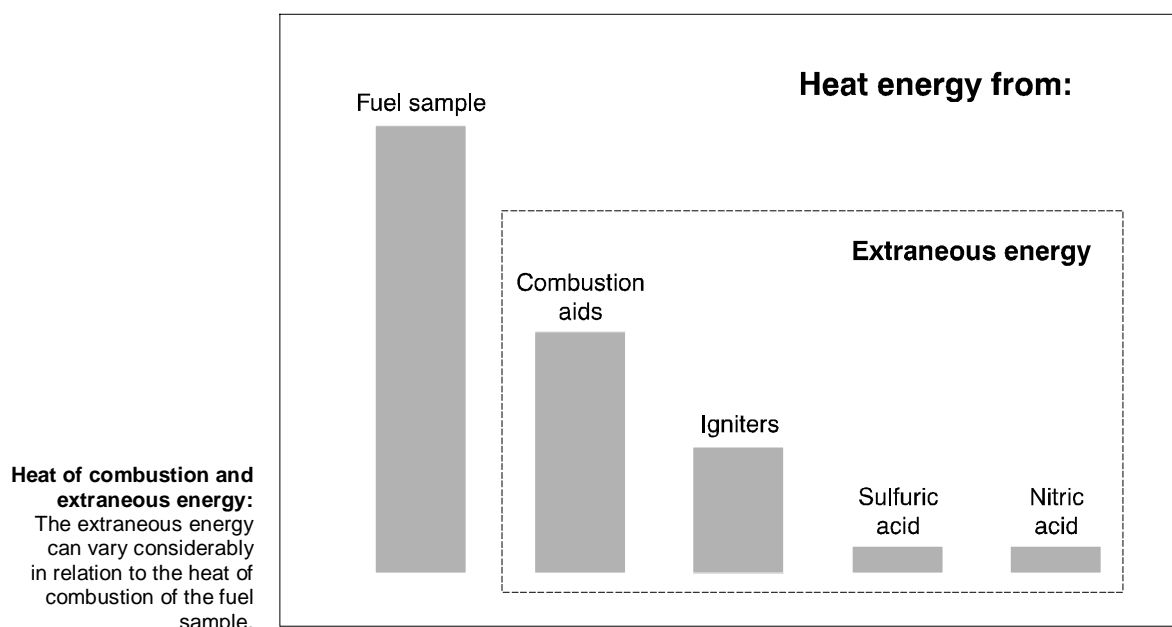
**Hu net calorific  
value**

The net calorific value Hu is equal to the gross calorific value reduced by the energy of condensation of the water that was contained in the combustible substance that is formed by combustion. The net calorific value is the technically more important quantity, since only the net calorific value can be evaluated in terms of energy in all important, technical applications.

On the calculation formulas for gross and net calorific value, see Section 16 "*Basic of calculations*"

## 3.2 Corrections

During a combustion experiment, as conditioned by the system, heat is not generated only by combustion of the sample; in addition heat also arises through extraneous energy:



### Igniter

The heat of combustion of the cotton thread that ignites the sample and the heat of electrical ignition would distort the measurement. This effect is taken into account in the calculation with a correction value.

### Combustion aid

Substances with low inflammability and substances that do not readily undergo combustion are burned together with a combustion aid. The combustion aid is first weighed and is then placed in the crucible with the sample. From the weight of the combustion aid and a specific gross calorific value that is of course already known, it is possible to determine the amount of heat that is introduced by the combustion aid. The result of the experiment must then be corrected by that quantity of heat.

### combustible crucible C14

The C14 combustible crucible can be used instead of a more traditional crucible. The combustible crucible is burned completely with no residue. When a combustible crucible is used, no additional cotton thread is required. The crucible is contacted directly by the fixed ignition wire of the decomposition vessel and is ignited.

The purity of the material of the combustible crucible prevents chemical contamination of the sample material (no blank values).

Decomposition vessel in which the combustible crucible is used must be retrofitted with an additional part (attachment C5010.4, see accessories). The sample is weighed in into the combustible crucible normally. In most cases, no additional combustion aid is required, because the combustible crucible itself serves as a combustion aid.



**The C14 combustible crucible cannot be used in combination with the sample rack.**

### Acid correction

Almost all substances to be analyzed contain sulfur and nitrogen. Under the conditions that prevail in calorimetric measurements, sulfur and nitrogen burn and

are reduced to  $\text{SO}_2$ ,  $\text{SO}_3$  and  $\text{NO}_x$ . In combination with the water from combustion and moisture, sulfuric acid and nitric acid are produced in addition to heat of solution. In order to obtain the standard gross calorific value, the gross calorific value is corrected by the effect of the heat of solution.

In order to obtain a defined final state and to measure all acids quantitatively, 5 ml of distilled water is placed in the decomposition vessel before the experiment. The gasses liberated during combustion form acids with the distilled water. After the combustion, the decomposition vessel is rinsed thoroughly with distilled water to collect the precipitate that has been deposited on the inner wall of the vessel as well. The water that was placed in the decomposition vessel is combined with the rinse water to be titrated for acid content.

### 3.3 Complete combustion

To determine the gross calorific value correctly, it is of fundamental significance for the sample to be burned completely. After the experiment, the crucible and all solid residues must be examined for signs of incomplete combustion.

#### Solid substances

Normally, solid combustion substances can be burned directly in powder form. Substances that burn rapidly, i.e. substances for which the combustion has the nature of an explosion (for example benzoic acid) must not be burned in loose form. These substances tend to spark, and complete combustion could therefore no longer be guaranteed. In addition, the decomposition vessel could be damaged. Such substances must be pressed into tablets before combustion (see Accessories).

#### Substances with low inflammability

Substances with low inflammability (substances with a high mineral content, low-calorific materials) often can be burned only with the aid of combustion capsules or combustion bags (see Accessories). It is also possible to use liquid combustion aids such as paraffin oil or hydrocarbon oil.

#### Liquid and highly volatile substances

Most liquid substances can be weighed directly into the crucible. Highly volatile substances are placed in combustion capsules (gelatin capsules or acetobutyrate capsules, see Accessories) and are burned together with the capsules. The igniters (cotton thread) must be completely burned as well. If unburned remainders of the igniter are left over, the experiment must be repeated or a correction must be introduced into the result through the extraneous energy.

#### Halogens

Substances with high halogen content can cause corrosion to appear on the decomposition vessel. Decomposition vessel C5012 should be used for these purposes.

### 3.4 Calibration

To ensure exact reproducible measurement results, the calorimeter system is calibrated after it is first placed in service, after maintenance work, after parts are replaced and at specific time intervals. During calibration, the heat capacity of the calorimeter system is re-determined.



**Regular calibration is absolutely essential to maintain accuracy of measurement. Furthermore, the system must be calibrated in the operating mode that will be used for the experiment (adiabatic, isoperibolic or dynamic).**

For this purpose, a specific quantity of a reference substance is burned in the decomposition vessel under the conditions of the experiment. Since the gross calorific value of the reference substance is known, it is possible to use the increase in temperature of the calorimeter system when the reference substance is burned to calculate the heat capacity.

The reference substance for calorimetry that is recognized at an international level is benzoic acid obtained from the National Bureau of Standards (NBS-Standard Sample 39), with a guaranteed gross calorific value.



**If a calorimeter is being operated with more than one decomposition vessel, the heat capacity of the system must be determined for each decomposition vessel.**

For more detailed information on calibration, please refer to the appropriate standards as they are listed in Section 4 *Features of the system*.

## 4 Features of the system

The **C 5000 control** and **C 5000 duo-control** calorimeter systems are used for routine determinations of the gross calorific value of solid and liquid substances. The two systems conform to all gross calorific value standards in accepted use, and are thus recognized worldwide. The extensive selection of accessories and the modular design of the systems ensure customized adaptation to laboratory tasks. During the process of an experiment, the software takes care of communication with external devices (for example analytical scale, sample rack) as well as management of samples, decomposition vessels and experiment results that eliminates mix-ups.

The two systems are distinguished by the following features:

- A fully automated measurement procedure eliminates the need for time-consuming routine tasks.
- Integrated oxygen filling and degassing.
- Measurement of gross and net calorific value according to DIN 51900, BS 1016 Part 5 1977, ASTM D3286-91, ASTM D240-87, ASTM E711-87, ISO 1928-1976, ASTM D1989-91 and BSI.
- Measurement range: max. 40,000 J  
This corresponds to an increase in temperature within the inner vessel of 4K.
- Work can be performed based on the adiabatic, isoperibolic or dynamic principle.
- Calculation of the gross calorific value based on the following correction methods:
  - standard without titration
  - standard with titration
  - carbon: hydrogen without titration
  - carbon: hydrogen with titration
  - carbon: volatile component parts without titration
  - carbon: volatile component parts with titration
  - acid correction based on ASTM 240
  - acid correction based on ASTM 1989



## 5 Transportation, storage and setup location

### 5.1 Conditions for transportation and storage

The system must be protected from mechanical bumps, vibrations, accumulations of dust and corrosive ambient air during transportation and storage. It is also important to observe that the relative humidity not exceed 80%. If the system is shipped back to the factory, only the original packaging may be used.

### 5.2 Setup location

To ensure high precision in measurements, a constant ambient temperature is required for the system. The following conditions must therefore be observed at the setup location:

- No exposure to direct sunlight.
- No drafts (for example next to windows, doors, air conditioners).
- A sufficient distance from heater blocks and other sources of heat.
- Adequate circulation of air must be ensured to divert the system's own heat.
- The minimum distance between the wall and the rear side of the unit must not be less than 25 cm.
- The system must not have laboratory material such as shelves, cable sleeves, ring leads, etc, built over it.
- The room temperature must fall within the range of 20 - 25°C.
- The system must be set up on a horizontal surface.



To operate the system, the setup location must provide a power supply that conforms to the specifications on the rating plates of the system components, as well as a supply of oxygen (99.95% pure oxygen, quality 3.5, pressure 30 bar) with the appropriate pressure indicator.

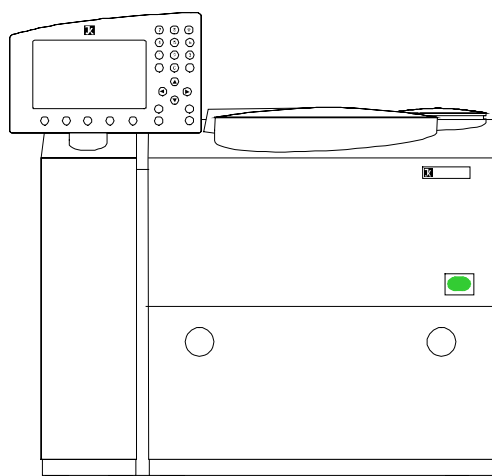
## 6 Unpacking

Please unpack the unit carefully and make note of any damages. It is important that any damage that occurred during shipping be noted at once while unpacking. If damage has occurred, you should take stock of this damage immediately (noting whether by mail, rail or express delivery, etc.).

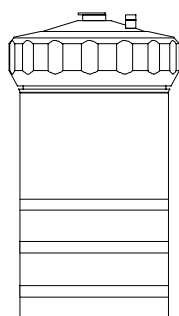
The following sections describe the entire range of components included with delivery, including the various system variants.

### 6.1 Included with delivery of the C 5000 control package 1

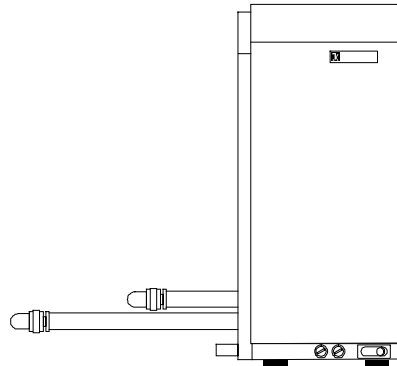
The packing for the C 5000 control package 1 contains:



- 1x Basic unit consisting of a controller with measurement cell
- 1x Accompanying set
- 1x Aqua-pro
- 1x Operating instructions



- 1x C50xx decomposition vessel
- 1x C 5050 set of working items



1x C 5001 cooling system

1x O<sub>2</sub> pressure hose:

Length: 5 m

Connections:  
 1 x M8x1; Opening 10  
 1 x 1/4"; Opening 17

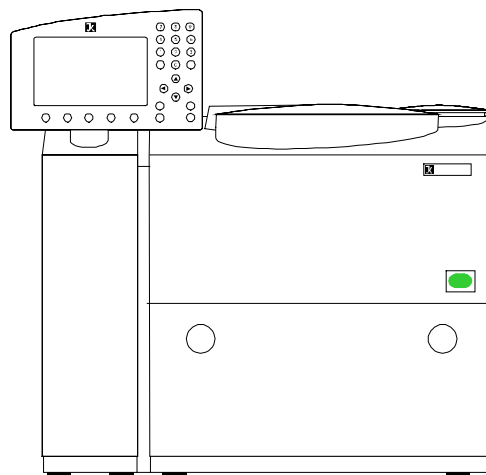


1x venting hose

Length: 1.5 m  
 Connection: M6, Opening 8

## 6.2 Included with delivery of the C 5000 control package 2

The packing for the C 5000 control package 2 contains:

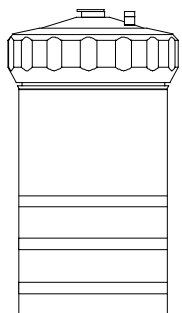


1x Basic unit consisting of a controller with measurement cell

1x Accompanying set

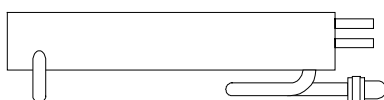
1x Aqua-pro

1x Operating instructions



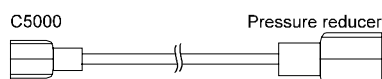
1x C50xx decomposition vessel

1x C 5050 set of working items



1x C 5004 cooling system

1x C 5004 datasheet



1x O<sub>2</sub> pressure hose:

Length: 5 m

Connections:

1 x M8x1; Opening 10

1 x 1/4"; Opening 17



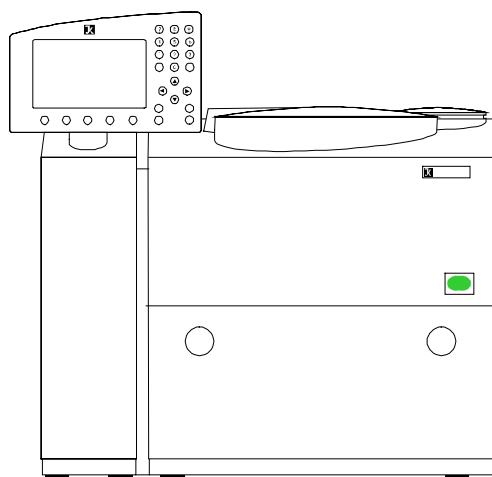
1x venting hose

Length: 1.5 m

Connection: M6, Opening 8

### 6.3 Included with delivery of the C 5000 duo-control package 3

The packing for the C 5000 duo-control package 3 contains:

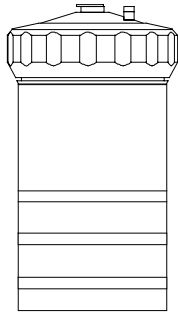


1x Basic unit consisting of a controller with measurement cell

1x Accompanying set

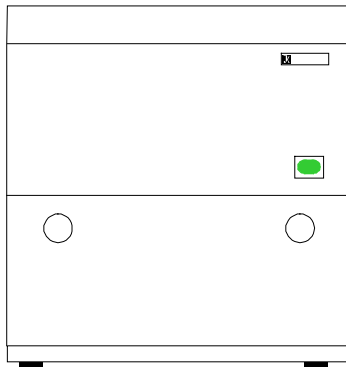
1x Aqua-pro

1x Operating instructions

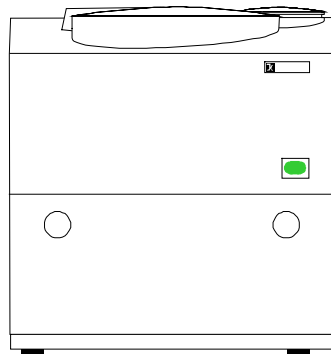


2x C50xx decomposition vessel

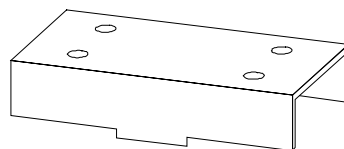
1x C 5050 set of working items



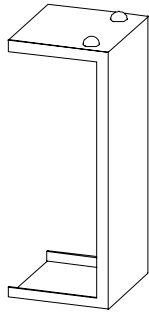
1x C5002 cooling system



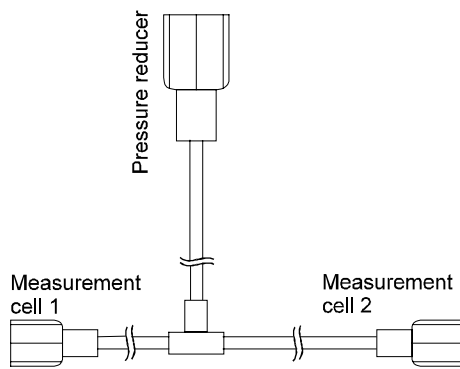
1x measurement cell



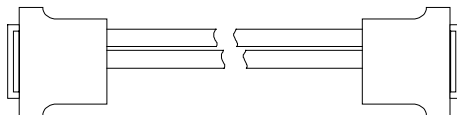
2x connection pieces



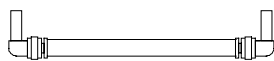
3x pivot plates



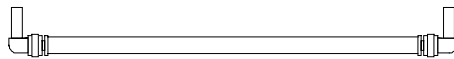
1x O<sub>2</sub> pressure hose:  
 Length: 5 m  
 Connections:  
 2 x M8x1; Opening 10  
 1 x 1/4"; Opening 17



1x Extension for control and connection cable

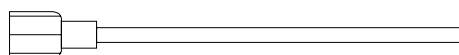


2x water hose, short



2x water hose, long

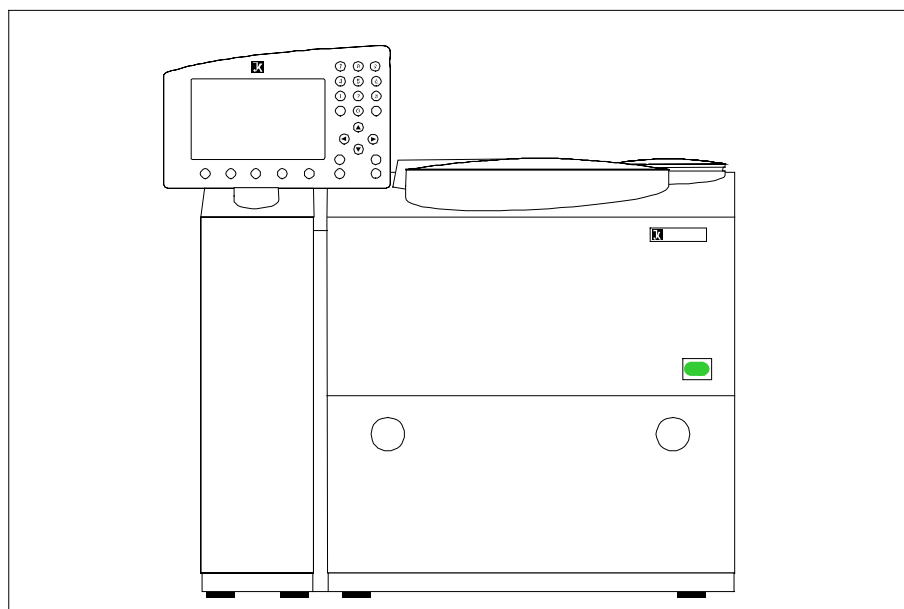
C5000



2x venting hose

## 7 Description of the system components

### 7.1 Controller with measurement cell

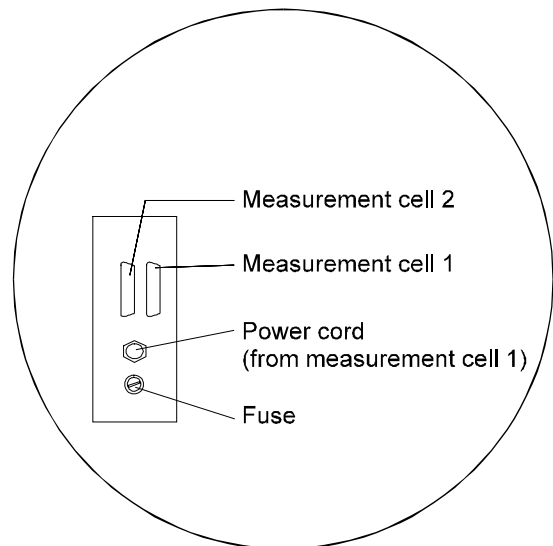
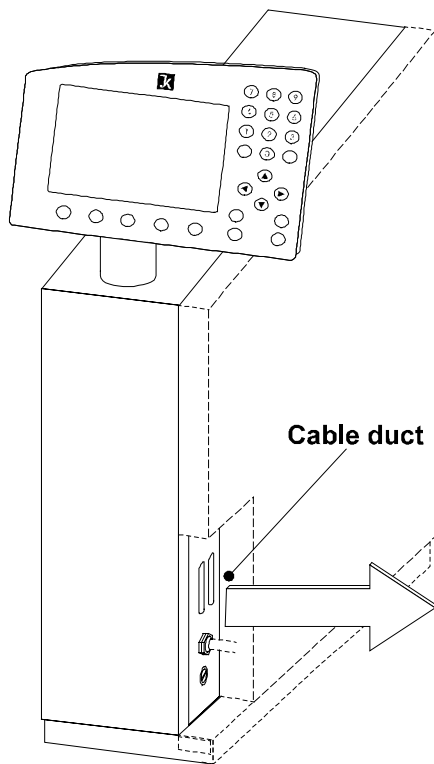
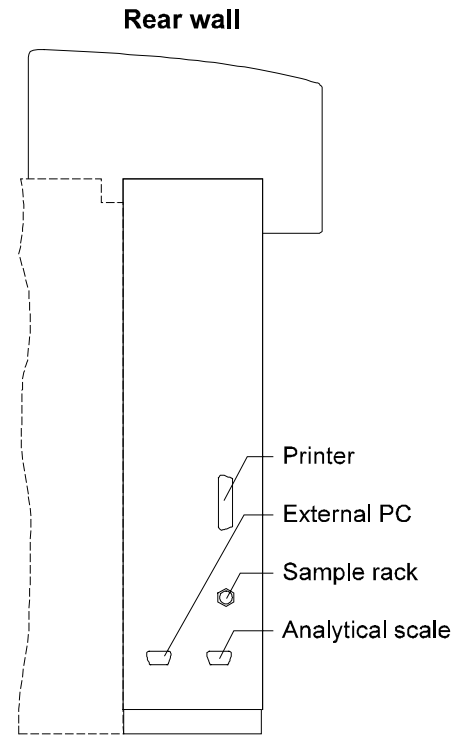
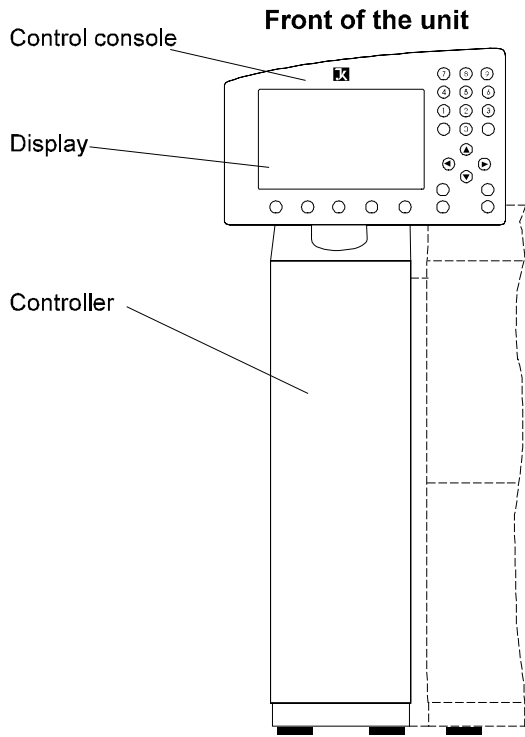


Controller with  
measurement cell

Together with the measurement cell, the controller makes up the core of the calorimeter system.

The controller works as a central control, interface and display unit for all system components. Operating commands and experiment parameters can be entered through the control console (see the following illustration).

During a gross calorific value test, it monitors and controls all phases of the measurement process. Current system states and test data appear on the display. To ensure that the experiment proceeds with no problems, the components of the system are monitored constantly. If malfunctions arise, the display generates a message. The results of the experiment are stored together with the parameters of the experiment and can be printed out if desired.



**Controller:** device connections

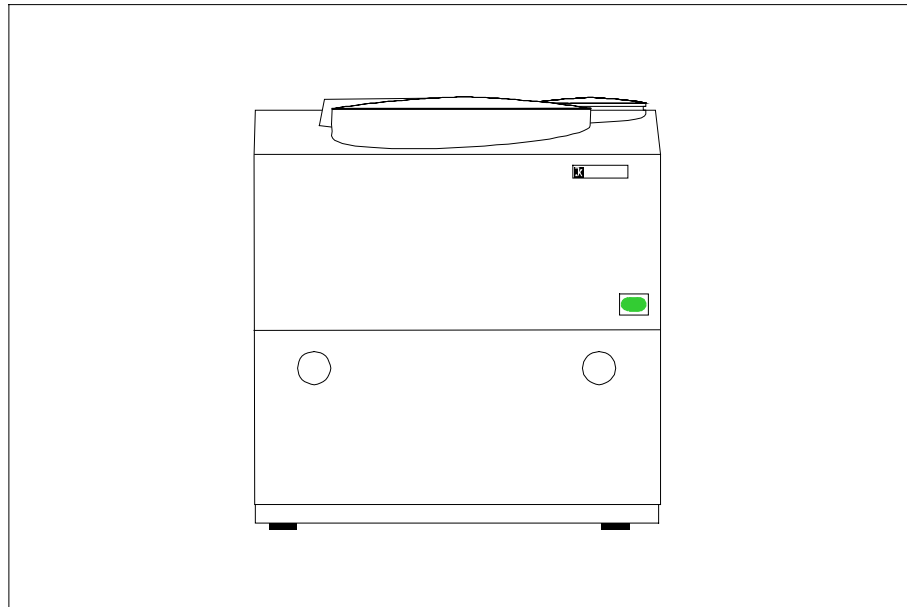


The individual tasks performed by the controller are as follow:

- Dialog with the user through the control console
- Store experiment data and experiment protocols ordered by experiment, experiment documentation
- Perform experiments automatically, control and monitoring of measurement cell(s)
- Communication with the peripheral devices:  
Printer, analytical scale, sample rack, external PC

### Technical data for the controller

Operating power	Electrical power is supplied through measurement cell to conform to rating plate.
Power consumption	C 5000 control: max 1300 Watts (controller with one measurement cell) C 5000 duo-control: max. 2500 Watts (controller with two measurement cells)
Device fuses	1 x 3.15 A, T; 230V / 1 x 6.25 A, T; 100V, 115V
Dimensions (WxDxH)	560 x 380 x 397 mm (Controller with measurement cell, w/o display)
Weight	41 kg (controller with measurement cell)
Ambient temperature	15 ... 30°C
Permissible humidity	80%
Enclosure rating	IP 21
Display	320 x 200 pixels, with illuminated background
Contamination level	II
Over-voltage category	2
Enclosure rating	1 (protective ground)



Measurement cell

The combustion of fuel samples takes place in the measurement cell under precisely defined conditions. When the gross calorific value is being determined, the measurement cell takes care of the following experiment conditions:

**Experiment conditions**

- Adiabatic measurement method according to DIN 51900 T3, ASTM 240 D
- Isoperibolic measurement method based on ASTM 1989 D
- Dynamic measurement method (same as adiabatic but shorter in time)

**Measurement cell components**

In order to achieve these experiment conditions, the following components are housed in the measurement cell

- Inner vessel with a water jacket
- Magnetic stirrer to create even distribution of heat within the inner vessel
- A water system with pump, expansion container and connection for an external cooling unit
- Heater and temperature controller
- O<sub>2</sub> filling and degassing device

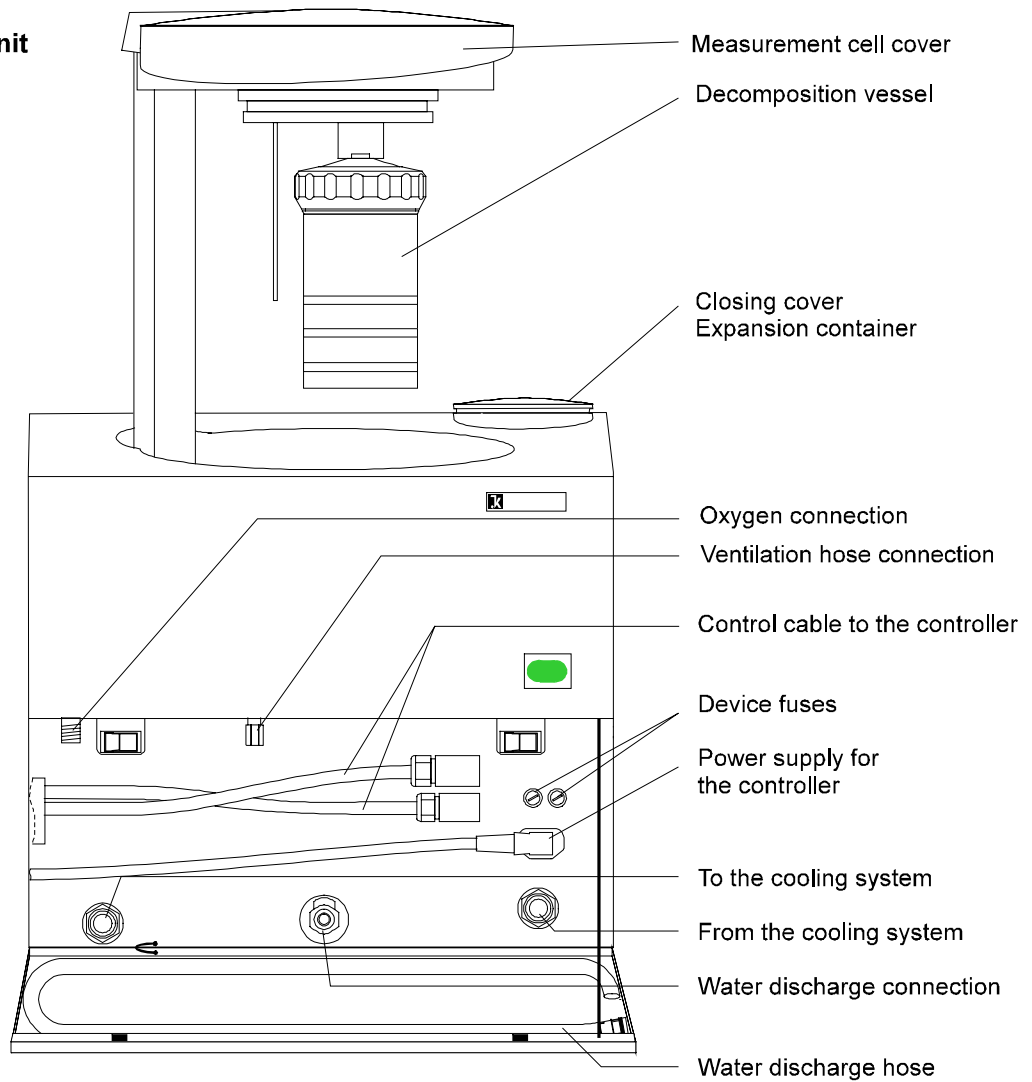
The measurement cell receives the signals for performing the individual steps of the experiment from the controller. The controller records and monitors the experiment data and operating states that are recorded by the sensors in the measurement cell.

The following processes take place during determination of gross calorific value in the measurement cell:

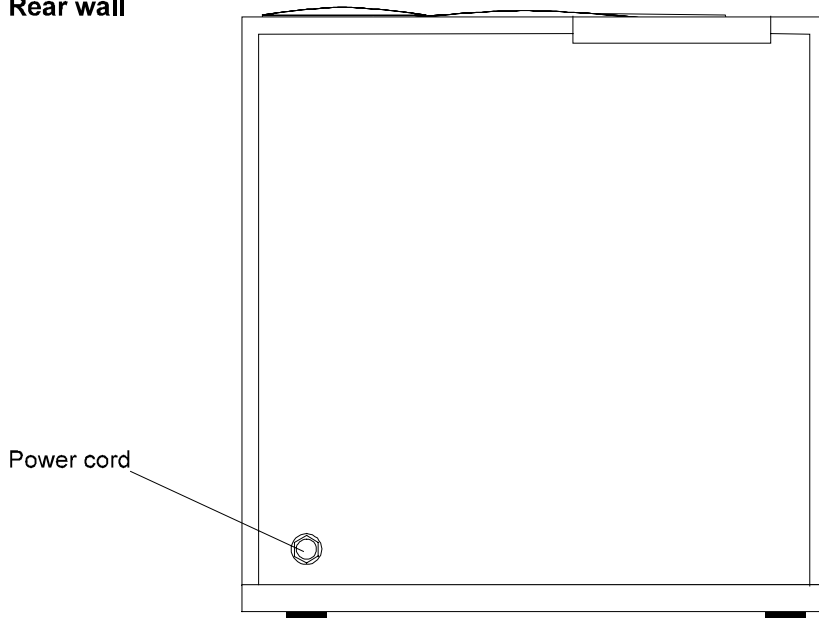
**Experiment  
process**

- The cover of the measurement cell closes automatically and the decomposition vessel with the fuel sample is immersed into the inner vessel.
- Pure oxygen flows through the oxygen filling device into the decomposition vessel until the pressure preset by the user is reached (normally 30 bar).
- The pump fills the inner vessel and takes care of circulation in the water system.
- The magnetic stirrer keeps the water in the vessel constantly in motion so that heat is distributed evenly.
- The fuel sample is electrically ignited by the ignition device.
- The water in the circuit is cooled off by an external cooling unit and is then heated back up to the required temperature by the heater in the measurement cell.
- After the end of the experiment, the over-pressure is allowed to escape from the decomposition vessel, the inner vessel is emptied and the cover of the measurement cell is opened. The decomposition vessel can then be removed.

Front of the unit



Rear wall



Measurement cell: device connections

### Technical data on the C 5003 measurement cell

Operating voltage	See rating plate
Power consumption	See controller
Device fuses	2 x 6.25 A, T; 230 V / 2 x 15 A, T; 100V, 115V
Dimensions (WxDxH)	440 x 380 x 397 mm
Weight	34 kg
Ambient temperature	15 ... 25°C
Permissible humidity	80%
Enclosure rating	IP 21

### Technical data on the decomposition vessel

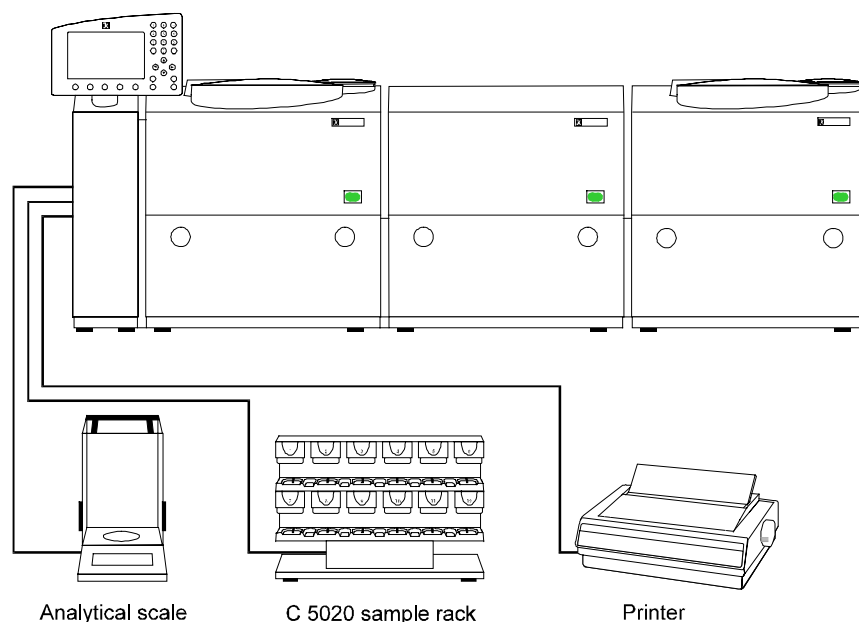
See information inscribed on the decomposition vessel and the manufacturer's certificate as well as the Operating Instructions for the C5010 and C5012 decomposition vessels.

#### System components, peripheral devices

With the maximum number of components included and attached, the calorimeter system includes the following components:

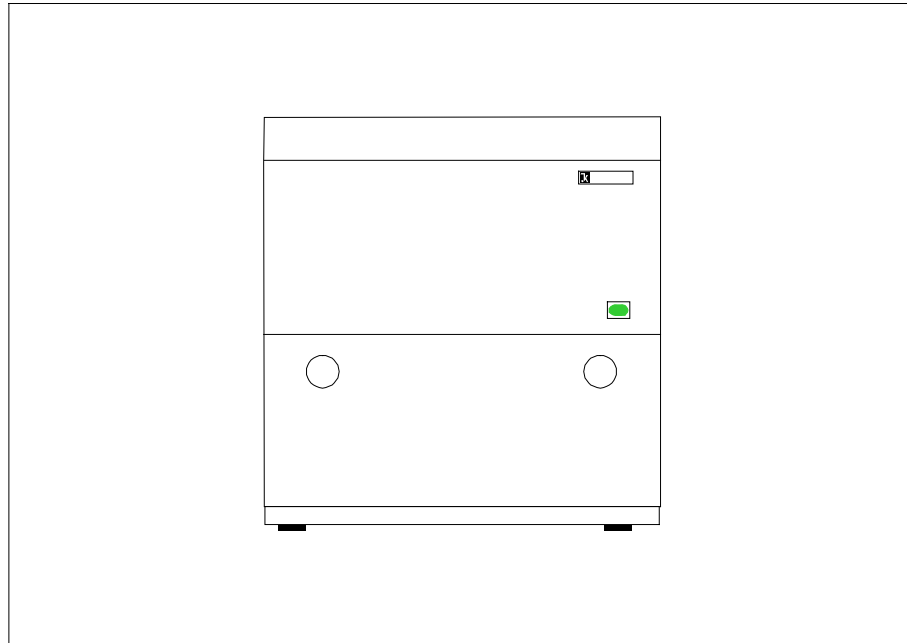
System components:      Measurement cell 1 with controller  
    Measurement cell 2  
    C 5002 cooling system

Peripheral devices:      Printer  
    Analytical scale  
    C5020 sample rack



**C5000 calorimeter system:**  
 System components and peripheral devices with maximum number of components

## 7.2 C 5002 cooling system



C5002 cooling system

The C5002 cooling system cools the water systems of the two measurement cells. One heat exchanger takes care of the cooling required for each circuit. A compressor with a liquifier and an evaporator generates sufficient cooling output for two measurement cells of the C 5000 calorimeter system.

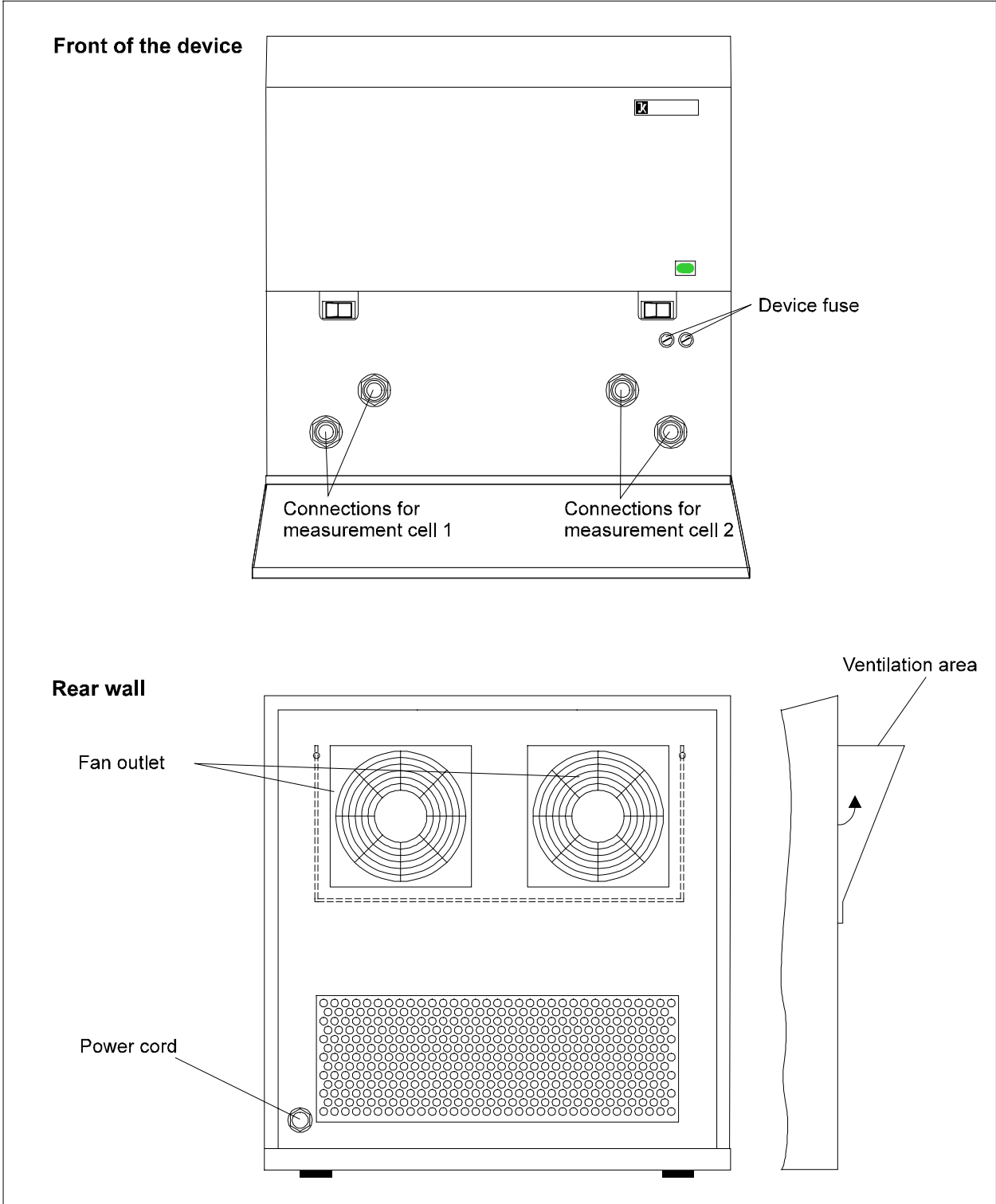
The ventilator takes in cool air through the bottom of the unit to draw off the heat it generates. The air escapes back out of the unit through ventilation slits in the rear wall.

### Technical data for the C 5002 cooling system

Operating power	See rating plate
Power consumption	Max 700 Watts
Cooling output	2 x 300 Watts
Device fuses	2 x 4.0 A, FF; 230V / 2 x 8.0 A, FF; 100V, 115V
Dimensions (WxDxH)	440 x 380 x 397 mm
Weight	33 kg
Ambient temperature	15 ... 25°C
Permissible humidity	80%
Enclosure rating	IP 21
Contamination level	II
Over-voltage category	2
Enclosure rating	1 (protective ground)

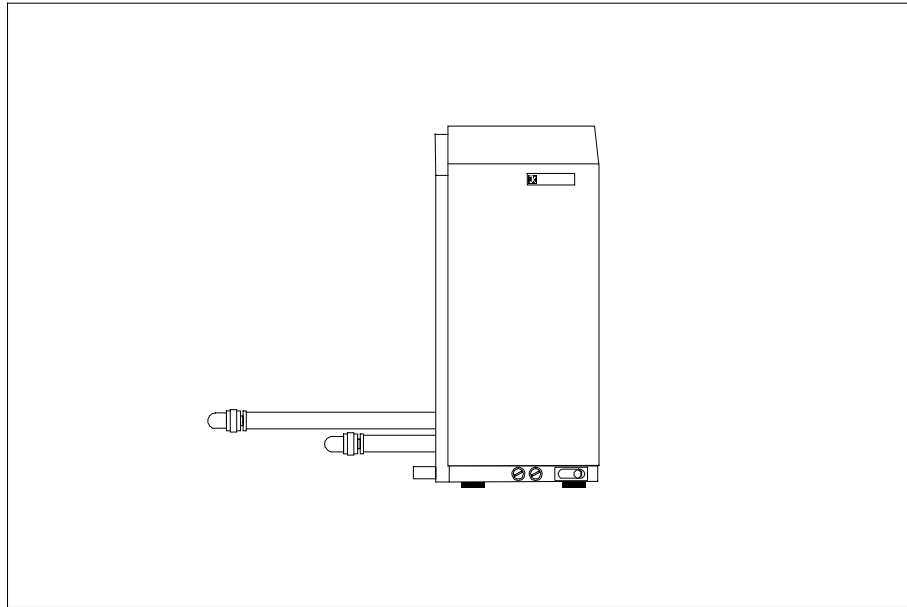


**For the operating security of the entire system, both measurement cells should always be in active operating mode (the OK window confirmed). This also applies when working with only one measurement cell.**



C 5002 cooling system

### 7.3 C 5001 cooling system



C5001 cooling system

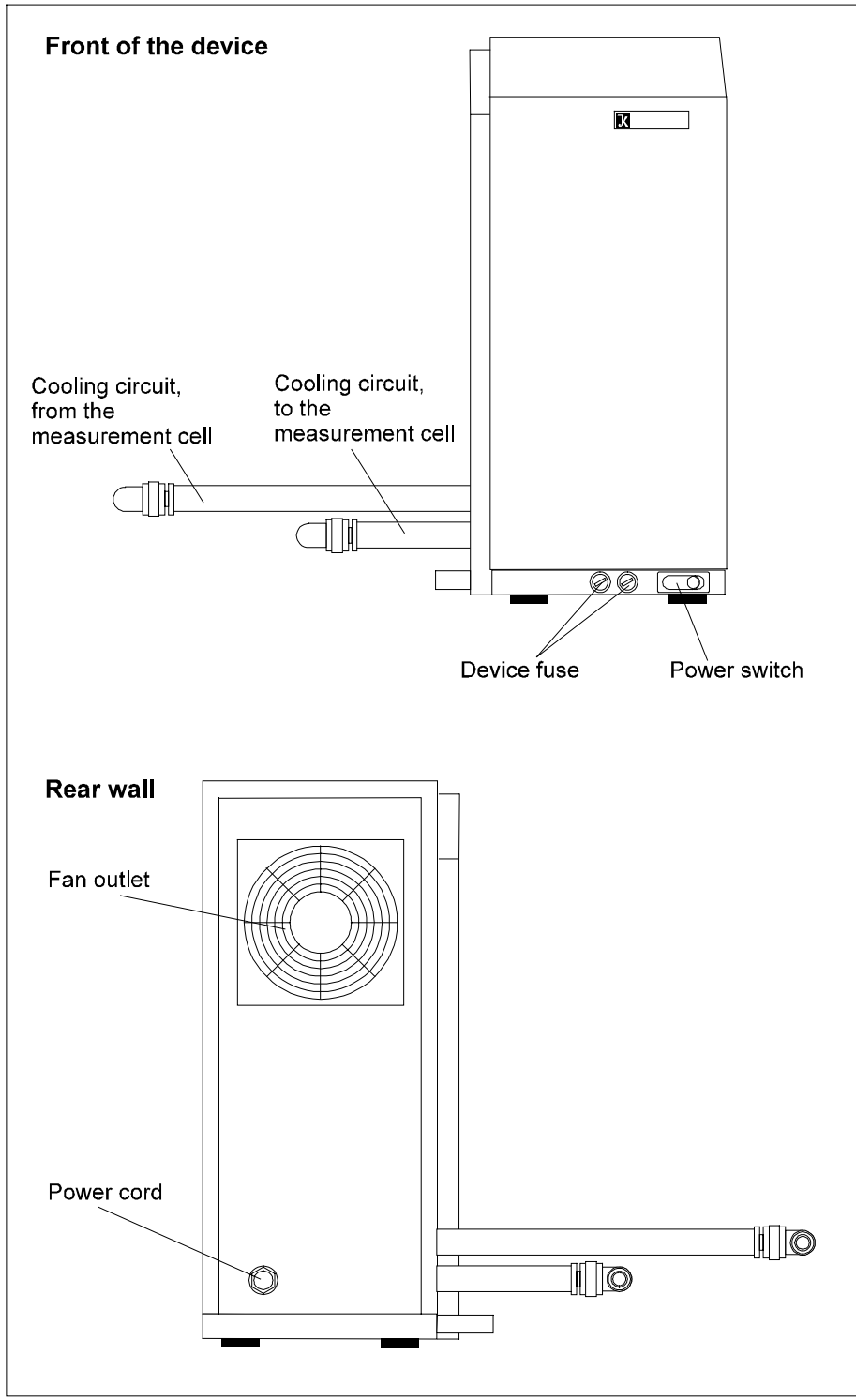
The C5001 cooling system cools the water systems of one measurement cell. One heat exchanger takes care of the cooling required for the circuit. A compressor with a liquifier and an evaporator generates sufficient cooling output for the measurement cell of the C 5000 calorimeter system.

The ventilator takes in cool air through the bottom of the unit and the rear wall to draw off the heat it generates. The ventilator then forces the air back out of the unit through ventilation slits in the rear wall.

#### Technical data for the C 5002 cooling system

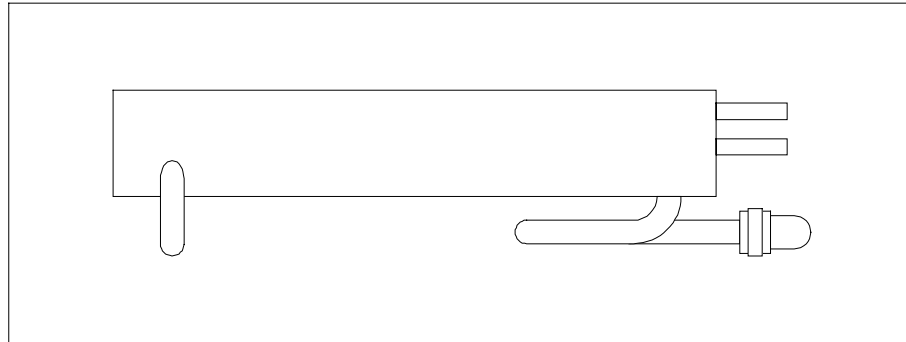
Operating power	See rating plate
Power consumption	Max 300 Watts
Cooling output	240 Watts
Device fuses	2 x 3.0 A, FF; 230V / 2 x 6.0 A, FF; 100V, 115V
Dimensions (WxDxH)	180 x 380 x 397 mm
Weight	17 kg
Ambient temperature	15 ... 25°C
Permissible humidity	80%
Enclosure rating	IP 21
Contamination level	II
Over-voltage category	2
Enclosure rating	1 (protective ground)





C 5001 cooling system

## 7.4 C 5004 cooling system



C5004 cooling system

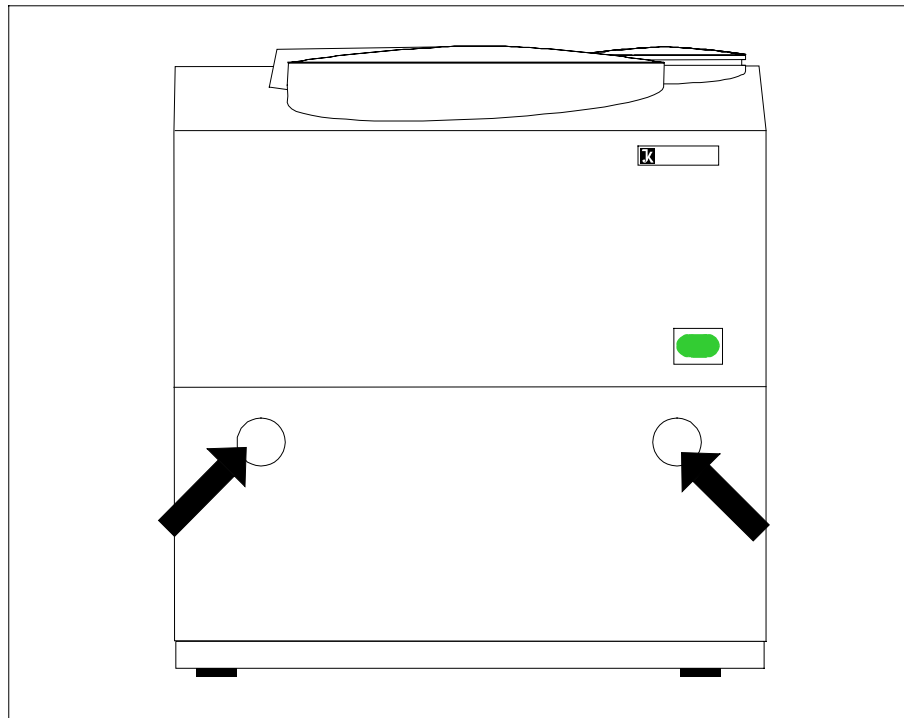
The C 5004 cooling system cools the water system with one measurement cell. The secondary circuit of the system is connected to an external water supply to divert heat.

### Technical data on the C 5004 cooling system

See data sheet C5004 (included with delivery)

## 8 Setting up and placing in service

The components of the C 5000 calorimeter system are unpacked and are located at the place where you will set them up (see Section 5, paragraph 5.2 on the location for setting up the unit). Open the front flap of the measurement cell or of the two measurement cells for the C 5000 duo-control by pushing on both recesses at the same time.



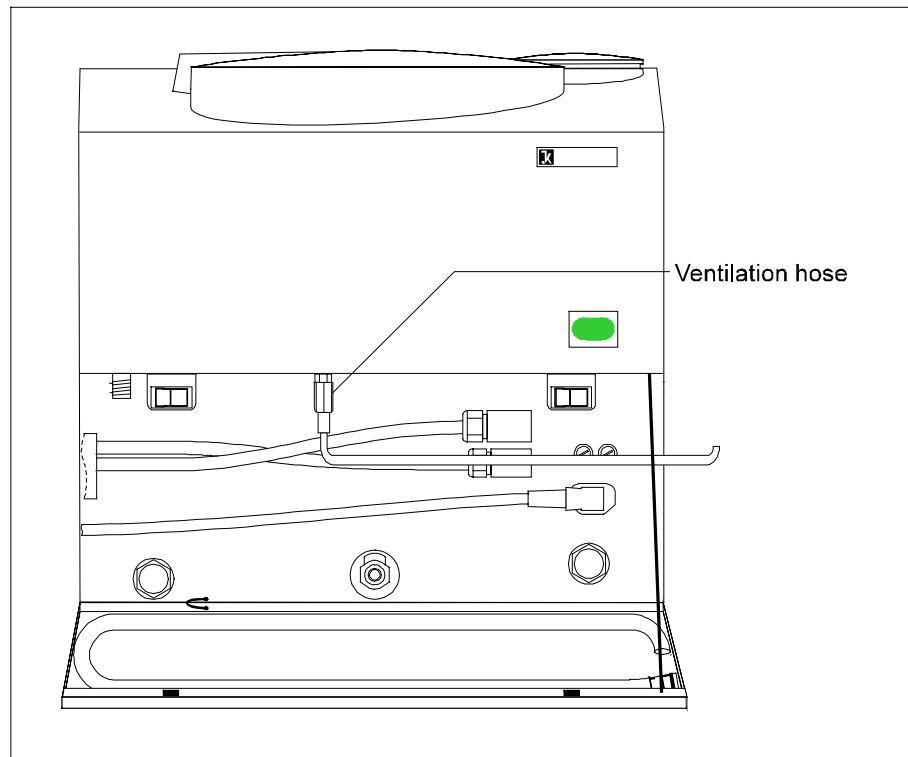
Opening the front flap

Then carry out each of the following steps:

## 8.1 Setting up package 1

①

Install the ventilation hose according to the following illustration:



**Ventilation hose:**  
Guide the hose along  
the right side of the  
housing towards the  
back.

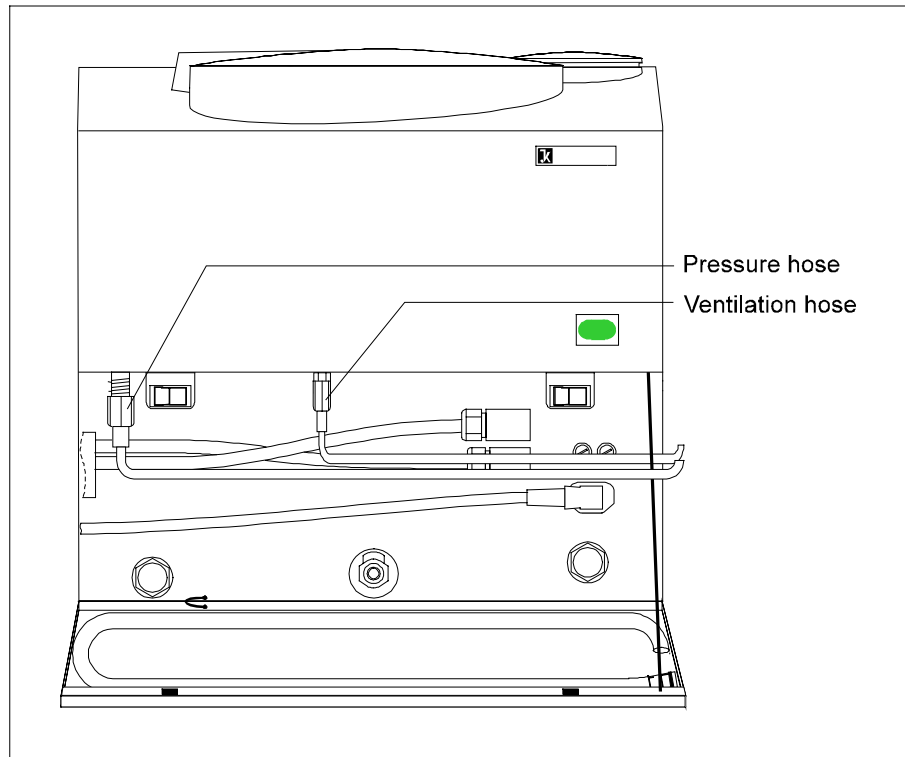
The combustion gasses are discharged through the ventilation hose after each combustion experiment. The ventilation hose should not be squeezed or kinked while the hose is being laid.



**Since combustion gasses are hazardous to your health, the ventilation hose should be connected to an appropriate device for purifying or drawing off gas (C7048.2).**

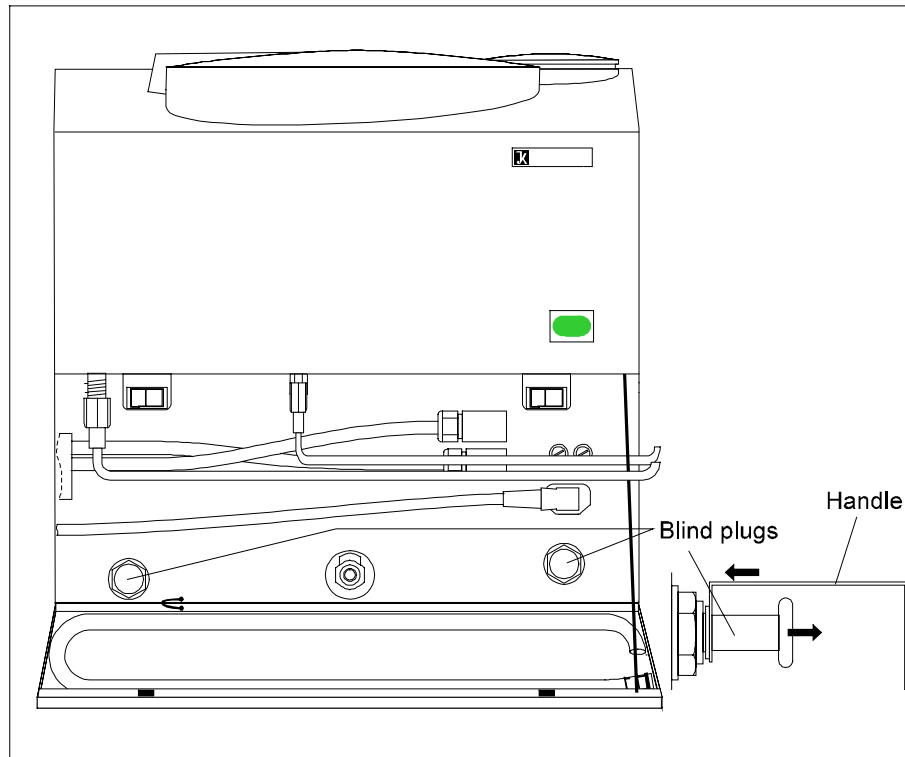
②

Screw the pressure hose (O<sub>2</sub> line) with the M8x1 cap screw onto the oxygen connection sleeve of the measurement cell with an open-ended spanner (opening 10, included with delivery), and install the hose according to the following illustration:



③

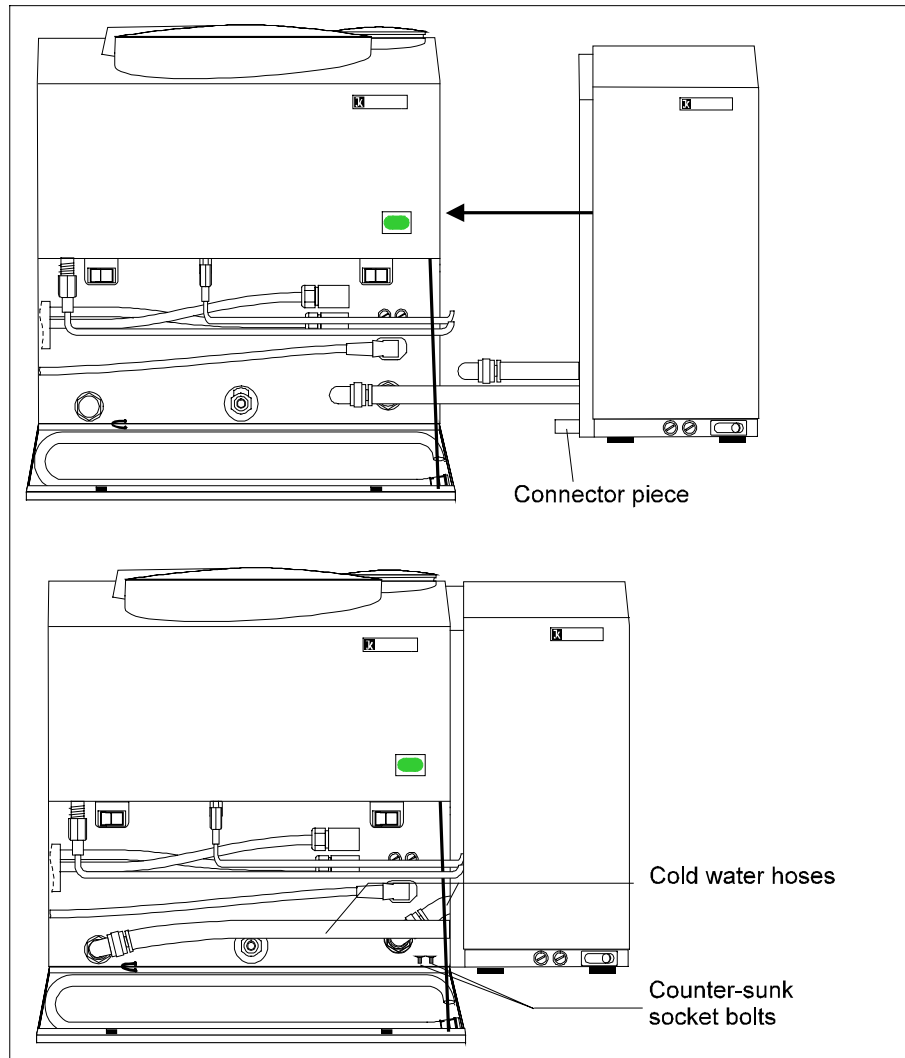
Using the handle (included with delivery of the decomposition vessel), remove the blind plugs from the cold water connections of the measurement cell. Removing the blind plugs allows residual water to escape. This water should be captured with an absorbent pad.



Removing the blind plugs

④

Place the C 5001 cooling system next to the measurement cell. Push the cooling system all the way up to the measurement cell. The joining piece on the C 5001 cooling system fits into the opening on the measurement cell that lines up with it. Fasten the two components in place by screwing in the two counter-sunk socket bolts. Insert the hoses into the water connections of the measurement cell.



Installing the C 5001 cooling system onto the measurement cell

⑤

Connect the pressure hose on the laboratory oxygen supply end.

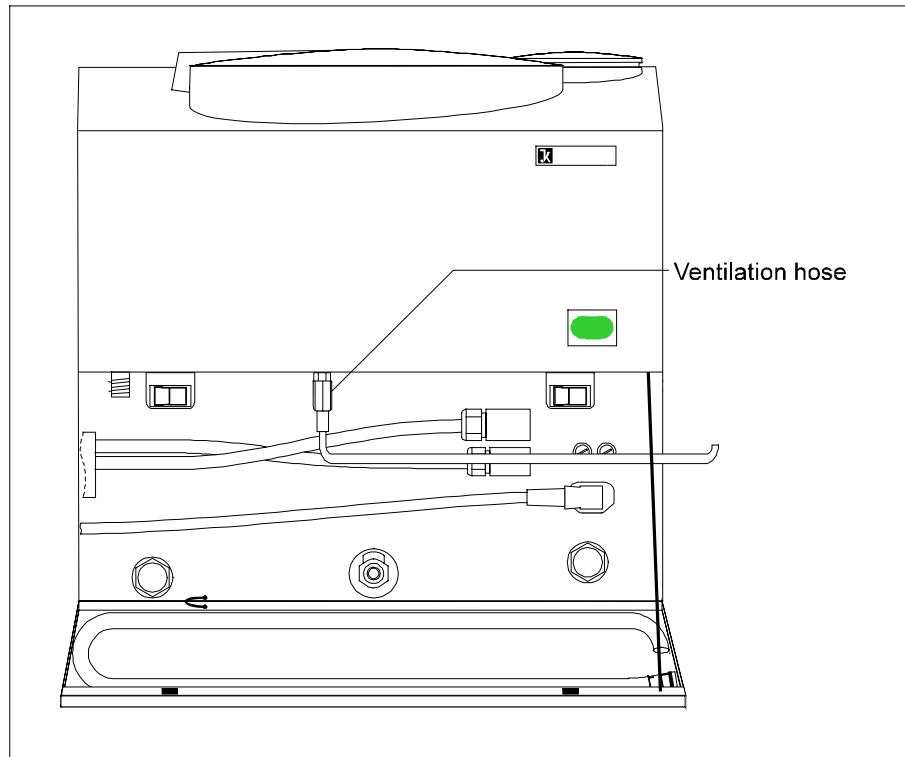


**The pressure of the oxygen should be 30 bar, but must not in any case exceed 40 bar. You should use oxygen of quality 3.5 (99.95 pure oxygen).**

## 8.2 Setting up package 2

①

Open the front flap of the measurement cell and install the ventilation hose according to the following illustration:



**Ventilation hose:**  
Guide the hose along  
the right side of the  
housing towards the  
back.

The combustion gasses are discharged through the ventilation hose after each combustion experiment. The ventilation hose should not be squeezed or kinked while the hose is being laid.

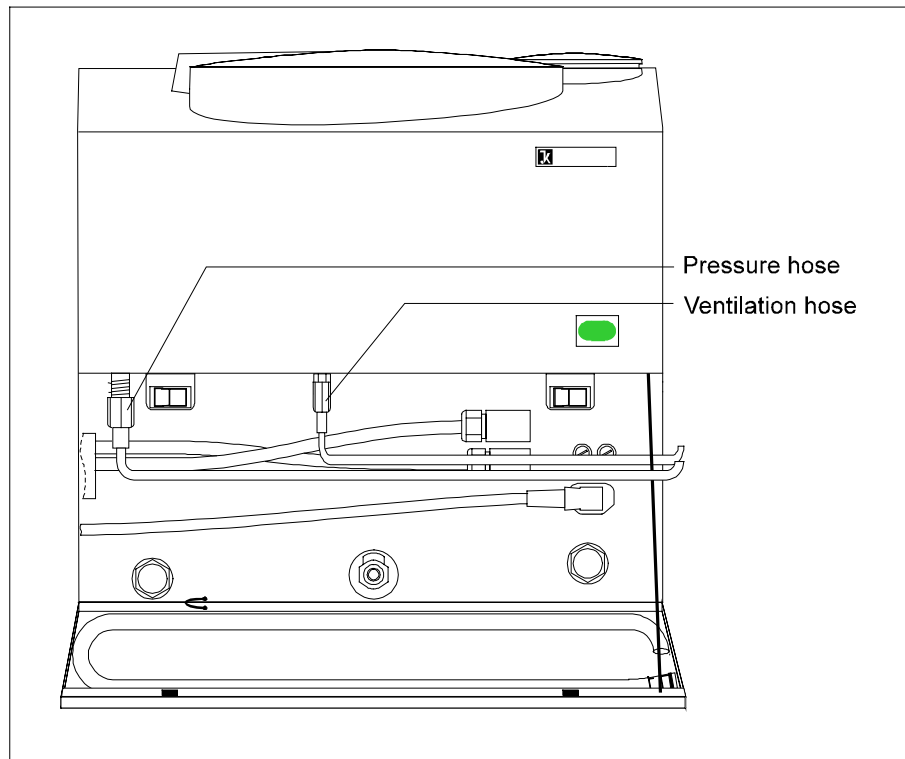


**Since combustion gasses are hazardous to your health, the ventilation hose should be connected to an appropriate device for purifying or drawing off gas (C7048.2).**



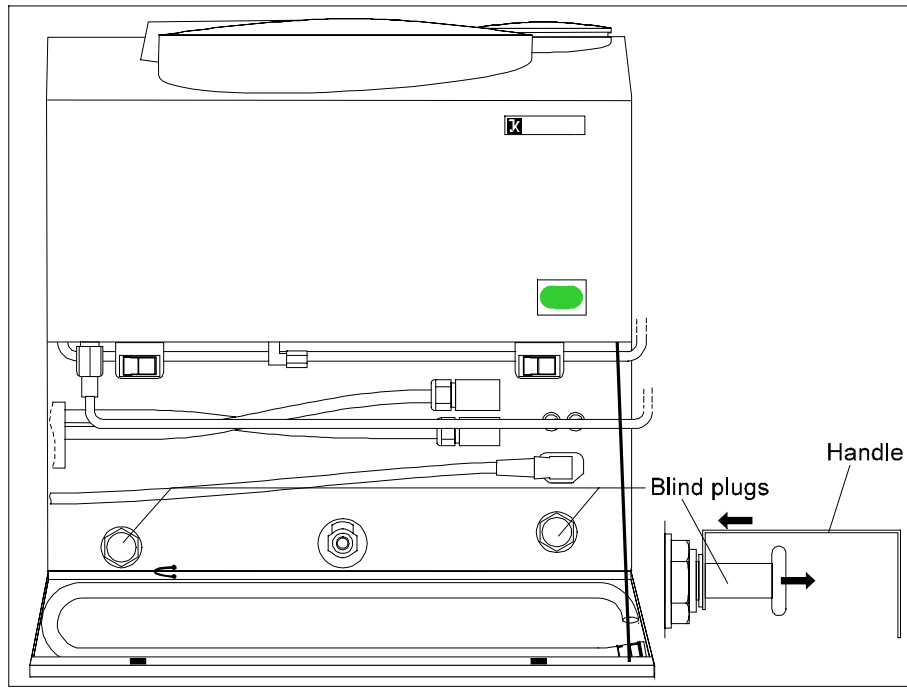
②

Screw the pressure hose (O<sub>2</sub> line) with the M8x1 cap screw onto the oxygen connection sleeve of the measurement cell (with an open-ended spanner, opening 10, included with delivery), and install the hose according to the following illustration:



③

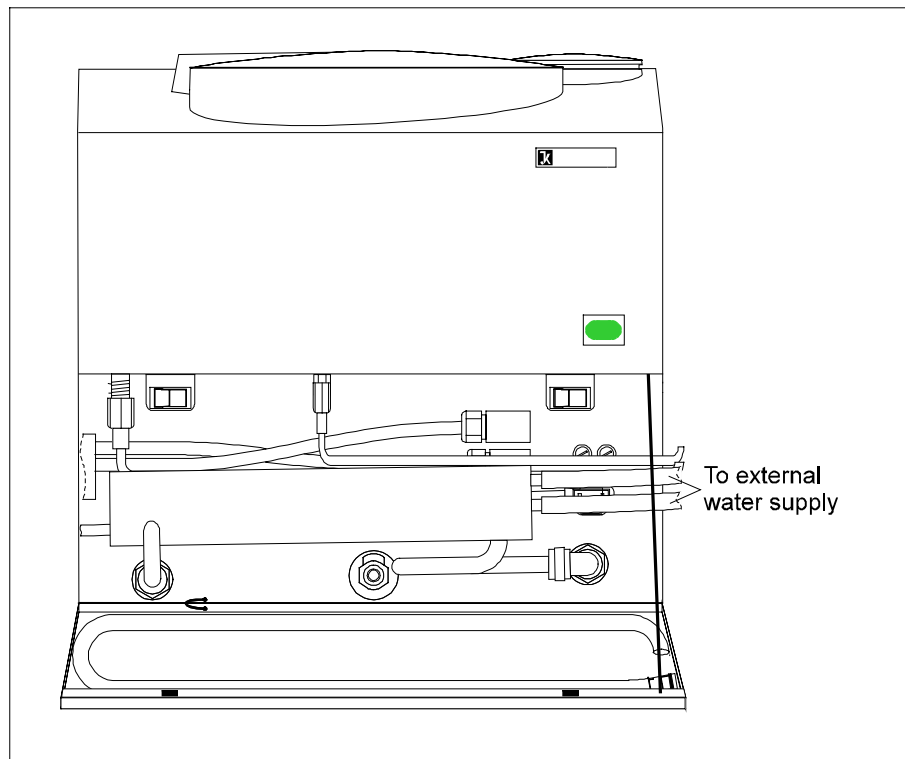
Using the handle (included with delivery of the decomposition vessel), remove the blind plugs from the cold water connections of the measurement cell. Removing the blind plugs allows residual water to escape. This water should be captured with an absorbent pad.



Removing the blind plugs

④

Place the C 5004 cooling system on the water connection of the measurement cell:



Installing the C 5004 cooling system onto the measurement cell

⑤

For information on connecting and operating the C5004 cooling system, see the datasheet included with delivery.

⑥

Connect the oxygen hose on the laboratory oxygen supply end.

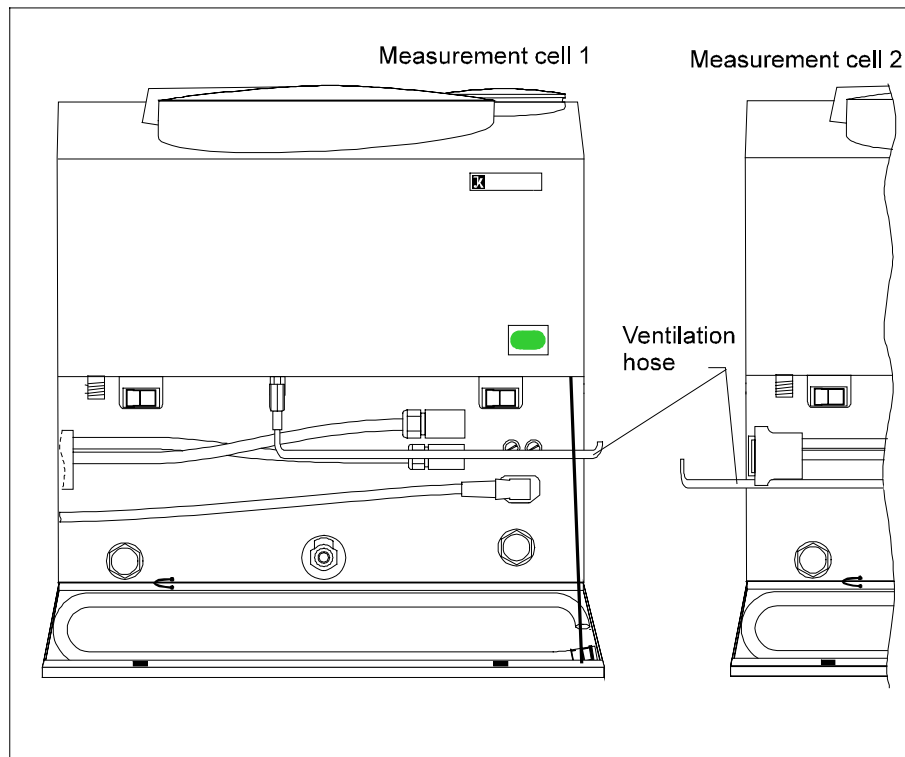


**The pressure of the oxygen should be 30 bar, but must not in any case exceed 40 bar. You should use oxygen of quality 3.5 (99.95 pure oxygen).**

### 8.3 Setting up package 3

①

Open the front flaps of both measurement cells and install the ventilation hose according to the following illustration:



**Ventilation hose:**  
Guide the hose of each measurement cell along the right or the left side of the housing towards the back.

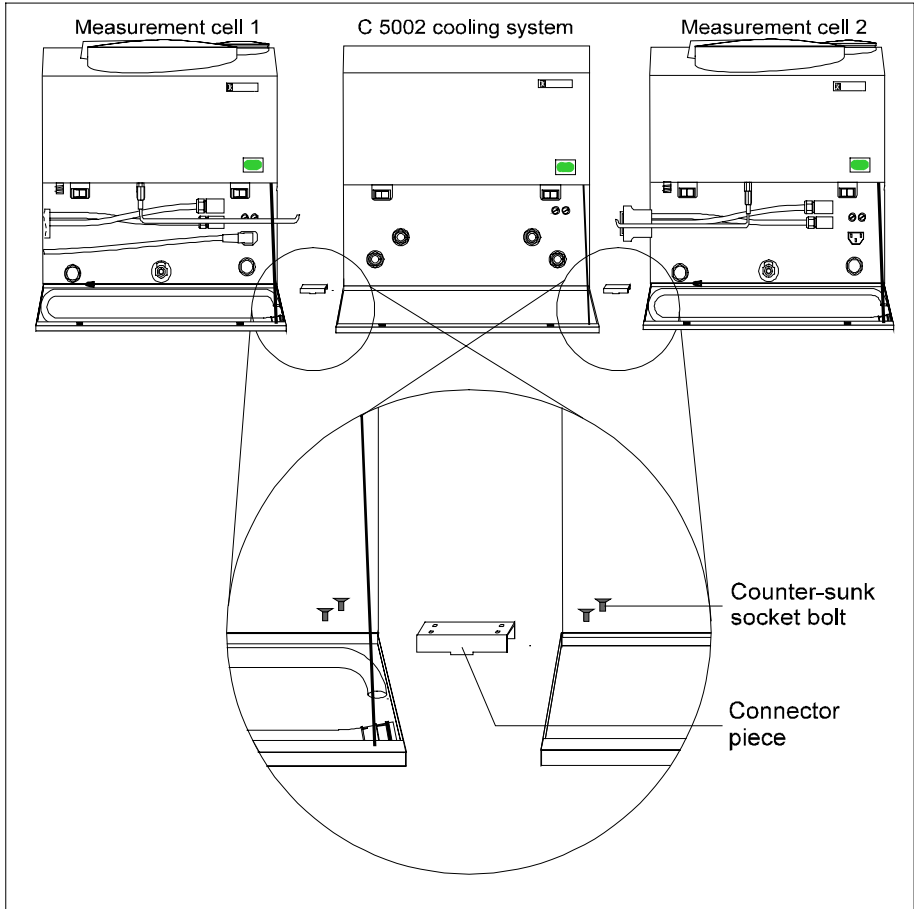
The combustion gasses are discharged through the ventilation hose after each combustion experiment. The ventilation hose should not be squeezed or kinked while the hose is being laid.



**Since combustion gasses are hazardous to your health, the ventilation hose should be connected to an appropriate device for purifying or drawing off gas (C7048.2).**

②

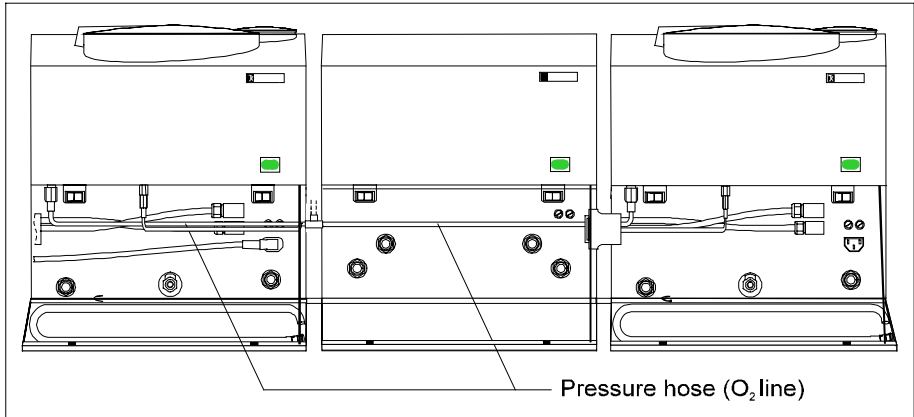
Assemble the basic unit, the C 5002 cooling system and the second measurement cell together as illustrated in the following illustration:



Assemble the measurement cell and cooling unit together

③

Screw the pressure hose with the two M8x1 cap screws onto the oxygen connection sleeves of the measurement cells (with an SW10 open-ended spanner, included with delivery).

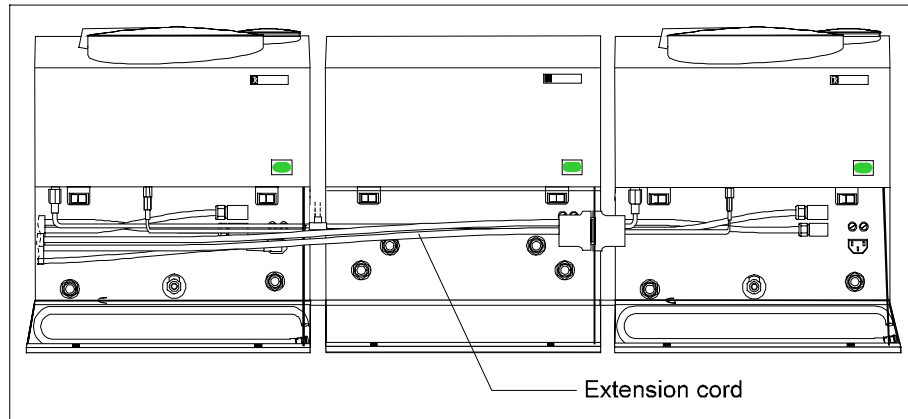


Assembling of the pressure hose

④

Connect the second measurement cell with the controller through the extension cord. The plugs should be screwed in place.

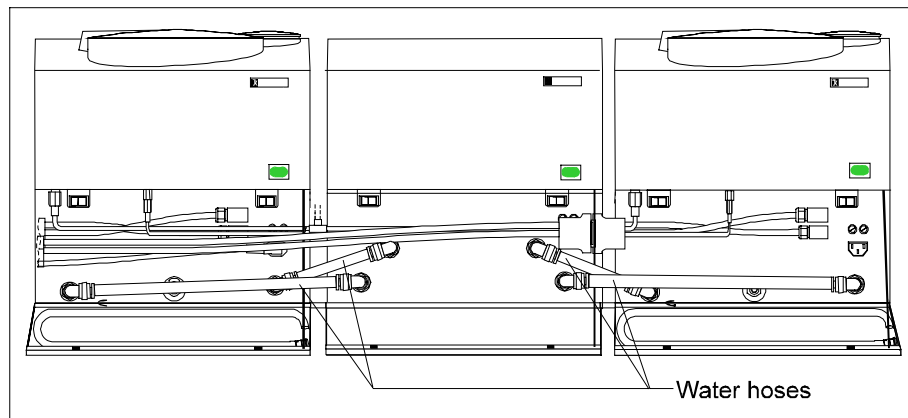
Assembling the extension cord



⑤

Remove the blind plugs and insert the water hoses into the water hose connections of the cooling system and the two measurement cells (see Section 8.2, Part 3).

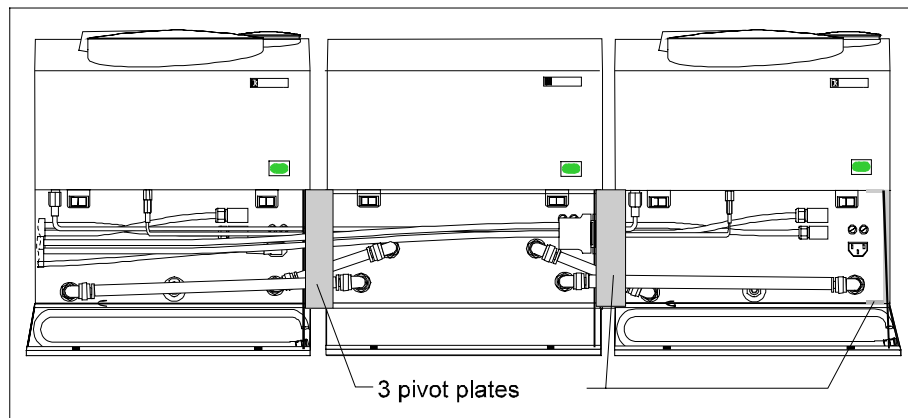
Assembling the water hoses



⑥

Set the pivot plates in place.

Setting the pivot plates in place



⑦

Connect the pressure hose on the laboratory oxygen supply end.

**The pressure of the oxygen should be 30 bar, but must not in any case exceed 40 bar. You should use oxygen of quality 3.5 (99.95 pure oxygen).**

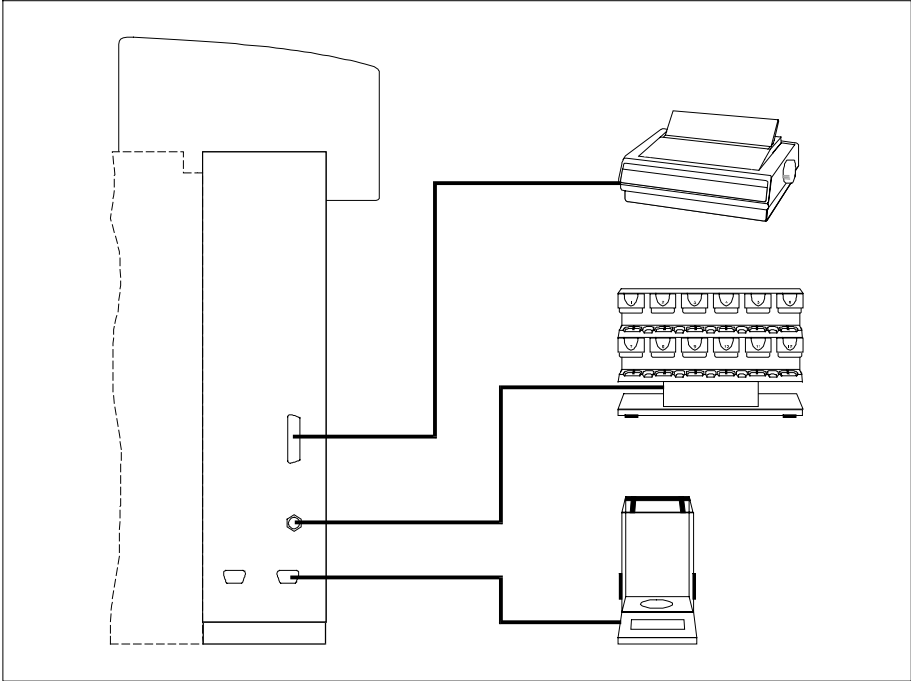
### 8.4 Connecting peripheral devices

If sample racks, electronic scale or a printer have been delivered with the calorimeter, they should be connected now.

The connection sockets are located on the rear wall of the controller. When connecting the sample rack, take note of the labeling for the connection cable.



**The power to peripheral devices must be turned off while they are being connected to the power switch.**



Connecting the printer, sample rack and scale

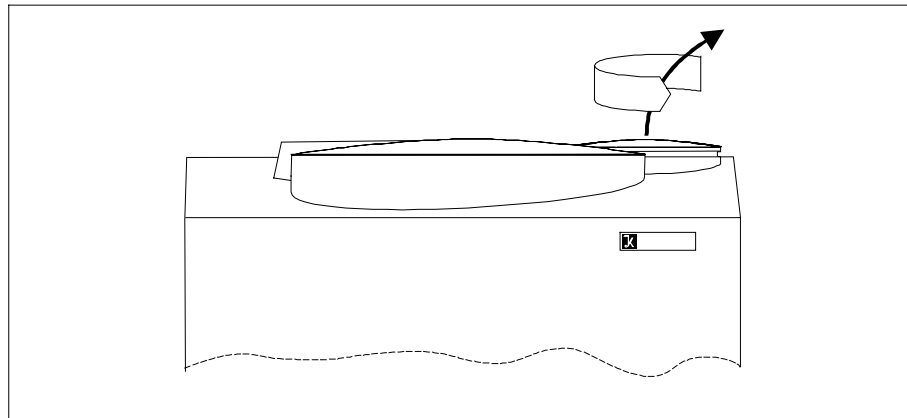
## 8.5 Filling the system circuit

①

The liquid with which the system is filled must be prepared as follows (about 5 liters per measurement cell):

- Fill a clean container with about 2.5 liters of distilled water
- Add 5 ml of Aqua-Pro
- Add the remaining 2.5 liters of distilled water to the container
- Stir the mixture, or close the container and shake

A clean container that can be grasped easily should be used to fill up the system circuit with liquid. Open the cover of the expansion container by rotating and add 1 liter to the expansion container of the measurement cell.



Opening the expansion container



**For the Duo-control, measurement cell 2 should be turned off first for the first filling. Measurement cell 2 should not be turned on and filled up until the procedure for filling up cell 1 has been completely finished.**

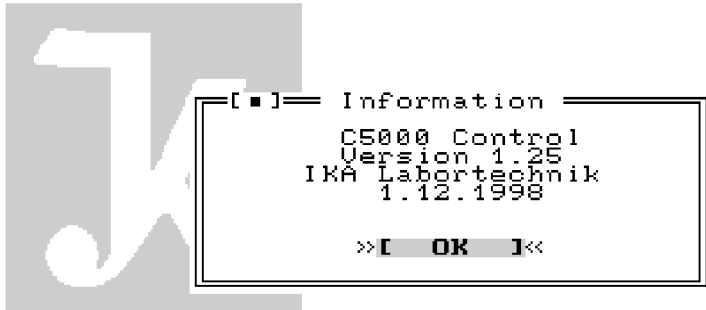
②

Connect the power plug with the power source. Turn on the measurement cell on the power switch (the cooling unit will not be turned on yet at this point).

The system begins to boot up. The cover of the measurement cell opens automatically and the opening screen appears on the display of the control console. You must confirm the opening screen with the *OK* key.



Mode: Adiabatic 10:49:15



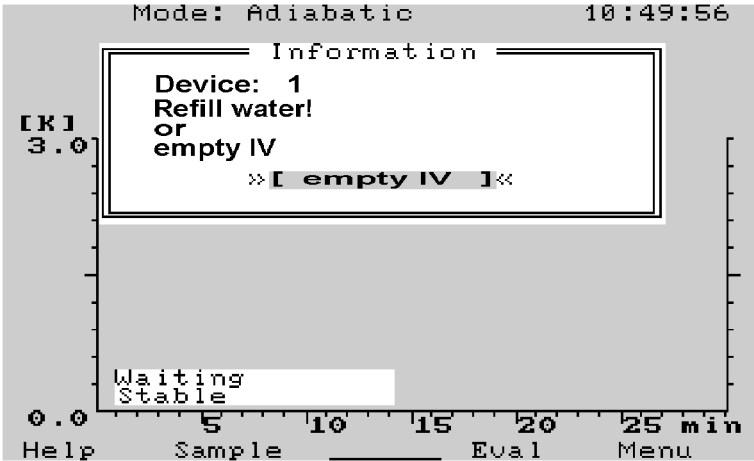
Opening screen Help Sample Eval Menu

Because there is only a small amount of water in the expansion container, the following message now appears:

*Refill with water or empty IV (IV - inner vessel)*



At the same time, an acoustic signal is heard. This message must be ignored at first, and you **must not** confirm it by pressing the OK key.



Water level error message

Now pour in about 1 – 1.5 liters (in any case, enough for the message on the display and the signal to go off) of **the prepared mixture** evenly and slowly. This will turn the pump on automatically and the water will be pumped from the expansion container into the system.

As the water level in the expansion container sinks, the same message appears again:

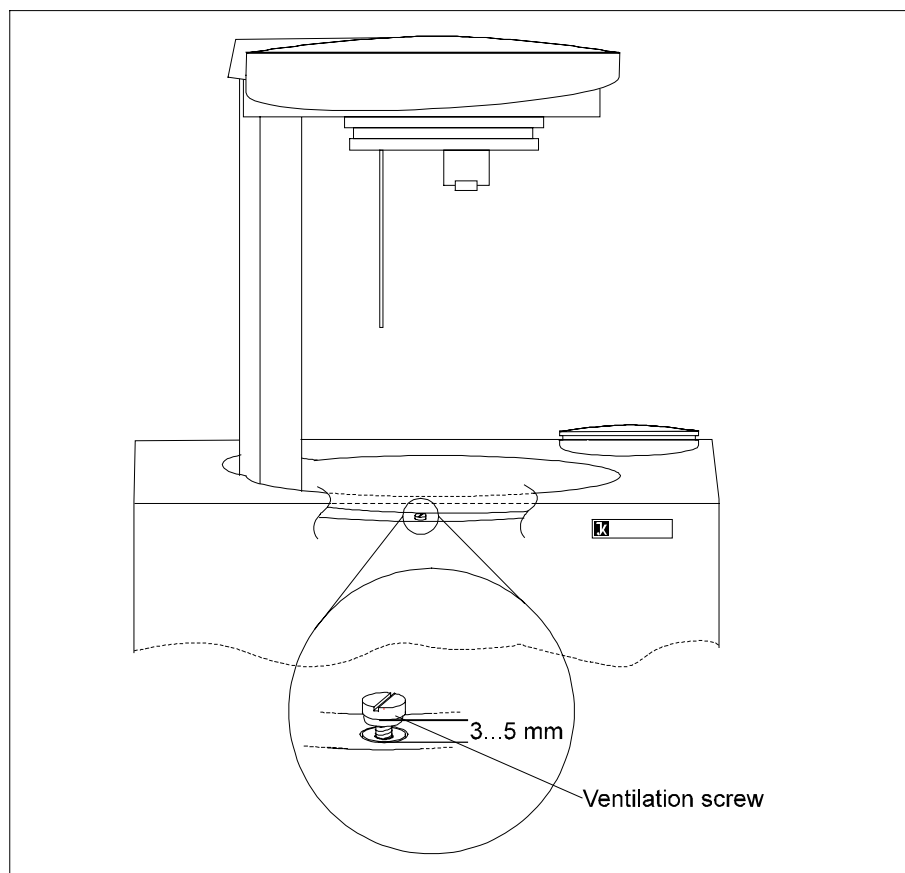
*Refill water or empty IV*



and the acoustic signal is heard. This message must be ignored again, and you **must not confirm it by pressing the OK key.**

③

Now the water system must be vented. Turn the ventilation screw out by about 3 ... 5 mm with a screwdriver (**do not screw the ventilation screw out entirely**). Watch the ventilation screw until water comes out and turn it shut again. There are still pockets of air trapped in the water system, but they should dissipate within the next 2 to 3 minutes through the expansion container. The pumping noises are then reduced significantly.



Venting the cooling water system

④

Continue filling with the prepared liquid until the message on the display goes off. The water capacity in the entire system is about 4.5 liters. Set the cover back on the expansion container with a turning motion. The rest of the mixture will be required later on for operating the unit.



The sieve insert in the filling sleeve of the expansion container must be checked when refilling the system for deposits, etc. Observe the references in this regard in Section 14 “Care and Maintenance”.

During routine operation, liquid is lost by evaporation and by adhering to the decomposition vessel.

During normal operation, if this error message appears on the display:

*Refill water or empty IV*



**at least 50 ml of the mixture should be added to the expansion container. If the message does not disappear, repeat the filling process in increments of 50 ml.**

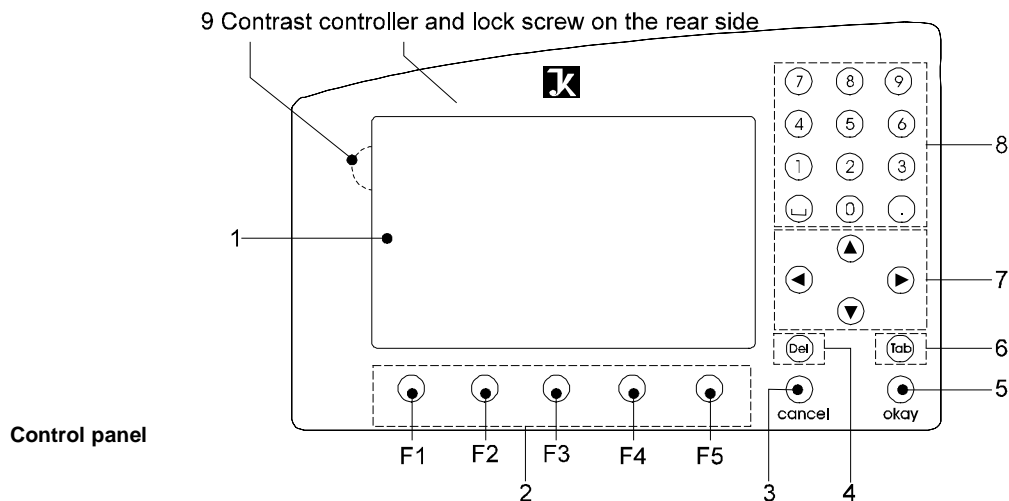
⑤

At this point, the cooling unit is turned on. The system is now ready for operation.

## 8.6 Control and display elements

Before you continue with the next steps in preparing the system for operation, you should become familiar with the display and control console.

The control console is equipped with the following elements:



1. **LC display** for showing system data, experiment data as well as menus and dialog boxes for entering data.
2. **Function keys** The assignment of the function keys depends on the operating state of the system at the moment. *F1* calls up a context-sensitive help system. The footer of the display indicates the current assignment of the function keys.
3. **Cancel key** The *cancel* function is active in the menu and dialog boxes. You can use *cancel* to leave a window without the system accepting any data that may have been entered.
4. **Del key** If you have entered a character sequence inside a dialog box, for example the weight of the combustion sample, you can delete the character immediately to the left of the cursor with the *Del* key. The *Del* key has a second function: outside a dialog box, you can open the menu bar on the upper edge of the screen by pressing the *Del* key.
5. **OK key** You can use the *OK* key to activate menu items and to close or confirm dialog boxes. In addition, *OK* is used to cause the system to accept data that was entered inside a dialog box.
6. **Tab key** *Tab* moves the cursor within a dialog box from one entry box to the next.

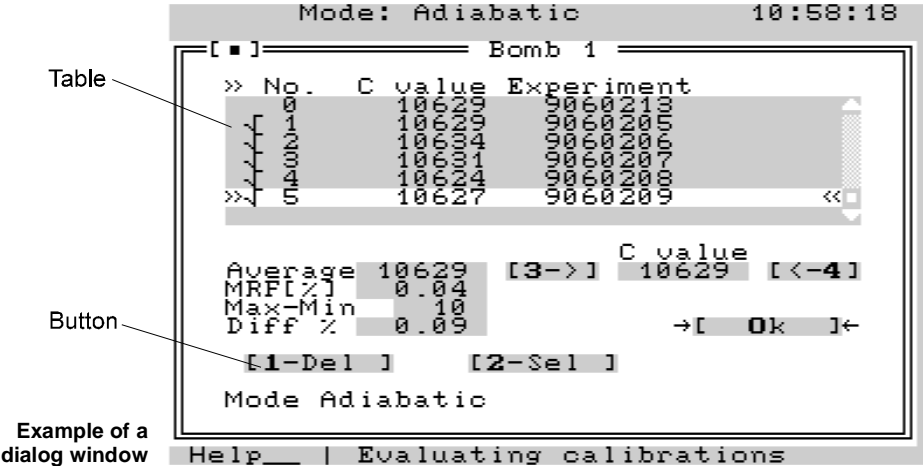


*Tab* is used to move from the display for measurement cell 1 to the display for measurement cell 2 in the duo control system.

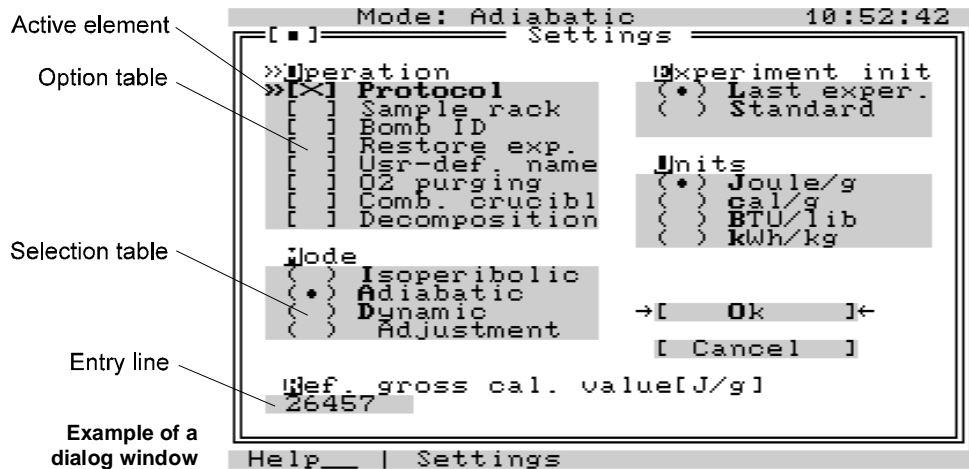
- 7. **Left, right, up and down arrow key** The arrow keys move the cursor within the entry lines, menu windows, tables and protocols.
- 8. **Number block** You can enter numbers, decimal points and blank spaces with these keys. You can open up or close an additional information window for service purposes with the decimal key outside of a dialog box. You can print out the content of this window with the space bar  $\odot$ .
- 9. **Contrast controller** For controlling the contrast of the display.  
**Lock screw** Loosening the lock screw will change the angle of inclination of the display. To lock it, the screw must be screwed in again until it is tight.
- 10. Various dialog elements can be selected within the dialog box in the display.

The following dialog elements are available:

- entry line
- button
- simple table
- selection table
- option table
- display elements (cannot be accessed)



Example of a dialog window



- Active dialog element** All dialog elements are labeled. Active dialog elements are identified by the character `»`. You can cycle through and make each element in turn the active element by repeatedly pressing `TAB`. Only the active dialog element can be accessed (have some function performed on it). The button is an exception to this rule.
- Entry line** Digits and decimal points can be entered in an active entry line. The character that was last entered can be deleted with `DEL`. Some entry lines offer the possibility of selecting letters and additional characters from a displayed table of characters with arrow keys and then bringing them into the entry text with the `.”` key. `TAB` ends entry and activates the next dialog element. `OK` ends entry and closes the window.
- Table** You can select or deselect the lines of an active table (also a selection and option table) with the `Up arrow` and `Down arrow` keys. The possibility for selecting is indicated in a selection table with `(•)`. For an option table, the option in the selected line can be activated (indicated by `[x]`) or deactivated again (indicated by `[ ]`) with the space bar. `TAB` completes work in the table and activates the next dialog element. `OK` ends work in the table and closes the window.
- Active button** An active button is switched with the `OK` key. If a table is active and the button is labeled with a number, it is possible to switch to it directly with the corresponding number key.
- Dialog window** Almost every dialog box has the buttons `OK` and `Cancel`. If the `OK` button is marked with `->` and `<-`, it can also be switched with the `OK` key from an active table or entry line. The result is that the dialog box closes and the entries and settings are taken over. A button labeled with `Cancel` can always be switched with the `CANCEL` key and also results in the window closing, but without the entries and settings being accepted. In no case can the actions previously initiated by other switches be undone.

### 8.7 Turning on the system

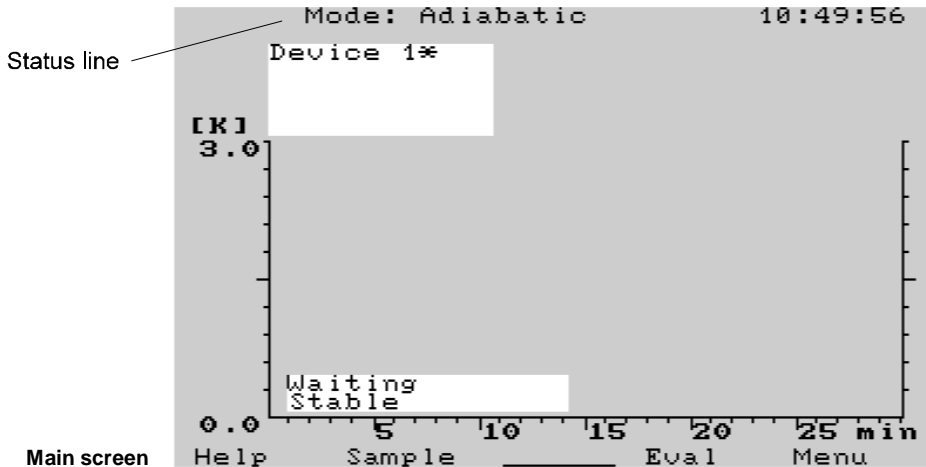
When you turn on the calorimeter system (measurement and cooler), the opening screen first appears (the cover of the measurement cell opens up automatically).

Mode: Adiabatic 10:49:15



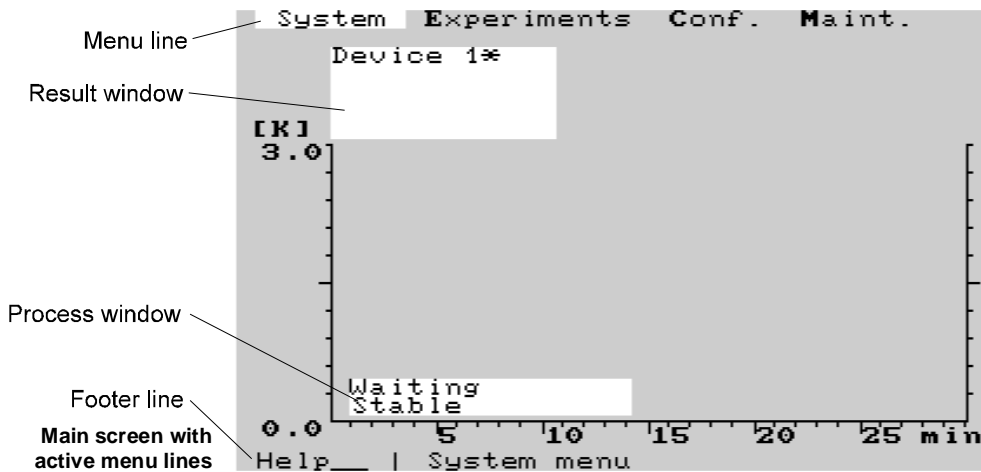
Opening screen Help Sample Eval Menu

In the footer line you can see the current assignment of the function keys. You must confirm the opening screen with the *OK* key to reach the main screen.

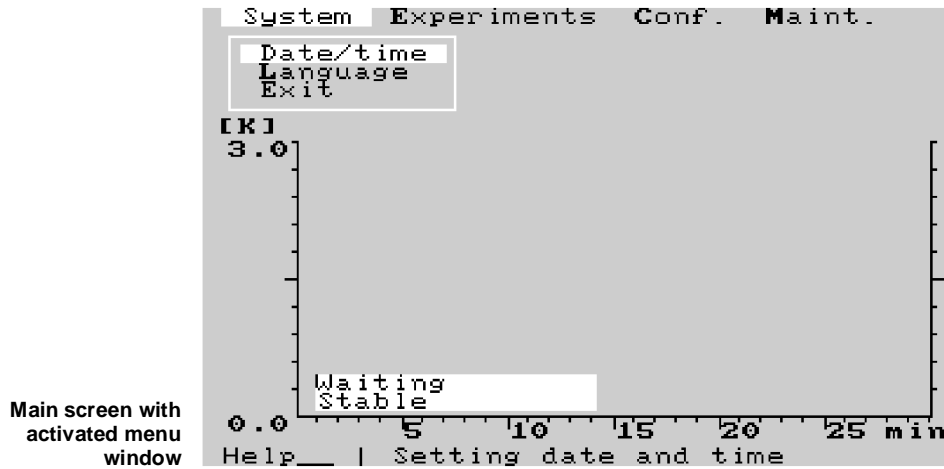


Main screen

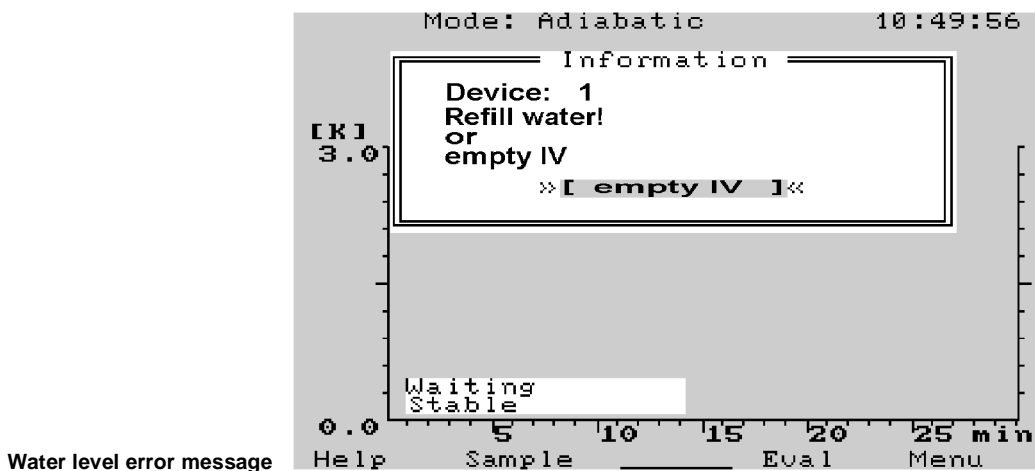
You can reach all menu and dialog windows from the main screen. You can reach a part of them through menu lines that are called with the *Menu* key or the *Del* key (duo-control).



You can move the cursor through menu lines with the arrow keys. You can also open a menu window with *Arrow down* or *OK*, and then a dialog box with *OK*.



If the error message *Refill water or empty IV* (IV = inner vessel) appears while you are confirming the opening screen, check to see the water level of the inner vessel (visual check).





If it should happen to be higher than 1 cm above the bottom of the vessel, then please confirm the error message in the display with the *OK* key. In this case, the remainder of the water is pumped out of the inner vessel into the expansion container.

If the error message is not eliminated in spite of the inner vessel being emptied, you must pour 50 ml of the prepared liquid into the expansion container. The message then disappears. If this quantity alone is not sufficient, then repeat the last step in 50-ml increments.



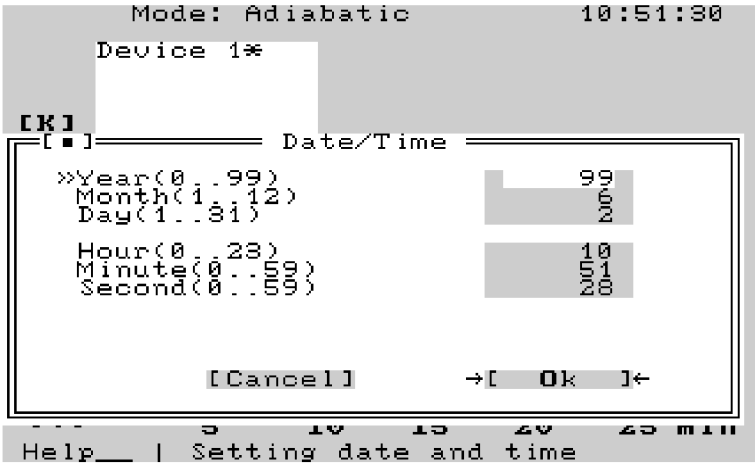
**Before you pour the liquid into the expansion container, however, always check the water level in the inner vessel first. If you should find a residual volume of water there and other additional liquid is added to the expansion container, this could cause the system to overflow the next time the inner vessel is emptied.**

### 8.8 Configuring the system

Some configuration tasks and system settings can now be performed.

#### Checking the date and time of day

- ① Open the *System* menu.
- ② Open the *Date/time* dialog box.



Date/time dialog box

Meaning of the entries:

<i>Year (0...99)</i>	number of the year, for example 97 = 1997, 02 = 2002
<i>Month (1...12)</i>	Calendar month, for example 03 = March
<i>Day (1...31)</i>	Day of the month
<i>Hour (0...23)</i>	Hour entry; 0 = midnight
<i>Minute (0...59)</i>	Minute entry
<i>Second (0...59)</i>	Second entry

③

Compare the entries with the current date and time of day and correct the entries as needed. If you confirm the dialog box with *OK*, the system clock and calendar will accept these values.

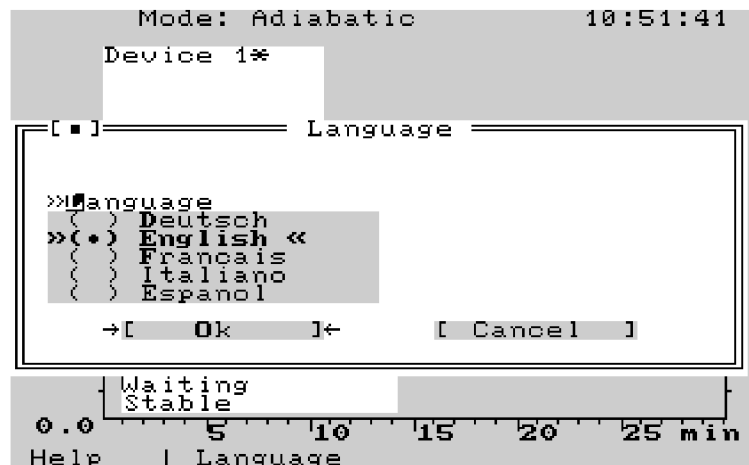
### Selecting the language

①

Open the *System* menu

②

Open the *Language* dialog box. You will see a list of languages in which dialogs can be processed while working with the calorimeter system.



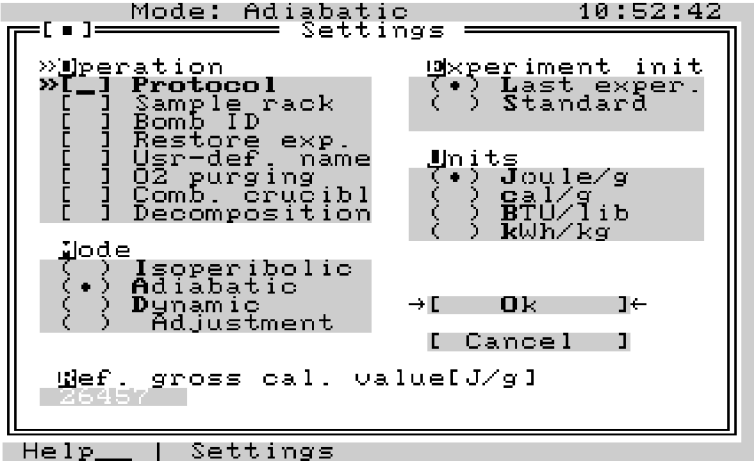
Language dialog box

③

Using the Up / Down arrows, select your language from the list and confirm the selection with *OK*. From now on, text on the screen, text in the help system and print-outs will be in the new language selected.

**System settings**

A few system settings must still be made for the experiment procedure, the method of working, the initialization of the experiment, the reference gross calorific value and the unit of measure for the gross calorific value. To do this, place the cursor in the menu line on *Conf.*, open the menu window and call up the *Settings* dialog box.



Settings dialog box

The window shows the configuration boxes *Operation*, *Operating mode*, *Reference gross calorific value*, *Experiment init.* and *Unit*. You can move the cursor to the next configuration box with *Tab*. To make settings in the *Operation* entry box, you must place the cursor on the desired line with the *Up/Down arrow* and then press the space bar  $\ominus$ . Your entry is confirmed with "x". If you press the space bar again, the "x" will be deleted.

● *Operation* entry box

*[ ] Protocol*

If you check this option, a protocol, or record, will be printed out for each experiment. This also applies to the individual temperature measurement values taken during the measurement.

*[ ] Sample rack*

A sample rack is connected and will be used. The *Sample* dialog box can no longer be called manually; instead placing samples on the sample rack, or removing them from it activates the same dialog box. This ensures secure management of even a large number of samples.

For more information on working with the sample rack, please refer to the C5020 Operating Instructions.

*[ ] Bomb ID*

Decomposition vessels are automatically identified by their coding. There is no manual entry of the code number of decomposition vessel. See Section 9, Coding decomposition vessel.

*[ ] Rest. experim.*

The experiment can be restarted at another time if it is interrupted before the ignition. The experiment parameters are retained. To restart the experiment, the decomposition vessel must be removed from the measurement cell and then reinserted again.

**[ ] User def. name**

Here you can specify whether you will enter the *Sample* name yourself in the *Sample name* entry box and in the *Sample* dialog box, or whether the system will automatically assign the *Sample name*. If you do not select this option, the system assigns experiment numbers in the *Sample name* box.

**[ ] O2 rinsing**

With this option, the decomposition vessel is briefly filled with oxygen, after which the oxygen is released each time, before the actual filling with oxygen. The purpose of this is to remove atmospheric nitrogen.

**[ ] combustible crucible**

If you are using a combustible crucible, the default entry for extraneous energy is 70 J (electrical ignition energy). Otherwise it is 120 J (70 J + 50 J). The additional 50 J are derived from burning the cotton thread.

**[ ] Decomposition**

If the sample is to be subjected to a subsequent decomposition after it has undergone combustion, the decomposition vessel must be depressurized outside of the calorimeter. To do this, a special depressurization station C5030 is connected (there is no automatic depressurization of the decomposition vessel).

- **Operating mode entry box**

In this box, you can select an option for the temperature control of the water jacket in the outer vessel. The following options are available:

- Isoperibolic**      The temperature of the water jacket is regulated to a constant temperature.
- Adiabatic**      The temperature of the water jacket is regulated adjusted to match the temperature of the inner vessel.
- Dynamic**      The combustion experiments are performed according to a quick procedure.
- Adjustment**      Internal parameters of temperature control are determined for this option. The device has been adjusted during the functional test in the factory. It possesses a temperature compensation so that under normal laboratory circumstances no adjustment is required when it is first placed in service. For more detailed information, see Section 14.3, Adjustment.

- **Reference gross calorific value [J/g] entry box**

In most cases, certified benzoic acid is used. The indicated gross calorific value should be entered. If you are working with another reference combustion substance, you must enter the gross calorific value of this combustion substance here yourself.

- **Experiment init. entry box.**

You can use the experiment initialization to specify how the *parameters User and Sample properties* should be set in the *Sample* dialog box, as well as all parameters in the *Experiments* dialog box. These setting options are dis-

cussed again in Section 11, "Determining gross calorific values". The following options are available:

( ) *Last experim.* The system accepts the *User* and *Sample properties* parameters for a new experiment as well as the indicated post-experiment parameters of the last experiment to be evaluated. If the *User def. name* option has been selected, the sample name is also accepted. This must then be edited or reentered to make up for the difference.

( ) *Standard* The post-experiment parameters are set to 0 for a new experiment. The extraneous energy is set to 120 J / 70 J, and the *User* and *Sample properties* boxes in the *Sample* dialog box remain empty.

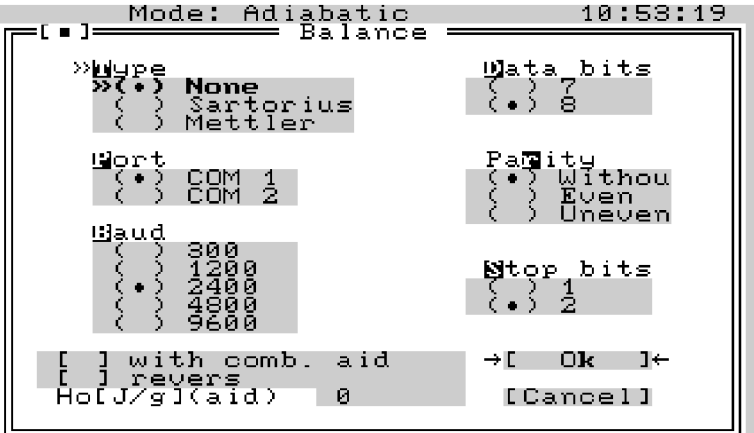
- *Unit entry box*  
The unit of measure for the caloric results is specified here. This refers only to the result protocols! Available for selection:

- ( ) *Joules/g*
- ( ) *cal/g*
- ( ) *BTU/lib*
- ( ) *kWH/kg*

When you click on *OK*, the calorimeter system accepts the settings and closes the dialog box.

**Configuring the scale**

If electronic scale is attached to the system, the scale type must be configured. To do this, open the *Scale* dialog box in the *Conf.* menu box.



Scale dialog box

The window displays the configuration windows *Type*, *Port*, *Baud*, *Data bits*, *Parity*, and *Stop bits*. The parameters you select here must agree with the interface parameters of the attached scale. Please refer to the scale manual for the parameters. To move the cursor to the next configuration box, press *Tab*. *Up/Down arrow* moves the cursor within a configuration box. If you leave a box with *Tab*, the current setting is retained in the box.

You can make the following settings with the configuration boxes:

- *Type* configuration box  
Here you can indicate which scale is connected to the system. Either *no scale* or one of the types indicated are connected.
- *Port* configuration box  
No entry is possible in the *Port* box. The scale is always attached to COM1.
- *Baud* configuration box  
The data transmission rate between the scale and the calorimeter system is adjustable to 300, 1200, 2400, 4800, 9600 and 19200 Bit/s.
- *Data bits* configuration box  
Here you can select whether data will be transferred in 7-bit or 8-bit format.
- *Parity* configuration box  
Indicate whether the transferred data should be accepted *without* a check for parity by the calorimeter system, or whether a check should be performed for *even* or *odd* parity.
- *Stop bits* configuration box  
Select either *1* or *2* stop bits for the data transfer protocol. If you are using a combustion aid or the combustible crucible, it is possible to record the weight of the combustion aid or the combustible crucible by using a special weighing mode and to calculate the extraneous energy resulting from this measurement automatically.
- *With combustion aid* configuration box  
If the option *With combustion aid* is marked, the values of the scale are transferred in the following order:
  1. "Weighed in combustion aid"
  2. "Weighed in combustion aid + weighed in sample"
- *Reverse* configuration box  
If the box *reverse* is marked in addition to the configuration box *With combustion aid*, scale values will be transferred in the following order:
  1. "Weighed in sample"
  2. "Weighed in sample + weighed in combustion aid"After the transfer, the second measured value appears in the "New measurement" dialog box. The calculated value for extraneous energy is already entered there.

- *Gross calorific value of the combustion aid configuration box*  
In combination with the configuration box *With combustion aid*, the gross calorific value of the combustion aid must be entered in this box so that the system can calculate the extraneous energy.

## 9 System calibration

Before it is possible to make precise measurements with the calorimeter system, it must be calibrated. This is done by burning tablets of **certified benzoic acid** (see accessories) with a known gross calorific value. This makes it possible to determine the heat capacity (the C value) of the system based on the amount of heat that is required to raise the temperature of the calorimeter system by 1 degree Kelvin. This value is then used for subsequent determinations of gross calorific values.

The heat capacity is determined by the measurement cell and the decomposition vessel. It has a considerable effect on the precision of measurement, and must especially be determined when the system is first placed in service, after maintenance or repair work, and when parts are replaced.



**If a measurement cell is being operated with several decomposition vessels, the heat capacity of the system must be determined through calibration for each individual decomposition vessel. A decomposition vessel should only be used in the measurement cell for which it has also been calibrated.**

**The calorimeter system must be calibrated in each operating mode (adiabatic, isoperibolic and dynamic) in which measurements will later be made. Please observe the applicable standards in this regard.**

Calibration must take place under the same conditions as will be found during subsequent experiments. If measured quantities of substances (for example distilled water or solutions) will be used in the decomposition vessel, exactly the same quantity of these substances should be used during calibration.

### Calibration notes

- In order to achieve precise results, you should take care that the combustion not exceed an increase in temperature of 4 K. This applies as a rule of thumb if no benzoic acid is being used during the calibration.
- It should be mentioned here in advance that when determining gross calorific values, the increase in temperature must be roughly the same as for the calibration (for example 4 tablets – approximately 1 g of benzoic acid  $\approx$  2.6 K)

### Coding

When working with the calorimeter system, a maximum of 4 decomposition vessels can be used. The maximum for the duo-control system is 2 decomposition vessels per measurement cell. This is possible by coding the decomposition vessels from 1 to 4. The system recognizes which decomposition vessel an experiment is being performed with and assigns its calibration parameters to it.

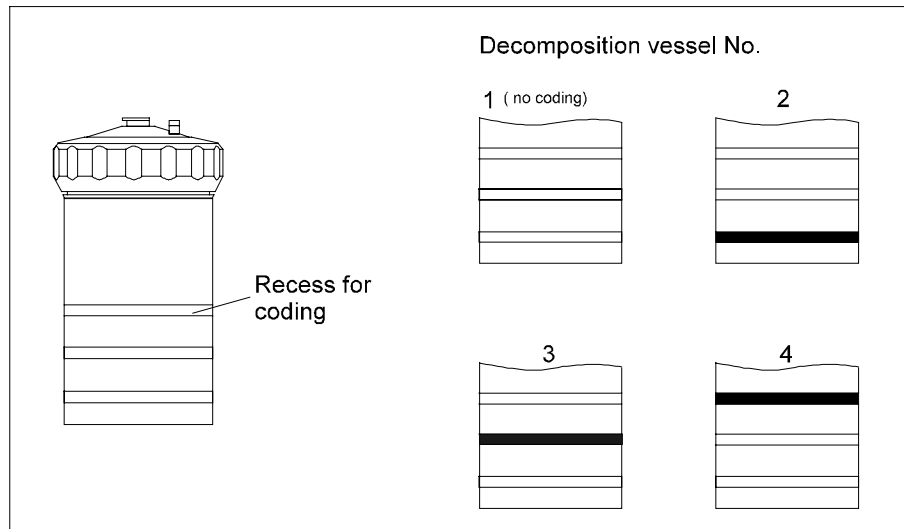


**Each decomposition vessel must be coded before it is used for the first time.**

To do this, attach the black coding rings into the recesses on the decomposition vessel provided for this purpose.

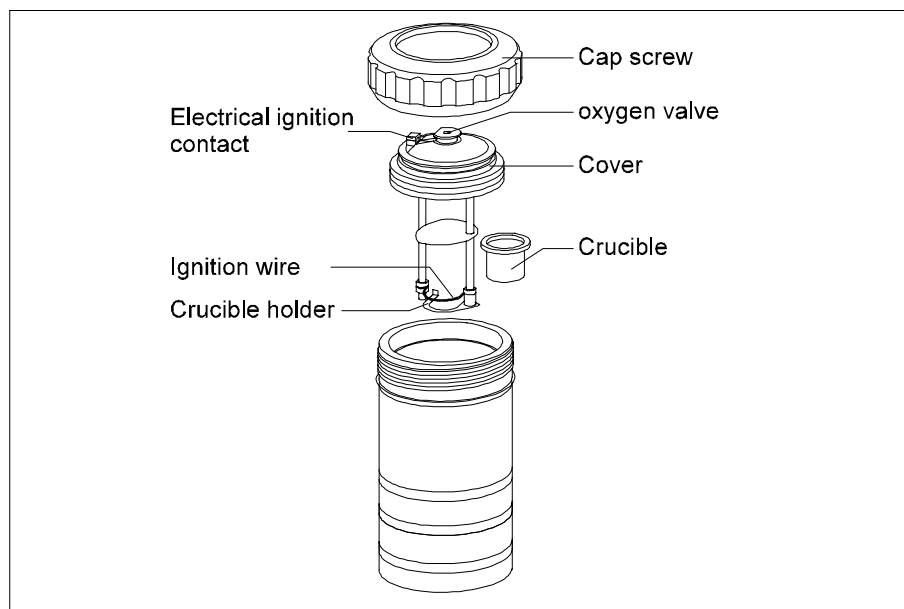


Coding of the decomposition vessel



### 9.1 Charging the decomposition vessel with the calibration substance

Individual parts of the decomposition vessel



Now you can charge the decomposition vessel with the calibration substance, for example certified benzoic acid.



**If more than one decomposition vessel is being used, the respective individual parts must not be exchanged between the various decomposition vessels (see the engraving on the individual parts).**

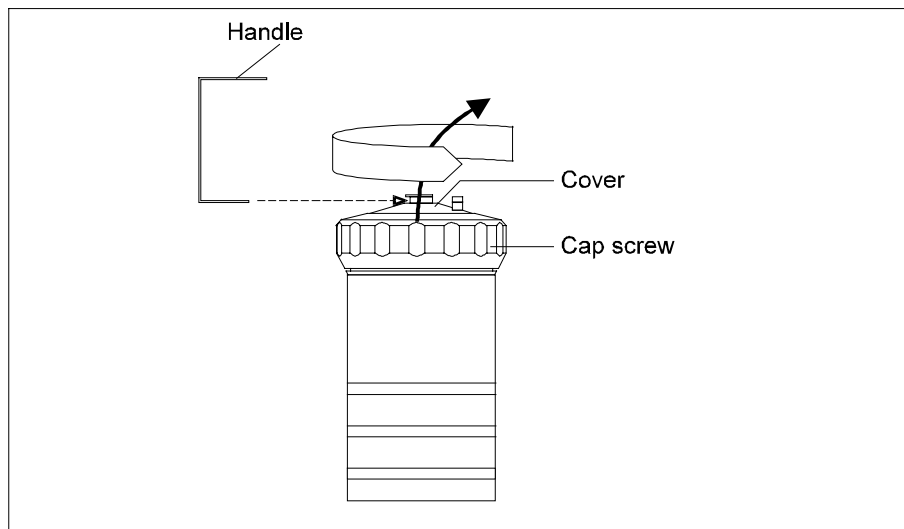
On cleaning the decomposition vessel, see Section 10.4

To prepare the coded decomposition vessel, follow the steps listed below:

①

Screw off the cap screw and take off the cover with the aid of the handle.

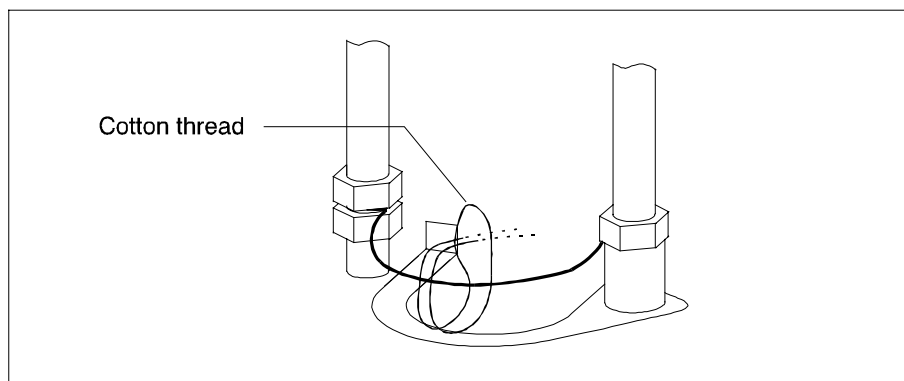
Opening the decomposition vessel



②

Secure a cotton thread with a loop in it on the middle of the ignition wire.

Assembling the cotton thread



③

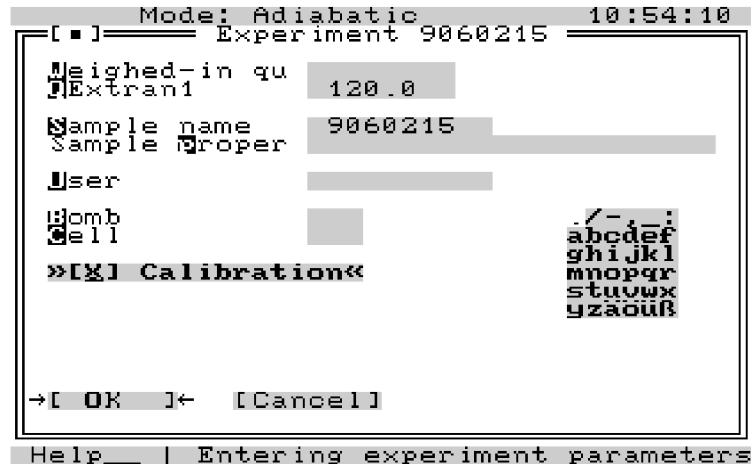
Weigh in the calibration substance (about 1 g, 4 tables of certified benzoic acid; see accessories), accurate to within 0.1 mg, and place in the crucible.



**In general, you must choose the weighed in quantity so that the increase in temperature does not exceed 4 K (maximum energy input: 40,000 J). Otherwise, the decomposition vessel may suffer damage. Bursting decomposition vessels can cause danger to life and limb. When working with unknown substances, very small amounts must be chosen at first to weigh in, in order to determine the energy potential.**

④

Make certain that the desired operating mode (isoperibolic, adiabatic, or dynamic) is set (see Section 8.8 "Configuring the system"). Open the *Sample* dialog window to enter parameters. If a sample rack is active, parameter input is opened automatically by setting or removing a crucible (see also C5020 Operating Instructions).



Sample dialog box

Enter the weight of the combustion sample in the *Weighed in quantity* box. If an electronic scale is connected to the calorimeter system, the weight can be accepted automatically. Depending on the type of scale, the *Sample* dialog window can be opened either through the sample key of the calorimeter or through the *Print/transfer key* of the scale. The space bar can be used to accept the scale value again.

You can move the cursor to the following entry boxes with the *Tab* key. The meanings of the other entry boxes are as follows:

**QExtran1** The correction value for heat energy that is not generated from the fuel sample. A value of 120 J appears here by default. This corresponds to the heat energy introduced from the electrical ignition and the combustion heat of the cotton thread that can be obtained from IKA. You can change the pre-set value for *QExtran1* at any time.

If the weight of the combustion aid is transferred in *With combustion aid* mode from electronic scale, the resulting extraneous energy calculated from the weight appears in the *QExtran1* box. Even without a scale, the gross calorific value of the combustion aid can be taken into consideration automatically. In this case, you should enter the weighed in quantity of the combustion aid in the *New measurement* dialog box under *QExtran* and then press the ↓ arrow. *QExtran* is calculated according to the formula

$$QExtran = (\text{Weighed in quantity of combustion aid} \times \text{calorific value of combustion aid}) + 120 \text{ J}$$

and is entered in the *QExtran1* box. If a value > 120 J is entered, it is assumed that the value already represents all extraneous energy (including the standard 120 J). In this case, there is no further conversion based on the formula given above.

**Note:** the 120 J for the ignition thread and the energy of the electrical ignition are already taken into consideration in all automatic calculations.

**Sample name** The software automatically assigns a sample number for each measurement of the format **yymmddnn**, where **y** is the year, **mm** is the month, **dd** the day and **nn** a running number. It is easy with sample numbers formed in this manner to select and work with specific groups of measurements from the library.

If you select the option *User def. name* under *Menu, Configuration, Settings*, you can assign your own numbers or names for measurements (automatic numbering continues to run in the background, but is no longer taken into consideration).

If you have selected the option *Last* in addition under *Menu, Configuration, Settings, Experiment init.*, the number of the last experiment appears as a suggestion for the current measurement. If you do not edit this suggestion, the sample number will be the same for all measurements!

Example of a *Sample name* = 6052401

1 Number of the year, 6 = 1996  
 05 Month, 0 ... 12, 05 = May  
 24 Day of the month, here May 24<sup>th</sup>  
 01 The running experiment number

*Sample properties* Any additional information on the sample. You can select letters and characters from the character table with the *arrow keys*. With the *decimal point* key, the system accepts the selected characters into the entry field (max. 40 characters).

*User* The name of the user (up to 8 characters). Entry as for *Sample properties*.

[ ] *Calibration* Mark this box with space bar for the system to use the experiment for calibration.

OK causes the system to accept entries in the dialog box.

⑤

The message *Bomb* ↓ appears from then on at the bottom of the screen. This means that from now on, the decomposition vessel can be suspended in the measurement cell cover.

⑥

If additional amounts of distilled water, solutions, etc. will be used in subsequent combustion experiments, the same quantity of the same substance should be included now in the decomposition vessel. The system should be calibrated in the same state in which you intend to be working later. If the operating mode changes (with/without an amount of water) the calibration should also be repeated.



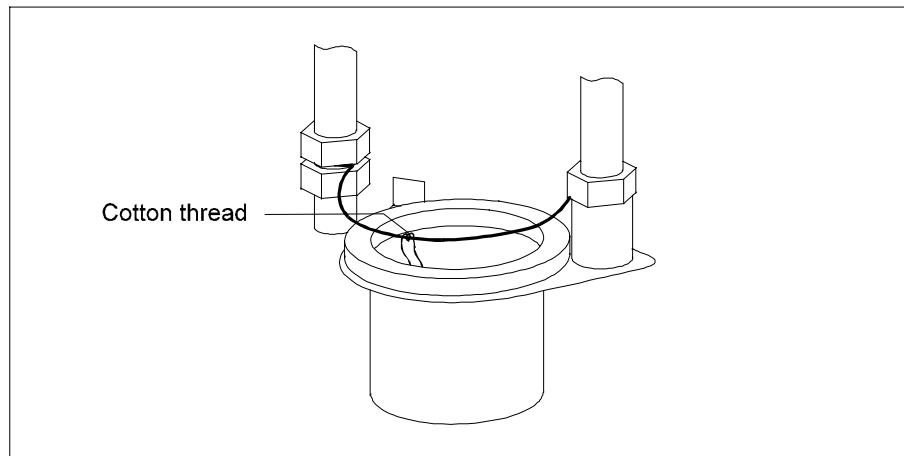
**To increase the life of parts subject to wear and tear (O rings, seals, etc.), we recommend in general that you always work with a measured amount of water in the system.**

⑦

Place the crucible in the crucible holder.

⑧

Align the cotton thread with a pair of tweezers so that it is suspended into the crucible and touches the sample. This will ensure that the burning thread will ignite the sample during the ignition process.



Aligning the cotton thread

⑨

Place the cover on the decomposition vessel and screw on the cap screw.

## 9.2 Calibration

①

Guide the decomposition vessel carefully until it interlocks with the filling head of the open measurement cover (No. 1 in the following illustration)



**Always hold the decomposition vessel by the top of the cap screw!**

The decomposition vessel fits into a defined position because of a depression of 0.8 mm in the center of the filling head (No. 2 in the following illustration). A spring element then contacts the electrical ignition contact on the decomposition vessel.

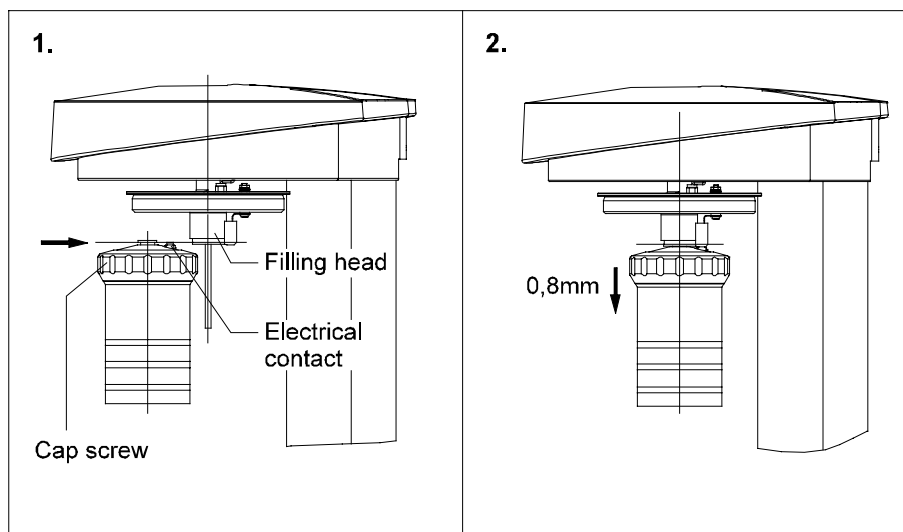


**The decomposition vessel is now suspended vertically in the receiving piece (visual check to make certain!).**

As soon as the electrical circuit in the decomposition vessel is closed through the ignition wire, the calorimeter goes into ready mode. The message *Bomb* ↓ changes to a display of the function key assignment *Start*. If the function key assignment *Start* does not appear, please check the ignition wire of the decomposition vessel. Take note of whether the status is shown in a stable state on the display.

After each measurement, the measurement cell is adjusted so that normal starting conditions are present for the next measurement. During this phase (about 3 to 5 minutes), “Unstable” is displayed in the process window of the measurement cell. As long as this message is displayed, no measurements are possible in adiabatic or isoperibolic operating modes. Experiments in dynamic mode can also be started during the adjustment phase.

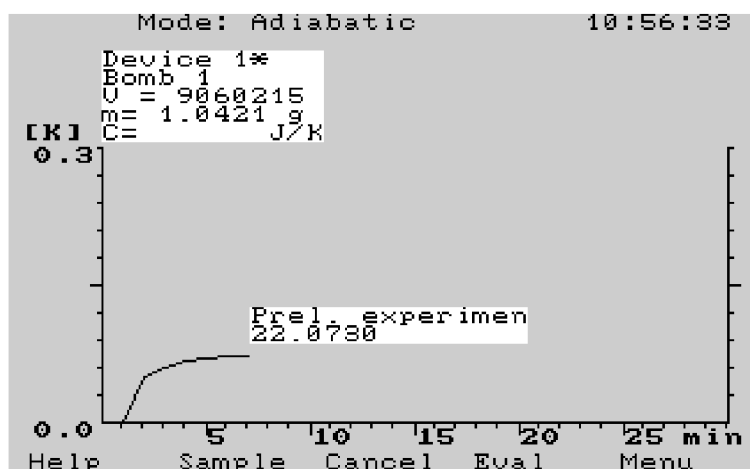
Suspending the decomposition vessel into the filling head of the measurement cell cover



②

Activate *Start*. The measurement cell cover closes. The decomposition vessel is then filled with oxygen. Next, the inner vessel is filled with water. As soon as the system begins with the experiment, the display shows a graph of the change over time in the temperature of the inner vessel.

Change over time in the temperature of the inner vessel during a calibration



③

If necessary:

You can interrupt the experiment at any time with *Cancel*. For the process, see item 5.

④

For systems **with two measurement cells**:

You can now perform steps 1 – 9 from Section 9.1 and steps 1 – 3, from the same section with the second measurement cell.

⑤

When the measurement is complete, the measurement cell cover opens and pressure is released from the decomposition vessel. At the same time, the inner vessel is emptied. After that, the cover opens up completely. As soon as the message *Bomb* ↑ appears in the bottom line, you can remove the decomposition vessel.

⑥

Open the decomposition vessel and check the crucible for any signs of incomplete combustion. If combustion was not complete, the results of the experiment must not be used for calibration. The experiment must be repeated.

⑦

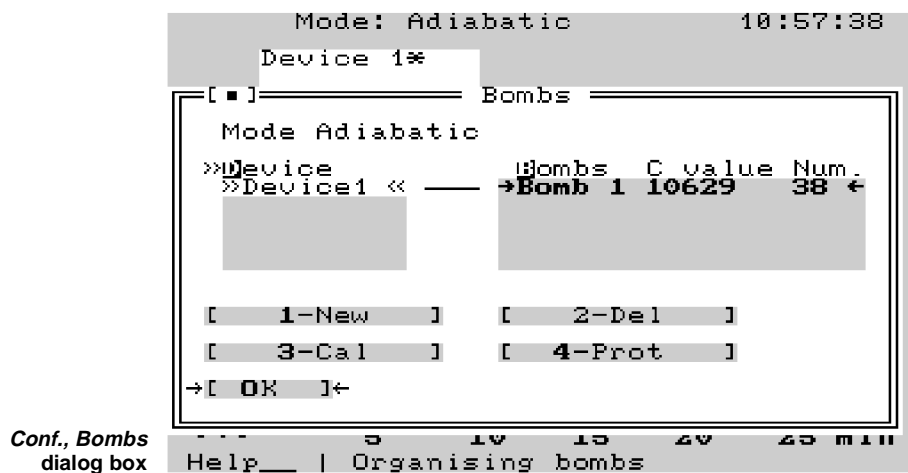
Clean the decomposition vessel as described in Section 10.4 (or the Operating Instructions for decomposition vessel C5010/C5012) and prepare the next experiment.

⑧

Perform a number of calibration experiments for each decomposition vessel as described in Section 9.1 “Charging the decomposition vessel with the calibration substance” and Section 9.2 “Calibration”, steps 1 – 7. For the number of calibrations required, refer to standard in use. For example, DIN 51900 recommends at least 5 calibrations.

⑨

After the last calibration: activate *Menu*, open the *Conf.* menu window and then open the *Bombs* dialog box.



⑩

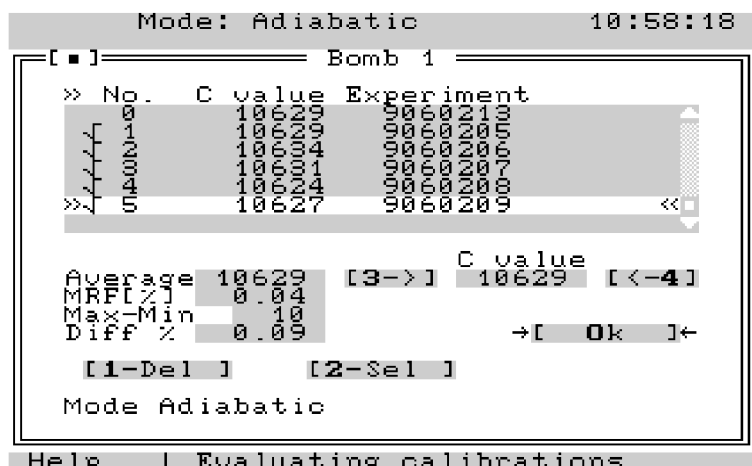
Using *Tab* and *Up arrow* / *Down arrow*, place the cursor on the number of the decomposition vessel with which the calibration experiments have just been performed.

For systems with **two measurement cells**:

Using *Tab* and *Up arrow* / *Down arrow*, place the cursor on the number of the measurement cell and then on the decomposition vessel with which the calibration experiments have just been performed.

①①

Open the *3-Cal* dialog box.



Calib. dialog box

The calibration experiments are listed in the dialog box. The columns in the experiment list have the following meanings:

<i>No.</i>	Running number of the calibration experiments
<i>C value</i>	The heat capacity of the calorimeter system determined with the experiment in question
<i>Experiment</i>	The <i>sample name</i> of the experiment in question

①②

Place the cursor on *2-Sel* with *Tab* and confirm with *OK*, or press the *2* key. You have now selected the test for calibration. The test is marked with "√" on the display

①③

Using the *Tab* key and the *Down arrow* key, place the cursor on the next experiment and activate *2-Sel*. With this, you have selected the next experiment for calibration. The average value of the selected experiments, the average, relative error as a percentage, as well as the scattering range (max-min) absolutely and as a percentage are displayed in the corresponding boxes.

<i>Average</i>	Calculated average value
<i>MRF [%]</i>	Average, relative error
<i>Max-min</i>	Scattering range
<i>Diff %</i>	The scattering range by percentage in reference to the average value

①④

Repeat step 13 for all values that are to be selected. *Average* then displays the average C value of these experiments.

①⑤

The following criteria apply to evaluating successful calibrations:

<i>MRF [%]</i>	Average, relative error, < 0.2% according to ISO 1928
<i>Diff. [%]</i>	Scattering range by percentage, < 0.4% according to DIN 51900



Depending on the standard used, other criteria may be of consequence. Normal requirements for accuracy of the calorimeter are however fulfilled with the values given above.

① ⑥

Using *Tab*, place the cursor on the button [*3→*] and confirm with *OK* or press *3*. This causes the average value from the selected calibration experiments to be assigned to the calorimeter system as the system heat capacity, or *C* value. If you place the cursor on [*← 4*] and confirm with *OK*, you can enter the system heat capacity into the *C* value box.

① ⑦

Place the cursor on the experiments that were not used for calculating the average value and delete with *1-Del*.

① ⑧

Exit the dialog box with *OK*. This ends the system calibration; you can now continue with determining gross calorific values.

## 10 Determining gross calorific values



**It is absolutely required to use a high-pressure decomposition vessel to hold fuel samples in experiments on fuel samples capable of exploding! Decomposition vessels C5010 and C5012 are not permitted for this purpose. The individual parts, and in particular the threading of the decomposition vessel must be checked regularly for wear and corrosion. Note in this regard Operating Instructions C5010 or C5012.**

The C 5000 calorimeter system is a precision measuring instrument for routine determinations of gross calorific values for solid and liquid substances. Exact measurements are only possible, however, if the individual steps of the experiment are performed with great care. The method of proceeding as it is described in the following sections must be adhered to precisely.



**If more than one decomposition vessel is being used, the respective individual parts must not be exchanged between the various decomposition vessels (see the engraving on the individual parts).**

### 10.1 Notes on the sample

#### Solid substances

A few points must be observed in reference to the substances to undergo combustion. Normally, solid combustion substances can be burned directly in powder form. Substances that burn rapidly (for example benzoic acid) must not be burned in loose form.



**These substances tend to spark, and complete combustion could therefore no longer be guaranteed. In addition, the inner wall of decomposition vessel could be damaged. Such substances must be pressed into tablets before combustion.**

The IKA tablet press C 21 is especially suitable for this purpose.

#### Liquid substances

Most liquid substances can be weighed directly into the crucible. Liquid substances with turbidity or water that will settle out must be dried or homogenized before being weighed in. The water content of these samples must be determined.

#### Highly volatile substances

For highly volatile substances, a gelatin capsule or acetobutyrate capsules (see accessories) are used. The gross calorific value of the capsules must be known so that it can be taken into account in the resulting combustion heat as extraneous energy.

#### Combustion aid

The capsules described above, or combustion bags made of polyethylene (see accessories) can also be used as combustion aids for substances with low inflammability or low-calorific substances. The combustible crucible C14 can also be used. Before the capsules or the combustion bags can be filled with the substance to be determined, they must be weighed to be able to calculate (from the weight and the gross calorific value) the additional extraneous energy (see scale mode *With combustion aid*). This energy must be taken into consideration for *QExtran1*. The amount of combustion aid used should be as little as possible.

## 10.2 Acid correction

### Acid formation, heat of solution

Almost all substances that will need to be analyzed contain sulfur and nitrogen. Sulfur and nitrogen are reduced under the pressures and temperatures prevailing in the decomposition vessel to  $\text{SO}_2$ ,  $\text{SO}_3$  and  $\text{NO}_x$ . In combination with the resulting water of combustion, sulfuric acid, nitric acid and heat of solution are generated. This resulting heat of solution is taken into account as specified in DIN 51900 when calculating the gross calorific value. In order to record and determine all resulting acids quantitatively, 5 ml of distilled water can be placed in the decomposition vessel before the experiment.



**The calibration of the device must have been carried out with the same amount of water placed in the decomposition vessel!**

After the combustion, this water must be collected and the decomposition vessel must be thoroughly rinsed with distilled water. The rinsing water and the water that was present are combined and titrated for their acid content (see DIN 51900). If the sulfur content of the combustion material and the nitric acid correction are already known, the water does not need to be analyzed.



**To increase the life of parts subject to wear and tear (O rings, seals, etc.), we recommend in general that you always work with a measured amount of water in the system.**

### Substances with high halogen content

Decomposition vessel C5012 must be used for substances with high halogen content.

## 10.3 Procedure for determining gross calorific value

After the system has been switched on and you have acknowledged the opening screen with *OK*, it requires about 30 minutes until the stable temperature conditions are prevalent in the measurement cell.

Before a measurement is started, the system must have previously been calibrated as described in Section 9 "Calibration".



The decomposition vessel must be clean and dry. See item 10.4. If necessary, a measured amount of distilled water or a solution must be placed in the decomposition vessel. Substances with low inflammability are weighed into the crucible with a combustion aid. The heat of combustion from the combustion aid must be known. Note in this regard Section 10.1 "Notes on the sample". Prepare the decomposition vessel as described in Section 9.1 "Charging the decomposition vessel with the calibration substance". Instead of the calibration substance, the decomposition vessel is charged with a representative sample of the substance to be examined.



In general, you must choose the weighed in quantity so that the increase in temperature does not exceed 4 K (maximum energy input: 40,000 J). Otherwise, the decomposition vessel may suffer damage. Bursting decomposition vessels can cause danger to life and limb. When working with unknown substances, very small amounts must be chosen at first to weigh in, in order to determine the energy potential.



The reproducibility of the results depends to a significant extent on whether the increase in temperature (the energy input) of the decomposition vessel during the combustion experiment comes close the value that was obtained during the calibration. If necessary, the optimal sample quantity must be determined through trial and error.

If distilled water or solutions are used during the combustion experiment, the calibration must previously have been carried with the same amounts of distilled water or solutions.

If you are using a combustion aid, you must add the energy from the combustion aid and enter it into the energy entry in the *Sample* dialog box in the *QExtran1* box, or else use the appropriate scale mode *With combustion aid*.

If a scale is used in the *With combustion aid* mode and the weight of the combustion aid is previously transferred, the extraneous energy calculated from the weight appears in this box.

## ②

Suspend the decomposition vessel into the open measurement cell cover until it reaches the stopper. The message *Bomb* ↓ on the bottom line of the screen changes to a display of the function key assignment *Start*. If the function key assignment *Start* does not appear, please check the ignition wire of the decomposition vessel. Take note of whether the status is shown in a *Stable* state on the display.

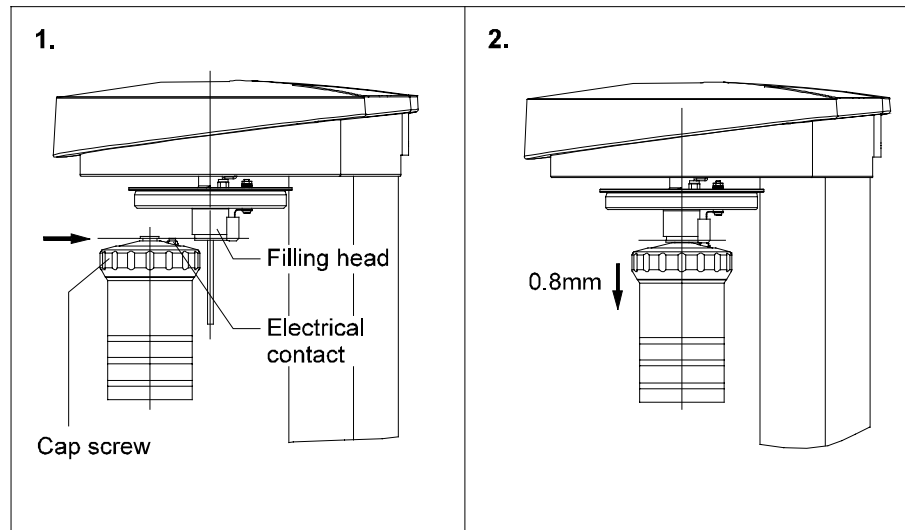
After each measurement, the measurement cell is adjusted so that normal starting conditions are present for the next measurement. During this phase (about 3 to 5 minutes), “Unstable!!” is displayed in the process window of the measurement cell. As long as this message is displayed, no measurements are possible in adiabatic or isoperibolic operating modes. Experiments in dynamic mode can also be started during the adjustment phase.

In addition to the adjustment requirements, the following conditions must be fulfilled in order to start a measurement:

- A measurement must be prepared
- The maintenance function must not have been activated
- The decomposition vessel must be removed and placed back in again
- The ignition contact and the ignition wire of the decomposition vessel being used must be in order.

Not until this point is the start button for the measurement cell enabled, i.e., “Start” appears on it.

Suspending the decomposition vessel into the filling head of the measurement cell cover



③

Activate *Start*. The measurement cell cover closes. The decomposition vessel is then filled with oxygen. Next, the inner vessel is filled with water. As soon as the system begins with the experiment, the display shows a graph of the change over time in the temperature of the inner vessel.

#### System

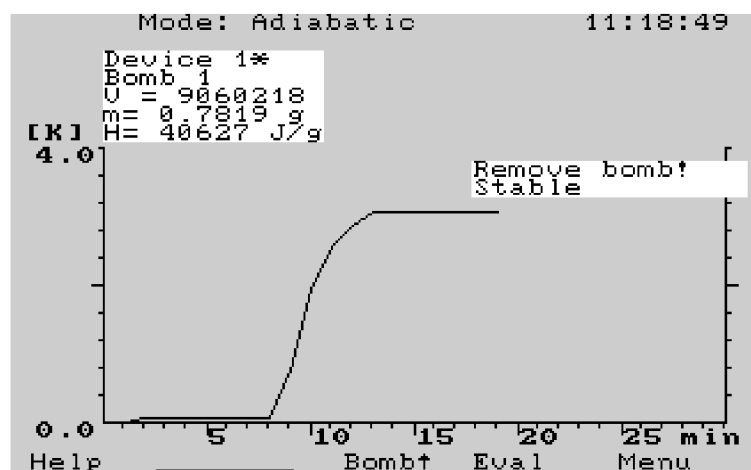
For a system with two measurement cells (duo-control) steps 1 – 2 are now possible with the second measurement cell. In other words, the second measurement can be started while the measurement in the first measurement cell is still running.

④

The sample is ignited and the change in temperature of the inner vessel over time is recorded.

After the end of the experiment, the system displays the results of the experiment

Change in the temperature over time with experiment results: weight of the fuel sample 0.7819 g, gross calorific value 40.627 J/g



⑤

The decomposition vessel is vented and the measurement cell cover opens.

⑥

#### User

As soon as the message *Bomb* ↑ appears, remove the decomposition vessel and open it.

⑦

Check the crucible for combustion residue. Both the cotton thread and the fuel sample must have been burned completely. If there are any signs of incomplete combustion, the experiment must be repeated.

## 10.4 Cleaning the decomposition vessel



**If there is any reason to suspect that the combustion sample or the combustion residue could be hazardous to health, you should wear protective clothing when handling and working with these materials (for example protective gloves, gas mask). Experiment residues that are hazardous to health or which are environmentally hazardous must be disposed of with special waste. We make explicit reference to the applicable regulations.**

It is of fundamental importance for the decomposition vessel to be clean and dry. Contamination can change the heat capacity of the decomposition vessel and result in imprecise measurement results. After each combustion experiment, the inner walls of the vessel, the inner fittings (supports, electrodes, etc.) and the combustion crucible (inside and outside) must be thoroughly cleaned.

### Inner walls of the vessel

In most cases, condensation must simply be removed from the inner walls of the vessel and the inner fittings. It is sufficient to carefully wipe off the parts with an absorbent cloth that will not leave lint.

If the decomposition vessel cannot be cleaned with the procedure described (for example because of burned or corroded spots), you should contact the service location.

### Crucible

The combustion residue in the crucible, for example soot or ash, should also be carefully wiped off with an absorbent cloth that will not leave lint.

## 10.5 Turning off the system

If you want to turn off the calorimeter system, open the *System* menu and call *Exit*. For a duo-control system, measurement cell No. 2 can be turned off separately.



**No decomposition vessel must be suspended in the measurement cell cover.**

If you are working with a duo-control system, you must activate the display for measurement cell No. 1 with *Tab. Exit* then turns off the entire system. If the display for measurement cell No. 2 is active, only measurement cell No. 2 is turned off.



**Turn off the unit only with the *Exit* item in the *System* menu, and not with the power switch (data will be lost!).**

Once the system is turned off, a message to that effect appears on the display. The message asks you to turn off the power switch for the calorimeter and the cooling unit.

# 11 Evaluating experiments

After the determinations of gross calorific value have been completed, you can evaluate the results. In addition to an overview of the experiments, the calorimeter system offers you the possibility of post-processing results and converting them to other references states. You can also print out or delete experiment results. You will find these functions in the menu items *Evaluation* and *Library* of the *Experiments* menu box.

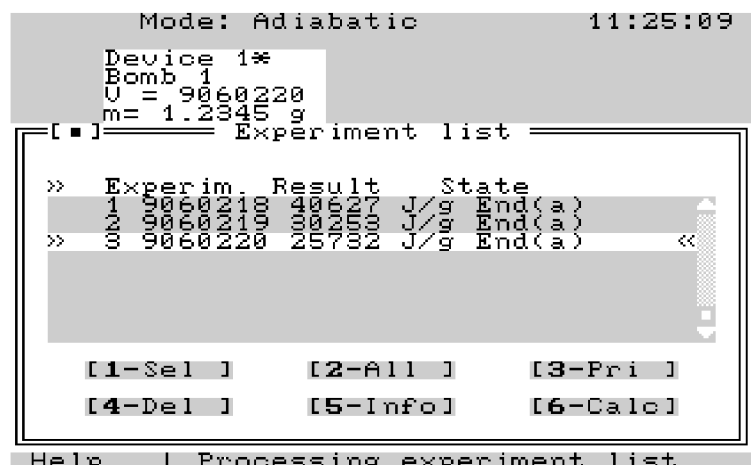
## 11.1 Post-processing experiments

The calorimeter system assigns the stored experiments in two groups, "Daily experiments" and "Library". The daily experiments are those that have been performed since the system was turned on. The library is long-term storage.

### Post-processing daily experiments

①

Activate *Eval*. The "Experiment list" dialog box appears.



②

A list of the daily experiments appears. The meanings of the columns are as follow:

*Experiment* The sample name and description of the combustion sample

*Result* Gross calorific value or C value that was determined during this experiment

*Status* *End*  
The experiment was completed with a result.  
The operating mode is displayed in brackets:  
a (adiabatic), i (isoperibolic), d (dynamic), A (adjustment)

*can*

The experiment was cancelled.

*+cal*

The experiment was performed for calibration purposes.

*+sim*

The experiment was a simulation.

*eval*

The experiment has been evaluated.

*wait*

The fuel sample is in the crucible and all parameters have been entered. The experiment can be started.

*prep.*

The crucible is in the sample rack with a fuel sample.

*run*

The experiment is currently being performed in the measurement cell.

The buttons have the following functions:

- |               |   |
|---------------|---|
| <i>1-Sel</i>  | Marks an experiment in the list<br>exception: calibration.  |
| <i>2-All</i>  | Marks all experiments in the list.  |
| <i>3-Pri</i>  | Prints the experiment list.   |
| <i>4-Del</i>  | Deletes experiments that have been previously selected with <i>1-Sel</i><br>exception: <i>Calib.</i> ; <i>Prep.</i> |
| <i>5-Info</i> | Opens an information window with the experiment parameters.   |
| <i>6-Ber</i>  | Opens a dialog box to convert the results of the experiment into various reference states.                          |

Using the *Up arrow/Down arrow* keys, you can select the experiment from the list that you would like to post-process. Then you can move the cursor with *Tab* from the list box to the buttons.

You can activate the buttons either by placing the cursor on them with *Tab* and pressing *OK* or by pressing the corresponding button number on the numeric keypad.

### ③

Place the cursor on the experiment that has just been completed and activate the *5-Info* button. An information window appears with the results of the experiment.



```

Mode: Adiabatic 11:26:12
Formular i=[↑]
02.06.1999 11:26
MEASURING PROTOCOL 9060220
=====
Cell: 1 Bomb No. 1
Sample : 9060220
User :
Sample properties :
Date of experim. : 02.06.1999
Start time of experim. : 11:23
Mode : Adiabatic
State : End
Sample mass : 1.2345 g
Temperature increase : 0.0000 K
Extraneous energy : 120.00 J
C Value : 10629 J/K
Ho (an) : 25732 J/g
Help_ | Print:SPACE Close:OKAY

```

Information window  
experiment results

### Post-processing experiments from the library

①

Open the Library dialog box in the Experiments menu box. The header indicates the number of experiments still available in memory.

```

987 Experiments free 11:29:38
Device 1*
Bomb 1
U = 9060220
m = 1.2345 g
Experiment library
>>Search mask
-
[ ] Add
[Cancel] → [ Ok ]←
./-.:
abcdef
ghijkl
mnopqr
stuvwx
yzaöüß
0.0 5 10 15 20 25 min
Help_ | Searching in library

```

Example of a search mask

②

A search mask appears into which you must enter the sample name of the experiment that you would like to post-process. If you want to select an entire series of experiments, you must enter the part of the sample name that is common to the entire series of experiments. If you enter a *decimal point* for *search mask*, the system lists all experiments that are stored in the library. If *search mask* is left empty, the list of the last search procedure is displayed. The *Add* option adds the list of the new search procedure to the list of the last search procedure. Confirm your entry with *OK*.

The search routine finds all measurements that meet the search mask criteria. The display is not sorted. No more than 100 measurements can be displayed. Measurements that are not displayed can be displayed after a follow-up search with more stringent criteria in the search mask. During and after the search procedure, the header line displays the number of measurements found.





Standard  
calculation  
modes**Standard without titration**

*QExtraneous* Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.

---

*H<sub>2</sub>O El Ana* The percentage of combustion water making up the sample.

---

*Sulfur (an)* The percentage of sulfur.

---

*Nitrogen* The percentage of nitrogen.

---

**Standard with titration**

*QExtraneous* Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.

---

*Hydrogen (an)* The percentage of hydrogen making up the combustion sample.

---

*Ba(OH)<sub>2</sub>* The titrated quantity of 0.1N barium hydroxide (titration of the distilled water with which the decomposition vessel was rinsed out after the experiment).

---

*Na<sub>2</sub>CO<sub>3</sub>* Quantity of sodium carbonate that was present in the decomposition vessel (20 ml according to DIN specification; 0.05N).

---

*HCl* The titrated quantity of 0.1N hydrochloric acid (titration of the distilled water with which the decomposition vessel was rinsed out after the experiment).

---

Carbon  
calculation  
modes

The modes following immediately below are used exclusively for examinations on carbon. In addition to the heat of solution from acid formation, they consider the percentage of ash, and calculate, depending on the carbon calculation mode, the percentage of water from the sample moisture as well as the percentage of volatile components.

**Carbon: H<sub>2</sub> input, without titration**

*QExtraneous* Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.

---

*Hydrogen (an)* The percentage of hydrogen making up the combustion sample.

---

*Sulfur (an)* The percentage of sulfur.

---

*Rough moist. (raw)* The percentage of water from rough moisture.

---

*Ash (an)* The percentage of ash.

---

*Hygr. moist. (an)* The percentage of water from hygroscopic moisture.

---

*Nitrogen* The percentage of nitrogen.

---

**Carbon: H<sub>2</sub> input, with titration**

<i>QExtraneous</i>	Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.
<i>Hydrogen (an)</i>	The percentage of hydrogen making up the combustion sample.
<i>Ba(OH)<sub>2</sub></i>	The titrated quantity of 0.1N barium hydroxide (titration of the distilled water with which the decomposition vessel was rinsed out after the experiment).
<i>Na<sub>2</sub>CO<sub>3</sub></i>	Quantity of sodium carbonate that was present in the decomposition vessel (20 ml, 0.05N).
<i>HCl</i>	The titrated quantity of 0.1N hydrochloric acid (titration of the distilled water with which the decomposition vessel was rinsed out after the experiment).
<i>Rough moist. (raw)</i>	The percentage of water from rough moisture.
<i>Ash (an)</i>	The percentage of ash.
<i>Hygr. moist. (an)</i>	The percentage of water from hygroscopic moisture.

**Carbon: Volatile input, without titration**

<i>QExtraneous</i>	Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.
<i>Sulfur (an)</i>	The percentage of sulfur.
<i>Rough moist. (raw)</i>	The percentage of water from rough moisture.
<i>Ash (an)</i>	The percentage of ash.
<i>Hygr. moist. (an)</i>	The percentage of water from hygroscopic moisture.
<i>Volat. comp. (raw)</i>	The percentage of volatile components.
<i>Nitrogen</i>	The percentage of nitrogen.

**Carbon: Volatile input, with titration**

<i>QExtraneous</i>	Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.
<i>Volat. comp. (raw)</i>	The percentage of volatile components.
<i>Ba(OH)<sub>2</sub></i>	The titrated quantity of 0.1N barium hydroxide (titration of the distilled water with which the decomposition vessel was rinsed out after the experiment).
<i>Na<sub>2</sub>CO<sub>3</sub></i>	Quantity of sodium carbonate that was present in the decomposition vessel (20 ml, 0.05N).
<i>HCl</i>	The titrated quantity of 0.1N hydrochloric acid (titration of the distilled water with which the decomposition vessel was rinsed out after the experiment).
<i>Rough moist. (raw)</i>	The percentage of water from rough moisture.
<i>Ash (an)</i>	The percentage of ash.
<i>Hygr. moist. (an)</i>	The percentage of water from hygroscopic moisture.

**Acid correction based on ASTM 1989**

<i>QExtraneous</i>	Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.
<i>Hydrogen (an)</i>	The percentage of hydrogen making up the combustion sample.
<i>Na<sub>2</sub>CO<sub>3</sub></i>	Titration quantity in ml (0.34N).
<i>Rough moist. (raw)</i>	The percentage of water from rough moisture.
<i>Ash (an)</i>	The percentage of ash.
<i>Hygr. moist. (an)</i>	The percentage of water from hygroscopic moisture.
<i>Sulfur (an)</i>	The percentage of sulfur.

**Acid correction based on ASTM 240**

<i>Q</i> Extraneous	Extraneous energy from electrical ignition, the combustion of the cotton thread, and any combustion aids.
<i>H</i> ydrogen ( <i>an</i> )	The percentage of hydrogen making up the combustion sample.
<i>R</i> ough moist. ( <i>raw</i> )	The percentage of water from rough moisture.
<i>A</i> sh ( <i>an</i> )	The percentage of ash.
<i>H</i> ygr. moist. ( <i>an</i> )	The percentage of water from hygroscopic moisture.
<i>NaOH</i>	The titrated quantity in ml (0.0866N).
<i>S</i> ulfur ( <i>an</i> )	The percentage of sulfur.

④

Enter the required parameters for the calculation mode selected and confirm the dialog box after the last entry with *OK*.

```

Mode: Adiabatic 11:27:55
[.] Formular 2=[+]
02.06.1999 11:27
MEASURING PROTOCOL
=====
Experiment : 9060220
Sample : 9060220
User :
Sample properties :
Date of experim. : 02.06.1999
Start time of experim. : 11:23
Bomb No. : 1
C value : 10629 J/K
Mode : Adiabatic
Help | Print:SPACE Close:OKAY

```

Measurement protocol

```

Mode: Adiabatic 11:28:31
[.] Formular 2=[f]
Sample mass : 1.2345 g
Temperature increase : 0.0000 K
Extraneous energy : 120.00 J

Carbon(DIN): Input: H2, without ti
H2O elementary analysis: 0.00 %

Surface moisture(raw) : 0.00 %
Total water(raw) : 0.00 %
Ash (an) : 1.0000 %
Ash (raw) : 1.0000 %
Hmgr. moisture(an) : 0.0000 %
Hmgr. moisture(raw) : 0.0000 %
Hydrogen(raw) : 0.0000 %
Hydrogen(an) : 0.0000 %
Hydrogen(waf) : 0.0000 %
Volatile(raw) : 0.0000 %
Volatile(an) : 0.0000 %
Volatile(waf) : 0.0000 %

Help_ | Print:SPACE Close:OKAY

```

Measurement protocol

```

Mode: Adiabatic 11:28:59
[.] Formular 2=[f]
Volatile(raw) : 0.00 %
Volatile(an) : 0.00 %
Volatile(waf) : 0.00 %

Sulphur(an) : 0.0000 %
Sulphur(raw) : 0.0000 %
Nitrogen(an) : 0.0000 %
HCl consumed : 0.0000 ml
Ba(OH)2 consumed : 0.0000 ml
Na2CO3(NaOH) pres. : 0.0000 ml
Q sulphur : 000.000 J
Q nitrogen : 000.000 J

Ho (raw) : 228438 J/g
Ho (an) : 225476 J/g
Ho (waf) : 228006 J/g

Hu (raw) : 22949 J/g
Hu (an) : 25157 J/g
Hu (waf) : 22481 J/g

Help_ | Print:SPACE Close:OKAY

```

Measurement protocol

## ⑤

A new window appears and shows the measurement protocol with the definitive results of the experiment. You can print the measurement protocol by pressing the space bar and close the protocol window with *OK*. You can scroll through the protocol with the arrow keys.

Meaning of the individual correction parameters:

<i>H<sub>2</sub>O elementary analysis</i>	The percentage of water in the fuel sample, as determined by elementary analysis.
<i>Rough moisture (raw)</i>	The percentage of rough moisture in the supply state.
<i>Total water (raw)</i>	The percentage of water in the fuel sample in the supply state.
<i>Ash (an)</i>	The percentage of ash in the fuel sample in the “analysis moist” reference state.



<i>Ash (raw)</i>	The percentage of ash in the fuel sample in the supply state.
<i>Hygr. moisture (an)</i>	The percentage of hygroscopic moisture in the “analysis moist” reference state.
<i>Hygr. moisture (raw)</i>	The percentage of hygroscopic moisture in the supply state.
<i>Hydrogen (raw)</i>	The percentage of hydrogen in the supply state.
<i>Hydrogen (an)</i>	The percentage of hydrogen in the “analysis moist” reference state.
<i>Hydrogen (waf)</i>	The percentage of hydrogen in the “water and ash-free” reference state.
<i>Volatiles (raw)</i>	The percentage of volatile components in the supply state.
<i>Volatiles (an)</i>	The percentage of volatile components in the “analysis moist” reference state.
<i>Volatiles (waf)</i>	The percentage of volatile components in the “water and ash-free” reference state.
<i>Sulfur (an)</i>	The percentage of sulfur in the fuel sample in the “analysis moist” reference state.
<i>Sulfur (raw)</i>	The percentage of sulfur in the fuel sample in the supply state.
<i>Nitrogen (an)</i>	The percentage of nitrogen in the fuel sample in the “analysis moist” reference state.
<i>HCl consumed</i>	The titrated quantity of hydrochloric acid.
<i>Ba(OH)<sub>2</sub> consumed</i>	The titrated quantity of barium hydroxide.
<i>Na<sub>2</sub>CO<sub>3</sub> present</i>	The quantity of Na <sub>2</sub> CO <sub>3</sub> present in the decomposition vessel.
<i>Q sulfur</i>	The heat of solution from the formation of sulfuric acid.
<i>Q nitrogen</i>	The heat of solution from the formation of nitric acid.
<i>Ho (raw)</i>	The specific gross calorific value of the fuel sample in the supply state.
<i>Ho (an)</i>	The specific gross calorific value of the fuel sample in the “analysis moist” reference state.
<i>Ho (waf)</i>	The specific gross calorific value of the fuel sample in the “water and ash-free” reference state.

<i>Hu (raw)</i>	The specific net calorific value of the fuel sample in the supply state.
<i>Hu (an)</i>	The specific net calorific value of the fuel sample in the “analysis moist” reference state.
<i>Hu (waf)</i>	The specific net calorific value of the fuel sample in the “water and ash-free” reference state.

## 12 Experiment simulation

In many cases it is helpful to perform gross calorific value experiments or to calculate possible experiment results without actually performing the combustion experiment. Using the *Simulation* dialog box in the *Experiments* menu box, the calorimeter system simulates experiments on the basis of data that is provided.

This option is especially useful if a calibration was unintentionally performed instead of a determination of a gross calorific value or vice-versa. This can be corrected through simulation by using the increase in temperature of the misinterpreted measurement.

①

Open the Simulation dialog box in the Experiments menu window.

```

Mode: Adiabatic 11:57:18
[ ] Experiment 9060223
Weighed-in qu 1.0411
Extran1 120.0
Sample name 9060223
Sample proper
User
>> Bomb 1
Cell 1
[ ] Calibration
abcdefghijklmnopqrstuvwxyz
→ [ OK ] ← [ Cancel ]
Help_ | Entering experiment parameters
  
```

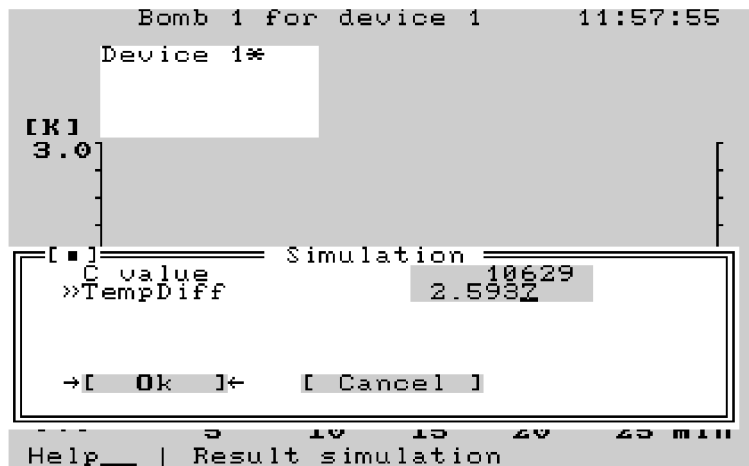
Simulation dialog box

②

Move the cursor with *Tab* to the entry boxes and enter the sample data with which the simulation will be performed.

③

As soon you have confirmed the data with *OK*, a dialog box appears for entering the simulation parameters.



Entry of simulation parameters

④

You must enter the following parameters:

*C value*: Heat capacity of the calorimeter system

*TempDiff* The temperature difference at which the simulated combustion is conducted.

⑤

Confirm the dialog box with *OK*. Using the *Evaluation* dialog box (See Section 12, *Evaluating experiments*), you can post-process the simulated experiment in the normal manner or convert the results into the desired reference state.

## 13 Care and maintenance

In order to ensure problem-free operation over a long time, the following maintenance tasks should be performed on the calorimeter system:

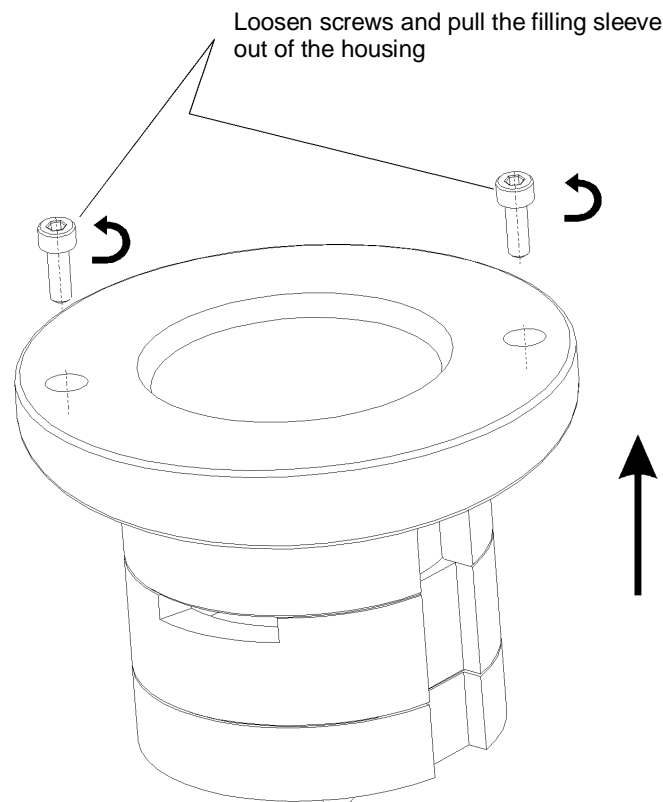
### 13.1 Sieve insert

**Check the sieve insert daily while refilling the prepared liquid.**

The entire volume of water in the system is constantly circulated and kept free of impurities by the sieve insert in the filling sleeve of the expansion container.

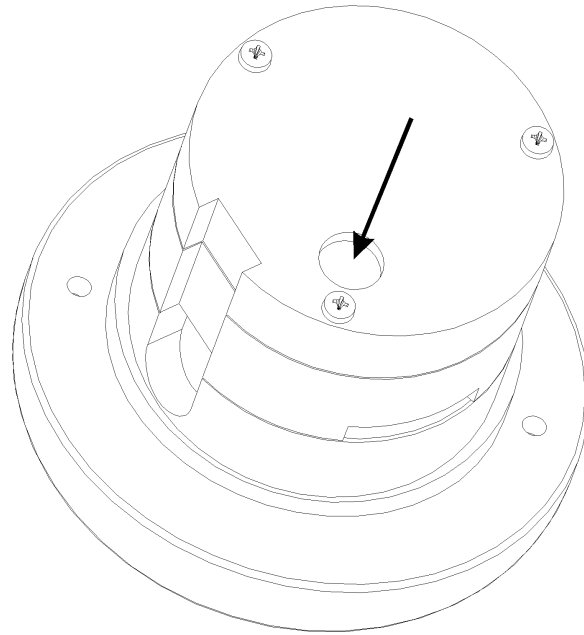
This sieve insert must nevertheless be checked at regular intervals for deposits and accumulations of dirt and so forth.

If clearly visible deposits have become attached to the surface of the sieve, the sieve must be cleaned. To do this, turn off the device and remove the complete filling sleeve by loosening the two screws from the housing (see the illustration below).



**Removing the filling sleeve**

After the filling sleeve has been removed, the filter insert can be rinsed off under running tap water. To do this, turn the filling sleeve around. The stream of water will meet the built-in sieve element as it flows in through the opening in the base.



#### Cleaning the sieve insert

- After cleaning the sieve, install the filling sleeve into the unit in the reverse order.
- For tough dried-on deposits, the sieve must be removed from the sleeve by loosening the three screws on the underside of the filter element. The deposits must be removed with a brush.
- During routine operation, liquid is removed from the system through evaporation and by adhering to the decomposition vessel.  
If this error message appears on the display during normal operation:

*Refill with water or empty IV (=inner vessel)*

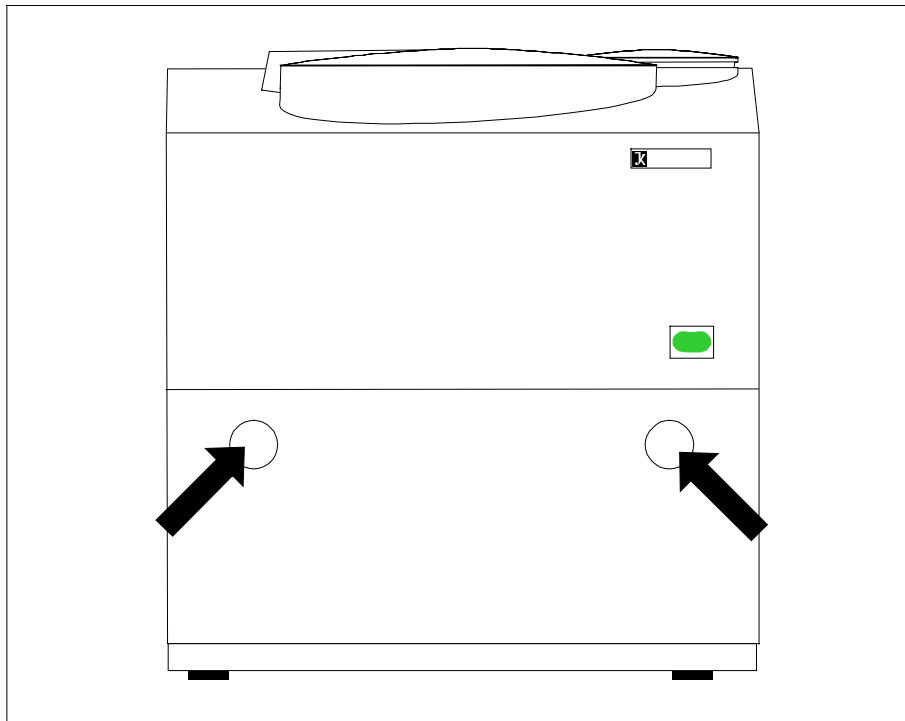
you should add 50 ml of the mixture to the expansion container. If the message on the display does not go away, repeat the refilling process in increments of 50 ml.

## 13.2 Changing the water

The liquid in the system should be changed every 3 to 4 months. Each time you replace the water, check the sieve for sludge (visual inspection).

①

Turn off the device using the **Exit** menu item (the cover on the measurement cell closes automatically) and then turn the power switch to "Off". Before draining the water, the device must be turned off on the power switch. Open the front flap of the measurement cell or of the measurement cells by pushing on both recesses at the same time.



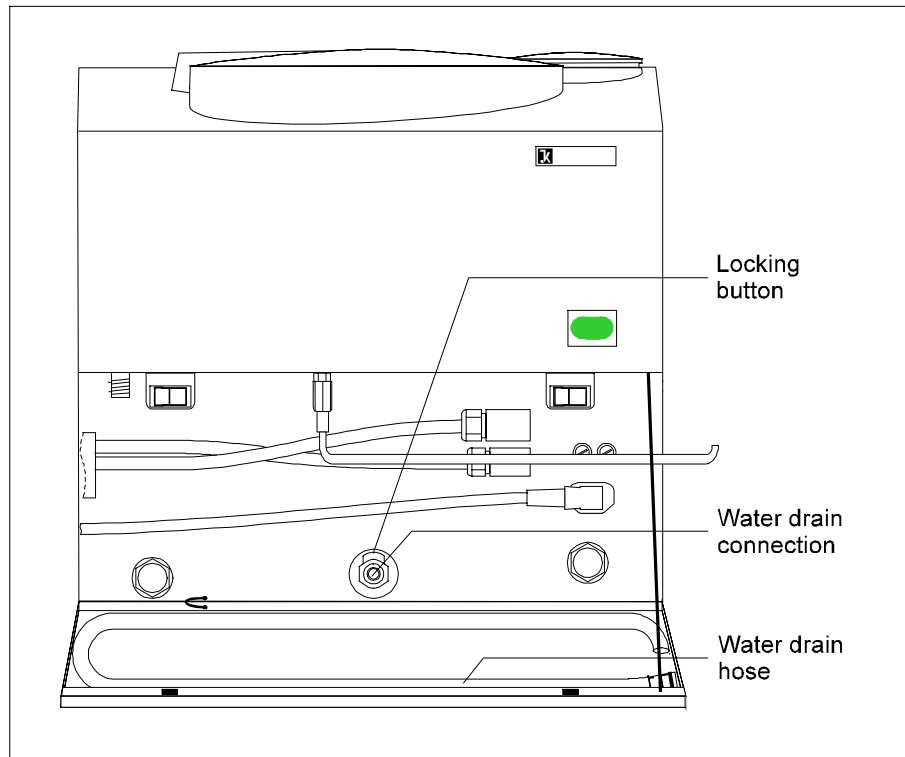
Opening the front flap

②

As soon as you push the water drain hose into the water drain connection, the cooling water system empties itself. Push on the locking button of the water drain connection to insert and remove the hose.



**After the water is replaced, the sieve insert may become dirty again in a matter of minutes. In this case, it should be cleaned as described in Section 13.1. In some cases this procedure must be repeated 2 or 3 times to remove all accumulated deposits from the system.**

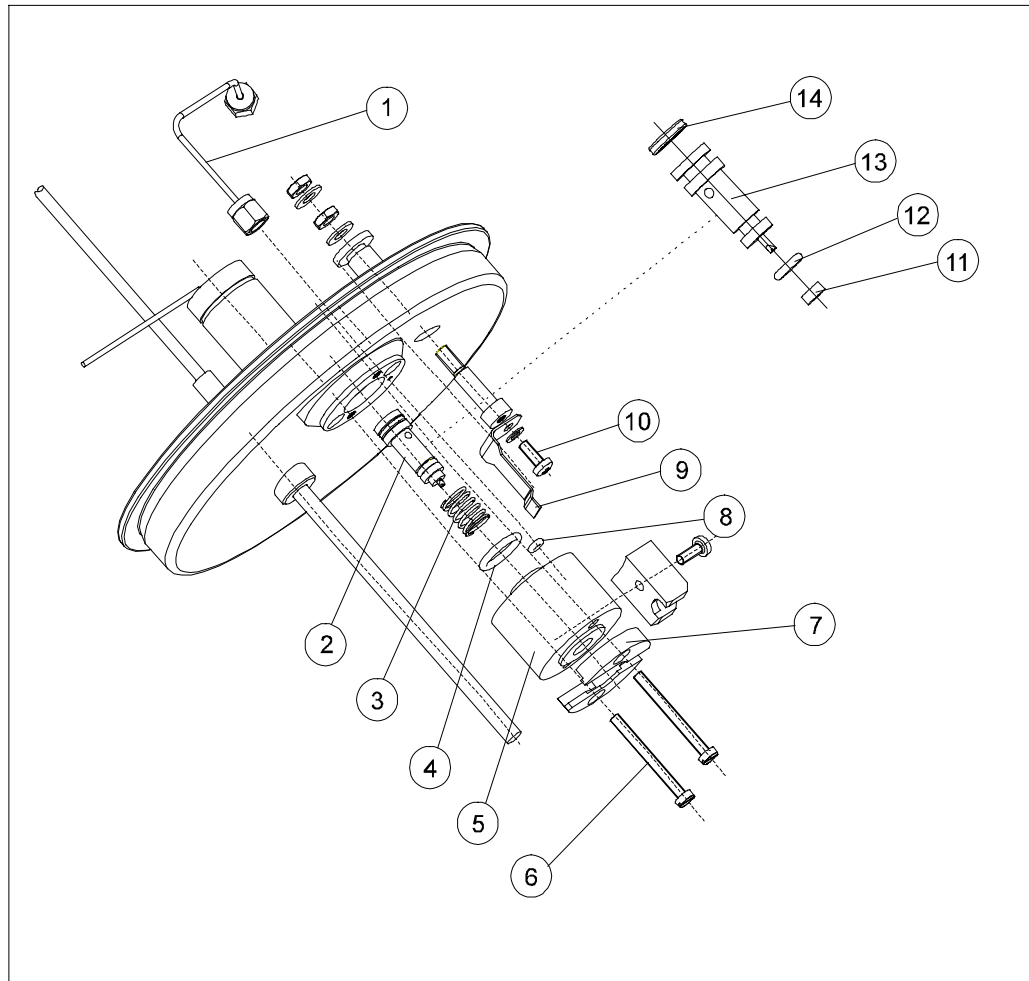


Inserting the water drain hose into the water drain connection



### 13.3 Replacing the inner cover / O<sub>2</sub> filling piston

If it should become necessary to break down the inner cover to replace parts, it can be put back together again as shown in the following illustration.



Replacement parts  
of the inner cover

#### Spare parts list

Item	Pieces	Name	
1	1	Capillary cpl.	
2	1	Piston cpl.	
3	1	Pressure spring VD123	1.4310
4	1	O ring 11 x 2	FPM (VITON)
5	1	Filling head	Peek
6	2	Cylinder screw DIN84 M3x30	A2
7	1	Centering ring	1.4301
8	1	O ring 2 x 1.6	V80G
9	1	Contact spring	2.1020.34
10	2	Lens screw DIN7985 M3x8 A2	1.4301
11	1	Seal disk	
12	1	O ring 4 x 1.5	
13	1	Piston	
14	1	Quad ring 5.28 x 1.78	

Only the parts with item numbers are available as replacement parts.

If it should become necessary to replace the O<sub>2</sub> filling piston (item 2), proceed as follows:

①

Loosen the screws (item 6) with a blade-screwdriver.

②

Remove the centering ring (item 7) together with the filling head (item 5), the pressure spring (item 3) and the piston (item 2)

Caution: The seals (item 4 and item 8) are free

③

Push the pressure spring (item 3) onto the new piston and insert both parts into the filling head. The remainder of the assembly takes place in the opposite order.

Caution: During reassembly, make certain the filling head is in the correct position (item 5). The seals (item 4 and item 8) should be aligned on the opposite side of the inner cover.



**After replacing the piston and the seal disks, the decomposition vessel must be refilled with oxygen using the maintenance menu (menu items O<sub>2</sub> fill / depressurize) and then depressurized in order to test the complete unit for proper seal.**

### 13.4 Replacing the O<sub>2</sub> seal

- If a leak is detected while filling the decomposition vessel with oxygen, the O<sub>2</sub> seal on the filling piston must be replaced:
  - Remove the decomposition vessel from the measurement cell.
  - Activate the *O<sub>2</sub> seal* menu item from the *Maintenance menu* to extend the piston.
  - Remove the small orange seal from the extended piston (see Section 13.3, item 11).
  - Insert the new seal (included with delivery) onto the piston.
  - Activate the *O<sub>2</sub> seal* menu item from the *Maintenance menu* again to retract the piston again.

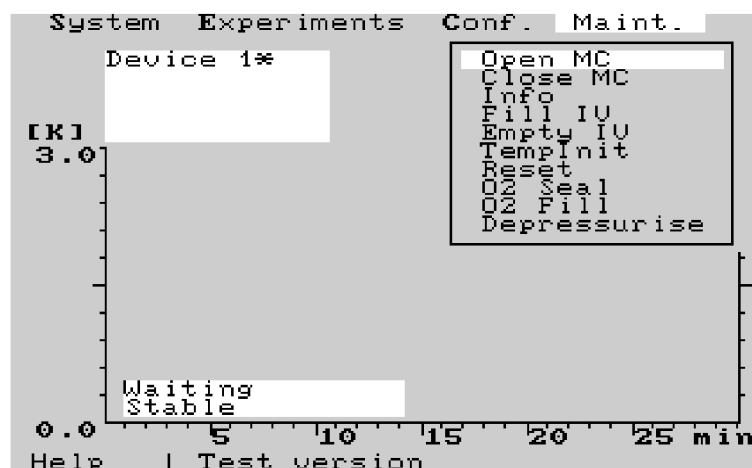
### 13.5 Decomposition vessels

For maintenance of the decomposition vessel, please read the C5010/5012 Operating Instructions.

## 14 Troubleshooting

The C 5000 calorimeter system is subject to strict quality control during manufacturing. If improper functionality should nevertheless occur, you will find a series of malfunction situations with the appropriate measures for eliminating the problem. Most malfunctions are displayed in the header line of the display. Alternatively or in addition, a message box may appear which the user must acknowledge. If your attempts at eliminating problems are unsuccessful, please contact our service department.

### 14.1 Maintenance menu



Maintenance menu

The maintenance menu offers the possibility of performing a series of system functions in the case of a malfunction. The commands of the maintenance menu can only be performed if the measurement cell is in maintenance status.

The functions *Open MC*, *Close MC*, *Fill IV*, *Empty IV*, *Fill oxygen*, *Depressurize* are activated by the corresponding menu command and end automatically. While the function is being performed, the menu item in question is locked.

*Open MC*      Opens the measurement cell cover.

*Close MC*     Closes the measurement cell cover.

*Info*            An information window is shown /hidden. The function can also be called up or closed with the "." key. You can print the information window with the Space bar ( ).

*Fill IV*         The inner vessel is filled with water. The filling process ends automatically after about 120 seconds.

*Empty IV*      The water is pumped out of the inner vessel. The emptying ends automatically when the inner vessel is empty.

*TempInit*      The temperature measurement is reinitialized.

*Reset*          Reset of the temperature control of the outer vessel.

- O<sub>2</sub> seal* The piston on the oxygen filling unit in the measurement cell cover is extended and then retracted again when it is activated a second time.
- Fill O<sub>2</sub>* A decomposition vessel that is suspended in the measurement cell cover is filled with oxygen. The process ends after about 50 seconds. The process is displayed in the process window. During this procedure, the decomposition vessel cannot be removed.
- Depressurize* Over-pressure is drained from a decomposition vessel that is suspended into the measurement cell cover. The procedure ends automatically after about 70 seconds. During this procedure, the decomposition vessel cannot be removed. The process is displayed in the process window.

## 14.2 Malfunction situations

**Malfunction situations with the associated message on the display:**

- **The coding on the decomposition vessel is not recognized:**

**Messages on the display:**

- **Bomb “x” not for cell “y” (duo control)**
- **No assignment possible**
- **Bomb “x” is already assigned**
- **Bomb not recognized**

The system displays the malfunction and interrupts the measurement. Check the coding ring on the decomposition vessel and the optical detection unit on the measurement cell for dirt or precipitates. Check the correct assignment of the decomposition vessel to the measurement cell. To continue your measurement series, you may have to turn off detection of decomposition vessels (*Conf. / Settings / Bombs ID*). Contact the service department.

- **The measurement cell cover does not open or close completely:**

**Messages on the display:**

- **Cover is not closed / open**

The system displays the malfunction and interrupts the measurement. Try to open the measurement cell cover manually and close it again. To do this, execute the commands *Close MC* and *Open MC* in the Maintenance menu. If the function cannot be restored again, please contact the service department.

- **Full status of the inner vessel is not achieved within 200 seconds:**

**Messages on the display:**

- **Water filling time exceeded**

The system displays the malfunction and interrupts the measurement. Repeat the attempt.

If the error appears again, please contact the service department.

- **No increase in temperature after electrical ignition:**

**Messages on the display:**

- **No increase in temperature**

The system displays the malfunction and interrupts the measurement. Check the ignition wire, the cotton thread, the fuel sample and the O<sub>2</sub> supply. It may be that you will have to use a combustion aid.

- **The combustion experiment lasts too long:**

**Messages on the display:**

- **Preliminary experiment > 13 minutes**

- **Main experiment > 16 minutes**

Adiabatic and isoperibolic measurements are interrupted after 13 minutes in the preliminary experiment (warm-up) and 16 minutes in the main experiment. Check the stirrer drive (rotating stirring rod after the inner vessel has been emptied), the seal of the decomposition vessel (see C5010 / C5012 Operating Instructions), the function of the cooling unit as indicated by heat escaping from the cooler fans.

- **Error while recording temperature:**

**Messages on the display:**

- **Error temp. meas.**

The malfunction is displayed in the header line of the display. An acoustic signal is heard at the same time. The temperature display stops changing. You can try to eliminate the error with the Temp-init. command in the Maintenance menu. If this does not work, the calorimeter must be restarted.

- **Water sensor error:**

**Messages on the display:**

- **Water sensor error**

Please contact your service department.

- **Problem with ignition wire:**

**Messages on the display:**

**- Problem with ignition wire**

This error message is displayed if the ignition capability can no longer be ensured during the measurement. The experiment is interrupted. Check the ignition wire, the ignition wire fastening and the contact spring on the filling head of the inner cover.

- **Sample rack invalid:**

**Messages on the display:**

**- Sample rack invalid**

For more information on this message, see the Operating Instructions for the C5020 sample rack.

- **Temperature control of the system unstable:**

**Messages on the display:**

**- “Unstable” status**

The “unstable” condition may last up to 10 minutes after turning on the machine, and up to 5 minutes between measurements. If this time is exceeded significantly, or if the “stable” condition is no longer reached at all, select the *Reset* option from the maintenance menu.

Check in addition the functionality of the cooler (make sure hot air is coming out of the outlet). If the “Stable” status is not reached within an additional 30 minutes, turn the machine off. If the problem persists after the unit has been turned back on again, please contact your service department.

- **Memory is too low:**

**Messages on the display:**

**- Memory is too low**

The total number of all measurements that the C5000 can manage during a run sequence is limited to about 240. This limitation is reached as early as after 50 hours of continuous operation (25 hours with the duo-control), and the message above appears. The machine should then be turned off. This number has nothing to do with the storage capacity of the library.

**A malfunction situation without a direct message on the display:**

- **Loss of power, controller in an undefined state:**

If no measurement was running, the system can be restarted by turning it off and back on again. If the system was in the process of performing a measurement, proceed as follows:

1. Turn the system off and back on, and start normally.
2. If there is residue in the inner vessel, confirm *Empty IV*.
3. Open the *Maintenance* menu
4. Bleed the over-pressure from the decomposition vessel with “*Depressurize*”

- **O<sub>2</sub> filling does not work:**

Check the O<sub>2</sub> supply to the unit (30 bar). If there is a detectable loss of seal during the filling procedure, replace the O<sub>2</sub> seal (see Section 13, Care and maintenance).

- **O<sub>2</sub> ventilation does not work:**

Check the O<sub>2</sub> supply to the unit (30 bar). Check the way the ventilation hose is installed and laid out. There must be no kinks or obstructions in the degassing hose. Check the settings in the *Config., Settings* menu. The *Decomposition* item must not be set, since this turns off O<sub>2</sub> ventilation.

- **Incomplete combustion:**

Check the O<sub>2</sub> supply to the unit (30 bar). Use a combustion aid if necessary (see also the applicable standards under "Taking samples/sample preparation").

- **Cover cannot be removed from the decomposition vessel:**

The O<sub>2</sub> filling / ventilation procedure is still running (see the status window in the display).

- **When suspending the decomposition vessel into the measurement cell, the display Bomb ↓ is not changed to *START*:**

Check the following items:

- "Stable" state not yet achieved (see status window).
- The decomposition vessel was not removed when an experiment was interrupted.
- No experiment is prepared.
- The maintenance menu is open.
- Problem with the contact spring.
- Ignition wire is defective.

### 14.3 Performing an adjustment (adiabatic mode)

If the unit is going to be operated in adiabatic mode, an adjustment is required first. This has already been performed at the factory during the functional test. Under normal laboratory conditions, the user must perform an adjustment again if:

- Measurement times for adiabatic measurements regularly take longer than 25 minutes.
- Adiabatic measurements are frequently interrupted because the time limit for the preliminary or the main experiment has been exceeded.

Procedure for an adjustment:

- Set the operating mode to "Adjustment" (see Section 8; Item 8.8 *System settings*). For a duo-control unit, this setting refers to both measurement cells.
- Prepare a mock measurement (see Section 10, Item 10.3). An empty crucible is used. Enter the fictitious value "1" for the weighed in quantity.
- Start the measurement.
- The process completes itself automatically within 72 to 120 minutes. Upon completion, a value  $D = \dots$  is displayed in the result window. You should make note of this value in your device materials. In this case the adjustment was successful.

Then switch back to the desired operating mode. If you would like to work in adiabatic mode, the **calibrations** must be **repeated** for all decomposition vessels.

If the adjustment is not successful within 120 minutes, the procedure is interrupted without any result.

In this case, please contact your service department.



## 15 Accessories and Consumables

Description of part	ID No.
<b>Accessories</b>	
C 5010 IKA Decomposition vessel	7114000
C 5012 IKA Decomposition vessel	7174200
C 5010.4 Attachment for combustibile crucible	3016900
C 5010.5 Attachment for large crucible	3055900
C 5020 Sample rack	7145000
Ventilation station C 5030	7198000
C 21 Pelleting press	1605300
C 29 Reduction valve	0750200
C 30 Oxygen purifier	0750300
C 44 Standard sheet DIN 51900, German	0750800
C 44 E Standard sheet DIN 51900, English	2103500
LX 300 EPSON printer (230 V, 50/60 Hz)	7000100
LX 300 EPSON printer (115 V, 50/60 Hz)	7000101
SBC 31 Analytical scale (230 V, 50/60 Hz)	7064900
SBC 31 Analytical scale (115 V, 50/60 Hz)	7064901
BP 61 Sartorius analytical scale, compl. (230 V, 50/60 Hz)	7062000
BP 61 Sartorius analytical scale, compl. (115 V, 50/60 Hz)	7062001
AC 121 S Sartorius analytical scale, compl. (230 V, 50/60 Hz)	7062200
AC 121 S Sartorius analytical scale, compl. (115 V, 50/60 Hz)	7062201

Description of part	ID No.
<b>Consumables</b>	
C 5050 Equipment set, (3x C5.1, 1x C5010.3, 1x C710.4, 1x C723)	7114100
C 710.4 Cotton thread, cut to length (500 pieces)	1483700
C 5010.3 Ignition wire, replacement (5 pieces)	7122800
C5010.6 Electrode deflector set	7113800
C 5010.7 O ring set	7113900
C 4 Quartz dish	1695500
C 5 VA Combustion crucible set (25 pieces)	1749500
C 6 Quartz dish, large	0355100
C 710.2 VA Combustion crucible set, large (25 pieces)	1483500
C 9 Gelatin capsules (100 pieces)	0749900
C 10 Acetobutyrate capsules (100 pieces)	0750000
C 12 Combustion bags, 40 x 35 mm (100 pieces)	2201500
C 12A Combustion bags 70 x 40 mm (100 pieces)	2201400
C 43 Benzoic acid (NBS 39i, 30g)	0750600
C 43A Benzoic acid (NBS 39i, 100g)	0750700
C 723 Benzoic acid in tablet form (50 pieces)	1505500
C 14 Combustible crucible (100 pieces)	7224500

## 16 Basic calculations

The following sections list mathematical formulas that are used to calculate results of measurements. The calorimeter system acquires the data required for the measurements partially during the combustion process and the data is partially the results of analyses of examination on fuel samples or on combustion products. The calculations correspond to the applicable standards (DIN 51900, ASTM 240D, ISO 1928, BSI) for gross calorific values and net calorific values.

The following indices are used for the various reference states:

raw	-	supply state
an	-	analysis moist or air dry
waf	-	water and ash free

Section 16.9 contains an alphabetical list with the meanings of the formula symbols.

### 16.1 Calculations for calibration

#### Heat capacity (C value) of the calorimeter system

$$C = \frac{H_{OB} \cdot m + Q_1}{\Delta T}$$

#### average value MW

$$MW = \frac{M_1 + M_2 + \dots + M_n}{n}$$

#### Average relative error MRF

$$MRF = 100 \cdot \frac{1}{MW} \cdot \sqrt{\frac{D_1^2 + D_2^2 + \dots + D_n^2}{n-1}}$$

### 16.2 Calculations during an experiment

#### Gross calorific value of the fuel sample

$$H_{Oan} = \frac{C \cdot \Delta T - Q_z}{m}$$

Remark: This is the provisional gross calorific value without acid or water correction.

### 16.3 “Standard without titration” mode

**Net calorific value of the fuel sample**

$$H_{Uan} = H_{Oan} - (H_2O \cdot 24.41)$$

**Energy from the formation of sulfuric acid**

$$Q_S = S_{an} \cdot m \cdot 94.62$$

**Energy from the formation of nitric acid**

$$Q_N = N_{an} \cdot m \cdot 43$$

### 16.4 “Standard with titration” mode

**Percentage of sulfur**

$$S_{an} = \frac{Q_S}{m \cdot 94.62}$$

**Energy from the formation of sulfuric acid**

$$Q_S = 15.1 \cdot (\text{Ba}(\text{OH})_2 + \text{HCl} - \text{Na}_2\text{CO}_3)$$

**Energy from the formation of nitric acid**

$$Q_N = 6 \cdot (\text{Na}_2\text{CO}_3 - \text{HCl})$$

**Sum of extraneous energy**

$$\sum Q = Q_Z + Q_S + Q_N$$

**Gross calorific value of the fuel sample**

$$H_{Oan} = \frac{C \cdot \Delta T - \sum Q}{m}$$

**Net calorific value of the fuel sample**

$$H_{Uan} = H_{Oan} - (H_{2an} \cdot 218.13)$$

## 16.5 “Carbon: H2 input, without titration” mode

Conversion factor from reference state *an* to *raw*

$$F_1 = \frac{100 - H_{2O_{raw}}}{100 - hF_{an}}$$

Percentage of sulfur

$$S_{raw} = S_{an} \cdot F_1$$

Percentage of water

$$H_{2O_{raw}} = gF_{raw} + hF_{raw}$$

Percentage of ash

$$A_{raw} = F_1 \cdot A_{an}$$

Hygroscopic moisture

$$hF_{raw} = F_1 \cdot hF_{an}$$

Percentage of hydrogen

$$H_{2raw} = F_1 \cdot H_{2an}$$

$$H_{2waf} = H_{2an} \cdot \frac{100}{100 - (A_{an} + hF_{an})}$$

Volatile components

$$fB_{raw} = \frac{100 - (H_{2O_{raw}} + A_{raw})}{100} \cdot fB_{waf}$$

$$H_{2O_{raw}} = gF_{raw} + hF_{an}$$

$$fB_{an} = \frac{fB_{raw}}{F_1}$$

$$fB_{\text{waf}} = \frac{0.115 - \sqrt{0.115^2 - 4[(H_{2\text{waf}} - 2.98) \cdot 0.00142]}}{0.00284}$$

Remark: This approximation formula applies for mineral coal with a percentage of volatile components between 6% and 40%.

### Energy from the formation of sulfuric acid

$$Q_S = S_{\text{an}} \cdot m \cdot 94.62$$

### Energy from the formation of nitric acid

$$Q_N = N_{\text{an}} \cdot m \cdot 43$$

### Gross calorific value of the fuel sample

$$H_{\text{Oraw}} = H_{\text{Oan}} \cdot F_1$$

$$H_{\text{Oan}} = \frac{C \cdot \Delta T - Q_z - Q_S}{m}$$

$$H_{\text{Owaf}} = \frac{100}{100 - hF_{\text{an}}} \cdot \frac{100}{100 - \left( A_{\text{an}} \cdot \frac{100}{100 - hF_{\text{an}}} \right)} \cdot H_{\text{Oan}}$$

### Net calorific value of the fuel sample

$$H_{\text{Uraw}} = (H_{\text{Uan}} + 24.41 \cdot hF_{\text{an}}) \cdot F_1 - (H_2O_{\text{raw}} \cdot 24.41)$$

$$H_{\text{Uan}} = H_{\text{Oan}} - (H_{2\text{an}} \cdot 218.13 + hF_{\text{an}} \cdot 24.41)$$

$$H_{\text{Uwaf}} = (H_{\text{Oraw}} + 24.41 \cdot H_2O_{\text{raw}}) \cdot \frac{100}{100 - hF_{\text{an}}} \cdot \frac{100}{100 - \left( A_{\text{an}} \cdot \frac{100}{100 - hF_{\text{an}}} \right)}$$

## 16.6 “Carbon: H2 input, with titration” mode

Conversion factor from reference state *an* to *raw*

$$F_1 = \frac{100 - H_{2O_{raw}}}{100 - hF_{an}}$$

Percentage of sulfur

$$S_{an} = \frac{Q_s}{m \cdot 94.62}$$

$$S_{raw} = S_{an} \cdot F_1$$

Percentage of water

$$H_{2O_{raw}} = gF_{raw} + hF_{raw}$$

Percentage of ash

$$A_{raw} = F_1 \cdot A_{an}$$

Hygroscopic moisture

$$hF_{raw} = F_1 \cdot hF_{an}$$

Percentage of hydrogen

$$H_{2raw} = F_1 \cdot H_{2an}$$

$$H_{2waf} = H_{2an} \cdot \frac{100}{100 - (A_{an} + hF_{an})}$$

Volatile components

$$fB_{raw} = \frac{100 - (H_{2O_{raw}} + A_{raw})}{100} \cdot fB_{waf}$$

$$H_{2O_{raw}} = gF_{raw} + hF_{raw}$$

$$fB_{an} = \frac{fB_{raw}}{F_1}$$

$$fB_{\text{waf}} = \frac{0.115 - \sqrt{0.115^2 - 4[(H_{2\text{waf}} - 2.98) \cdot 0.00142]}}{0.00284}$$

Remark: This approximation formula applies for mineral coal with a percentage of volatile components between 6% and 40%.

### Energy from the formation of sulfuric acid

$$Q_S = 15.1 \cdot (\text{Ba}(\text{OH})_2 + \text{HCl} - \text{Na}_2\text{CO}_3)$$

### Energy from the formation of nitric acid

$$Q_N = 6 \cdot (\text{Na}_2\text{O}_3 - \text{HCl})$$

### Sum of extraneous energy

$$\sum Q = Q_Z + Q_S + Q_N$$

### Gross calorific value of the fuel sample

$$H_{\text{Oraw}} = H_{\text{Oan}} \cdot F_1$$

$$H_{\text{Oan}} = \frac{C \cdot \Delta T - \sum Q}{m}$$

$$H_{\text{Owaf}} = \frac{100}{100 - hF_{\text{an}}} \cdot \frac{100}{100 - \left( A_{\text{an}} \cdot \frac{100}{100 - hF_{\text{an}}} \right)} \cdot H_{\text{Oan}}$$

### Net calorific value of the fuel sample

$$H_{\text{Uraw}} = (H_{\text{Uan}} + 24.41 \cdot hF_{\text{an}}) \cdot F_1 - H_{2\text{O}_{\text{raw}}} \cdot 24.41$$

$$H_{\text{Uan}} = H_{\text{Oan}} - (H_{2\text{an}} \cdot 218.13 + hF_{\text{an}} \cdot 24.41)$$

$$H_{\text{Uwaf}} = (H_{\text{Oraw}} + 24.41 \cdot H_{2\text{O}_{\text{raw}}}) \cdot \frac{100}{100 - hF_{\text{an}}} \cdot \frac{100}{100 - \left( A_{\text{an}} \cdot \frac{100}{100 - hF_{\text{an}}} \right)}$$



## 16.7 “Carbon: volatile input, without titration” mode

Conversion factor from reference state *an* to *raw*

$$F_1 = \frac{100 - H_{2O_{raw}}}{100 - hF_{an}}$$

Percentage of sulfur

$$S_{raw} = S_{an} \cdot F_1$$

Percentage of water

$$H_{2O_{raw}} = gF_{raw} + hF_{raw}$$

Percentage of ash

$$A_{raw} = F_1 \cdot A_{an}$$

Hygroscopic moisture

$$hF_{raw} = F_1 \cdot hF_{an}$$

Percentage of hydrogen

$$H_{2waf} = 2.98 + 0.115 \cdot fB_{waf} - 0.00142 \cdot fB_{waf}^2$$

$$H_{2an} = \frac{H_{2waf} [100 - (A_{an} + hF_{an})]}{100}$$

$$H_{2raw} = F_1 \cdot H_{2an}$$

Volatile components

$$fB_{an} = \frac{fB_{raw}}{F_1}$$

$$fB_{waf} = fB_{raw} \cdot \frac{100}{100 - (H_{2O_{raw}} + A_{raw})}$$

Energy from the formation of sulfuric acid

$$Q_s = S_{an} \cdot m \cdot 94.62$$

**Energy from the formation of nitric acid**

$$Q_N = N_{an} \cdot m \cdot 43$$

**Gross calorific value of the fuel sample**

$$H_{Oraw} = H_{Oan} \cdot F_1$$

$$H_{Oan} = \frac{C \cdot \Delta T - Q_z - Q_s}{m}$$

$$H_{Owaf} = \frac{100}{100 - hF_{an}} \cdot \frac{100}{100 - \left( A_{an} \cdot \frac{100}{100 - hF_{an}} \right)} \cdot H_{Oan}$$

**Net calorific value**

$$H_{Uraw} = (H_{Uan} + 24.41 \cdot hF_{an}) \cdot F_1 - H_2O_{raw} \cdot 24.41$$

$$H_{Uan} = H_{Oan} - (H_{2an} \cdot 218.13 + hF_{an} \cdot 24.41)$$

$$H_{Uwaf} = (H_{Oraw} + 24.41 \cdot H_2O_{raw}) \cdot \frac{100}{100 - hF_{an}} \cdot \frac{100}{100 - \left( A_{an} \cdot \frac{100}{100 - hF_{an}} \right)}$$

**16.8 “Carbon: volatile input, with titration” mode****Conversion factor from reference state *an* to *raw***

$$F_1 = \frac{100 - H_2O_{raw}}{100 - hF_{an}}$$

**Percentage of sulfur**

$$S_{an} = \frac{Q_s}{m \cdot 94.62}$$

$$S_{raw} = S_{an} \cdot F_1$$

**Percentage of water**

$$H_{2O_{raw}} = gF_{raw} + hF_{raw}$$

**Percentage of ash**

$$A_{raw} = F_1 \cdot A_{an}$$

**Hygroscopic moisture**

$$hF_{raw} = F_1 \cdot hF_{an}$$

**Percentage of hydrogen**

$$H_{2raw} = F_1 \cdot H_{2an}$$

$$H_{2waf} = 2.98 + 0.115 \cdot fB_{waf} - 0.00142 \cdot fB_{waf}^2$$

**Volatile components**

$$fB_{an} = \frac{fB_{raw}}{F_1}$$

$$fB_{waf} = fB_{raw} \cdot \frac{100}{100 - (H_{2O_{raw}} + A_{raw})}$$

**Energy from the formation of nitric acid**

$$Q_N = 6 \cdot (Na_2O_3 - HCl)$$

**Energy from the formation of sulfuric acid**

$$Q_S = 15.1 \cdot (Ba(OH)_2 + HCl - Na_2CO_3)$$

**Sum of extraneous energy**

$$\sum Q = Q_Z + Q_S + Q_N$$

**Gross calorific value of the fuel sample**

$$H_{Oraw} = H_{Oan} \cdot F_1$$

$$H_{Oan} = \frac{C \cdot \Delta T - \sum Q}{m}$$

$$H_{Owaf} = \frac{100}{100 - hF_{an}} \cdot \frac{100}{100 - \left( A_{an} \cdot \frac{100}{100 - hF_{an}} \right)} \cdot H_{Oan}$$

**Net calorific value**

$$H_{Uraw} = (H_{Uan} + 24.41 \cdot hF_{an}) \cdot F_1 - H_2O_{raw} \cdot 24.41$$

$$H_{Uan} = H_{Oan} - (H_{2an} \cdot 218.13 + hF_{an} \cdot 24.41)$$

$$H_{Uan} = H_{Oan} - (H_2O \cdot 24.41)$$

$$H_{Uwaf} = (H_{Oraw} + 24.41 \cdot H_2O_{raw}) \cdot \frac{100}{100 - hF_{an}} \cdot \frac{100}{100 - \left( A_{an} \cdot \frac{100}{100 - hF_{an}} \right)}$$

## 16.9 Formula symbols

$A_{an}$	= Percentage of ash in the reference state analysis moist [%]
$A_{raw}$	= Percentage of ash in the supply state [%]
$Ba(OH)_2$	= Titrated quantity 0.1 N barium hydroxide [ml]
$C$	= Heat capacity of the calorimeter [J/K]
$D_x$	= Difference between the average value AV and the individual measured value $M_x$
$F_1$	= Conversion factor from reference state "an" to "raw"
$fB_{an}$	= Volatile components in the supply state [%]
$fB_{raw}$	= Volatile components in the reference state analysis moist [%]
$fB_{waf}$	= Volatile components in the reference state water and ash-free [%]
$gF_{raw}$	= Rough moisture
$H_{Oan}$	= Gross calorific value in reference state analysis moist [J/g]
$H_{OB}$	= Gross calorific value of the calibration substance
$H_{Oraw}$	= Gross calorific value of the sample in supply state [J/g]
$H_{Owaf}$	= Gross calorific value of the sample in the reference state water and ash-free [J/g]
$H_{Uan}$	= Net calorific value in reference state analysis moist [J/g]
$H_{Uraw}$	= Net calorific value in the supply state [J/g]
$H_{Uwaf}$	= Net calorific value in the reference state water and ash-free [J/g]
$HCl$	Titrated quantity of hydrochloric acid [ml]
$hF_{an}$	= Hygroscopic moisture [%]
$H_{2an}$	= Percentage of hydrogen in reference state analysis moist [%]
$H_{2raw}$	= Percentage of hydrogen in supply state [%]
$H_{2waf}$	= Percentage of hydrogen in the reference state water and ash-free [%]
$H_2O$	= Percentage of total water 9sum of combustion water, rough moisture and hygroscopic moisture [%]
$H_2O_{raw}$	= Percentage of total water in supply state [%]
$m$	= mass of the fuel sample [g]
$M_x$	= $x^{th}$ measured value
$MRF$	= Average relative error
$MW$	= Average value
$n$	= Number of calibration measurements
$N_{an}$	= Nitrogen in the reference state analysis moist [%]
$Na_2CO_3$	= The quantity of sodium carbonate present [ml]
$\Sigma Q$	= The sum of extraneous energy, as a function of the calculation mode [J]
$Q_N$	= Extraneous energy from the formation of nitric acid
$Q_S$	= Extraneous energy from the formation of sulfuric acid
$Q_Z$	= Extraneous energy from ignition, combustion of the cotton thread, combustion aids. Set by default to 120 J, the value can be changed manually in the entry box <i>QExtran1</i> [J].
$Q_1$	= Extraneous energy from electrical ignition and from combustion of the cotton thread [J]
$S_{an}$	= Percentage of sulfur in the reference state analytical moist [%]
$S_{raw}$	= Percentage of sulfur in the supply state [%]
$\Delta T$	= Increase in temperature of the calorimeter system during a combustion experiment [K]

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