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IKA® Calorimeter System
C 2000 *basic*
C 2000 *control*



OPERATING INSTRUCTIONS *EN*
USA

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Wir erklären in alleiniger Verantwortung, dass dieses Produkt den Bestimmungen der Richtlinien 89 / 336 / EG und 2006 / 95 EG entspricht und mit folgenden Normen und normativen Dokumenten übereinstimmt: DIN EN IEC 61 010-1 und DIN EN IEC 61 326-1.

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We declare under our sole responsibility that this product corresponds to the regulations 89 / 336 / EEC and 2006 / 95 EEC and conforms with the standards or standardized documents: DIN EN IEC 61 010-1 and DIN EN IEC 61 326-1.

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Nous déclarons sous notre responsabilité que se prodiut est conforme aux réglementations 89 / 336 / CEE et 2006 / 95 CEE et en conformité avec les normes ou documents normalisés suivant: DIN EN IEC 61 010-1 et DIN EN IEC 61 326-1.

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Declaramos por nuestra responsabilidad propia que este producto corresponde a las directrices 89 / 336 / CEE y 2006 / 95 CEE y que cumple las normas o documentos normativos siguientes: DIN EN IEC 61 010-1 y DIN EN IEC 61 326-1.

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Dichiariamo, assumendone la piena responsabilità, che il prodotto è conforme alle seguenti direttive 89 / 336 / CCE e 2006 / 95 CCE, in accordo ai seguenti regolamenti e documenti: DIN EN IEC 61 010-1 e DIN EN IEC 61 326-1.

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Explanation of symbols



This symbol identifies information **that is of absolute importance to ensure your health and safety.** Failure to observe this information may be detrimental to your health or may result in injuries.



This symbol identifies information **that is of important to ensure problem-free technical operation of the device.** Failure to observe this information may result in damage to the calorimeter system.

This symbol identifies information that is important to ensure problem-free operation of calorimetric measurements and for working with the calorimeter system. Failure to observe this information may result in inaccurate measurement results.

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1 For your safety

In order to be able to use the appliance properly and safely, every user must first read the operating instructions and observe the safety instructions contained therein. Take care of these operating instructions and keep them in a place where they can be accessed by everyone.

intended purpose

The C 2000 calorimeter system may only be used to determine the gross calorific value of solid and liquid materials materials in accordance with DIN 51900, BS 1016 T5, ISO 1928, ASTM 5468, ASTM 5865 and ASTM 4809. For this purpose, use only original IKA® decomposition vessels C 5010, C 5012 and C 62. For further details please see the operating instructions of the decomposition vessels C 5010, 5012 and C 62.

operating requirements

The maximum amount of energy input into the decomposition vessel must not exceed **40000 J**. (Select the weight of the sample accordingly). The permissible operating pressure of **230 bar (23 Mpa)** must not be exceeded. The maximum permissible operating temperature must not exceed **50 °C**.
Do not fill the decomposition vessel too full of the sample. Only fill the decomposition vessel with oxygen up to a maximum pressure of **40 bar (4 Mpa)**. Monitor the adjusted pressure on the pressure reducer of your oxygen supply. Perform a leakage test before every combustion (please observe the operating instructions of the decomposition vessels C 5010, C 5012 and C 62, chapter ”Leakage test”).

explosive substances

Many substances tend to combust in an explosive manner (for example because of the formation of peroxide). This may cause the decomposition vessel to burst.
The standard decomposition vessels must not be used for examinations on samples that are capable of exploding. It is absolutely essential to use a special high-pressure decomposition vessel to contain the sample in these cases!

notes on the sample

Substances of which the combustion behavior is not known must be examined for their combustion behavior before combustion in the decomposition vessel C 5010, C 5012 or C 62 (danger of explosion). If you are burning **unknown samples**, leave the room or **keep a safe distance** between you and the calorimeter.
Benzoic acid must only be burned in the form of pellets! Combustible dust and powder must be compressed into pellets before combustion. Oven-dry dust and powder such as wood chips, hay, straw, etc. burn in an explosive manner! They must be moistened first! Readily combustible liquids with a low vapor pressure must not be come in direct contact with the cotton thread (for example tetramethyl dihydrogen disiloxan)!

combustion residue, auxiliary materials

In addition, toxic residues of combustion are possible in the form of gasses, ash or precipitates on the inner wall of the decomposition vessel, for example.



Observe the accident prevention requirements applicable to the activity and the work station. Wear personal safety equipment.

When handling combustion samples, combustion residues and auxiliary materials, the appropriate safety requirements must be observed. The following are examples of substances that may cause dangers:

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

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oxygen

When working with oxygen, observe the appropriate requirements.
Danger warning: As a compressed gas, oxygen promotes combustion, supports combustion intensively and may react violently with combustible substances.
Do not use any oil or grease!
Keep all gas lines and screw connections that carry oxygen free from grease.
Observe the accident prevention requirements applicable to the activity and the work station.
Close the main valve on the oxygen supply when work is complete.
Only carry out maintenance work when the system is depressurised.

using a crucible made of stainless steel

When using crucibles made of stainless steel, their condition should be carefully checked after every experiment.
A reduction in the thickness of the material may cause the crucible to burn and may damage the decomposition vessel. For reasons of safety, crucibles must not be used any more after a maximum of 25 combustion procedures.

specification of the decomposition vessel

The decomposition vessel is manufactured in accordance with the regulation for pressure vessels 97/ 23/ EEC. This can be recognized from the **CE symbol** with the identifying number of the testing station named. The decomposition vessel is a pressure device of Category III. The decomposition vessel has been subjected to an EC prototype test. The CE declaration of conformity represents our guarantee to you that this decomposition vessel complies with the pressure device described in the EC prototype test certificate. The decomposition vessel has been subjected to a pressure test at a test pressure of **330 bar** and a leakage test with oxygen at 30 bar.
Decomposition vessels are **experiment autoclaves** and must be tested by a **professionally trained person** each time before they are used.
An individual application is understood here to mean a series of experiments that are performed under roughly the same conditions in terms of pressure and temperature Experiment autoclaves must be operated in special chambers (C 2000, C 5000).

repeated tests

The decomposition vessel must be subject to repeated tests (internal tests and pressure tests) by a **person with professional training**. The intervals between tests must be determined by the operator based on experience, operating manner and the material used in the decomposition vessel.
The declaration of conformity loses its validity if mechanical modifications are made to the experiment autoclaves or if stability can no longer be guaranteed as a result of heavy corrosion (for example holes eaten in it by halogens).
The **threading** on the body of the decomposition vessel and cap screw in particular are subject to a high level of mechanical stress and must therefore be monitored regularly for wear and tear.
The condition of the seals must be checked for functionality and must be ensured by means of a leakage test (please observe the operating instructions of the decomposition vessels C 5010, C 5012 and C 62, chapter “Leakage test”).
Pressure tests and service tasks on the decomposition vessel must only be performed by **persons with professional training**.



We recommend that the decomposition vessel be sent into our factory for inspection and repairs if necessary after either 1000 experiments or after one year or, depending on the application, even sooner than this.

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definition of person with professional training

A person with professional training as defined in these operating instructions is someone

1. whose training, knowledge and experience gained through practical activities ensures that that person will perform the tests in a proper manner.
2. who is sufficiently reliable
3. who is not subject to any instructions in terms of testing activity
4. who is equipped with suitable testing equipment if necessary
5. who can provide suitable proof demonstrating compliance with the requirements listed in 1.

operating pressure containers

National regulations and laws for operating pressure containers must be observed! Anyone who operates a pressure container must maintain it in proper condition, must monitor it and perform necessary maintenance and repair tasks without delay, and must take measures appropriate for the circumstances to ensure safety.
A pressure container must not be operated if it exhibits defects that could endanger those working with it or third parties. You can obtain a copy of the pressure vessel regulation from Carl Heymann Verlag or Beuth Verlag.

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2 User notes

2.1 User notes on the Operating Instructions

In this chapter you can find out how to work through these Operating Instructions most effectively to be able to work reliably with the calorimeter system.



You must follow the instructions in Chapter 1 “For Your Safety”

work through Chapters 1 ... 12

Chapters 1 through 12 are designed for you to work through them in order.
Chapter 3, “Transportation, storage and set-up location” is relevant for the reliability of the system and to ensure a high degree of accuracy in measurements. Chapter 4 describes the system components and Chapter 5 contains the basic principles of calorimetry.

performing an experiment

The calorimeter system is ready to conduct measurements as soon as you have performed the procedures in Chapter 6 “Set-up and commissioning“ and Chapter 7 “Preparing and performing measurements“.
Evaluation of calibrations is described in Chapter 8.
Chapter 9 describes how to evaluate determinations of gross calorific value and how to calculate reference states. Chapter 10 explains the option of simulating experiments with the calorimeter system. In Chapter 11 you will find some important notes on system upkeep and maintenance. Chapter 12 contains a list of simple malfunctions and how to eliminate them.

You will find accessories, consumables and technical data on the device in Chapters 13 and 14, with the Index in Chapter 15.



The figures ①, ②, ③ etc. in the following chapters indicate actions that must always be carried out in the sequence given.

2.2 Warranty

In accordance with IKA warranty conditions, the warranty period is 12 months. For claims under the warranty please contact your local dealer. You may also send the machine direct to our works, enclosing the delivery invoice and giving reasons for the claim. You will be liable for freight costs.

The warranty does not cover wearing parts, nor does it apply to faults resulting from improper use or insufficient care and maintenance contrary to the instructions in this operating manual.

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2.3 Warranty claims and liability

Please read these Operating Instructions carefully and in their entirety. IKA® will only consider itself responsible for the safety, reliability and performance of the device if

- the device has been operated in accordance with the Operating Instructions
- only persons authorized by the manufacturer have performed repair and maintenance work on the device
- only original parts and original accessories have been used in repair work.

parts conducting electrical power

The calorimeter system may only be opened by a customer service center.
If service is required, we recommend that you contact our customer service department.

Furthermore, we refer you to the applicable safety conditions and accident requirements.
IKA® is not responsible for damages or costs resulting from accident, improper use of the device or impermissible modifications, repairs or renovations.

2.4 System properties

The C 2000 calorimeter system is routinely used for determination of gross calorific values of solid and liquid substances. The accessories of the system ensure customized adaptation to laboratory tasks (see Chapter 13).

The system is characterized by the following features:

- Relieves you of routine tasks through automated measurement procedure
- Integrated oxygen filling
- Automatic detection of decomposition vessel (DV)
- Operation without the cooling unit: Connection to a water faucet; temperature range 12°C - 28°C; water consumption per measurement about 4 l; water pressure 1 bar to max. 1.5 bar; at higher pressures, use C 25.
- Operation with active cooling unit (for example IKA® KV 500, optional)
- Measurement and calculation of gross calorific value according to DIN 51900, ISO 1928, ASTM D240, ASTM D4809, ASTM D5865, ASTM D1989, ASTM D5468, ASTM E711
- Calculation of net calorific value according to DIN 51900, ASTM D240, ASTM D4809, ASTM D5865, ASTM D1989, ASTM D5468, ASTM E711
- Measurement range: max. 40,000 J
  (This corresponds to an increase in temperature in the inner vessel of about 5 K)

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- Operating mode based on the isoperibolic or dynamic principle
  At 25 °C or 30 °C (the beginning temperature of the water in the inner vessel) depending on the cooling water temperature (see Chapter 4.3).

Cooling water temperature	Operating mode
12 °C to 23 °C	isoperibolic 25 °C
12 °C to 23 °C	dynamic 25 °C
23 °C to 28 °C	isoperibolic 30 °C
23 °C to 28 °C	dynamic 30 °C

- A monitor and external keyboard can be connected
- PC-operation of one or several calorimeters (CalWin® software)
- A sample rack can be connected
- A printer can be connected

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3 Transportation, storage and set-up location

3.1 Transportation and storage requirements



During transportation and storage, the system must be protected against mechanical jarring, vibrations, accumulations of dust and corrosive ambient air. Care should also be taken so that the relative humidity does not exceed 80%.
The device must be completely emptied before being stored and transported.

In case of repair the device has to be cleaned and free from any materials which may constitute a health hazard.

If you require servicing, return the appliance in its original packaging. Storage packaging is not sufficient. Please also use suitable transport packaging.

3.2 Set-up location



When setting up the device, make certain that the applicable state regulations for operating pressurized containers are adhered to.
A constant ambient temperature is an important prerequisite in ensuring the high measurement precision of the system. The following conditions must therefore be maintained at the set-up location:

- No direct sunlight.
- No drafts (for example next to windows, doors, air conditioner outlets).
- The device must be at a sufficient distance from heater and other sources of heat.
- The minimum distance between the wall and the rear side of the device must be at least 25 cm.
- The system must not have laboratory hardware such as shelves, wire ducts, ring lines, etc. installed above it.
- The room temperature must be in the range of 20 °C ... 25 °C (constant).
- The system must be set up on a horizontal surface.

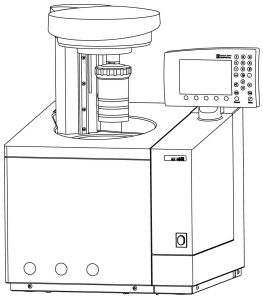
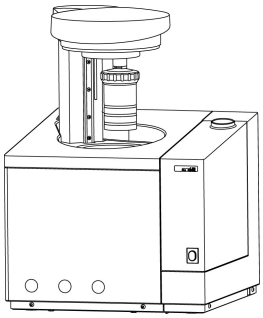
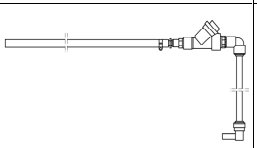
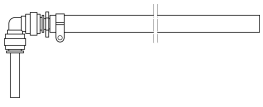
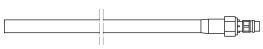
A power supply corresponding to the rating plates of the system components and the oxygen supply (99.95% pure oxygen, quality 3.5; pressure 30 bar, with a pressure display) must be available to operate the system at the set-up location. A shut-off valve for the oxygen supply must be installed. Observe the instructions on handling oxygen given in Section 1 "For your safety”.

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3.3 Unpacking

Please unpack the system components carefully and check closely for any damage. It is important to recognize any damage that occurred during shipping while you are unpacking. If damage has occurred, you will need to make a written assessment of the damage and send it to us by mail, rail or special delivery.

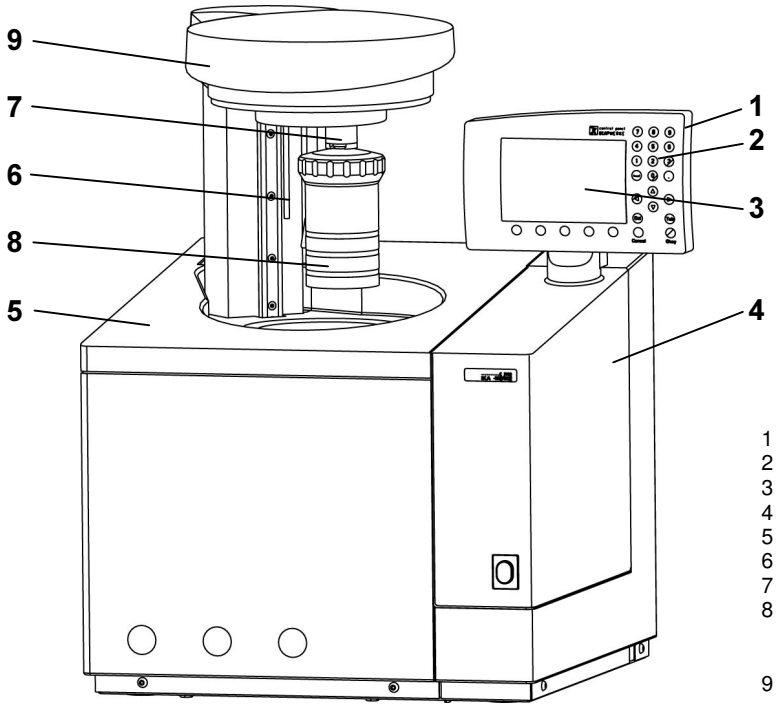
3.4 Scope of delivery

	C 2000 *basic*	C 2000 *control*	Beschreibung
	1x		Grundgerät Beilegeset Netzanschlusskabel Betriebsanleitung
		1x	Grundgerät C 5040 CalWin Software Beilegeset Netzanschlusskabel Betriebsanleitung
	1x	1x	O_2-Druckschlauch Länge: 2 m Anschlüsse: 1 x M8x1, SW 10 1 x ¼“ , SW 17
	1x	1x	Zuflussleitung Wasser Länge: 1,5 m
	1x	1x	Rückflussleitung Wasser Länge: 1,5 m
	1x	1x	Entleerschlauch Wasser Länge: 1m

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4 Description of the system components

4.1 C 2000 *basic*



C 2000 *basic*

1	Control panel
2	Keyboard
3	Display
4	Electronics unit
5	Measuring cell
6	Temperature sensor
7	Oxygen filling device
8	Decomposition vessel (Accessory, please order seperately)
9	Measuring cell cover

The control panel is the operating and display unit for the system. Commands and experiment parameters are entered in dialog mode via the keyboard; visual control takes place through the display.

All phases of the measurement procedure are controlled and monitored during an experiment The display shows the current system states and experiment data.
Any malfunctions are also displayed as a line of text.
The results of the experiment are stored together with the experiment parameters and may be printed out whenever desired.
Communication with external peripheral devices (for example printers, analytical scale, sample rack, PC, and keyboard) via the interfaces in the electronics unit is also shown on the display.

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The calorimetric experiment, which entails combustion of the fuel sample under precisely defined conditions, takes place in the measuring cell.
The following components are located in the measuring cell for this purpose:

- Inner vessel with insulating water jacket (outer vessel)
- Stirrer to ensure uniform distribution of heat in the inner vessel
- Water circuit with a heater element for controlled equalization of the system temperature and automatic filling of the inner vessel
- Temperature sensor to record measurement values
- Oxygen filling apparatus for the decomposition vessel

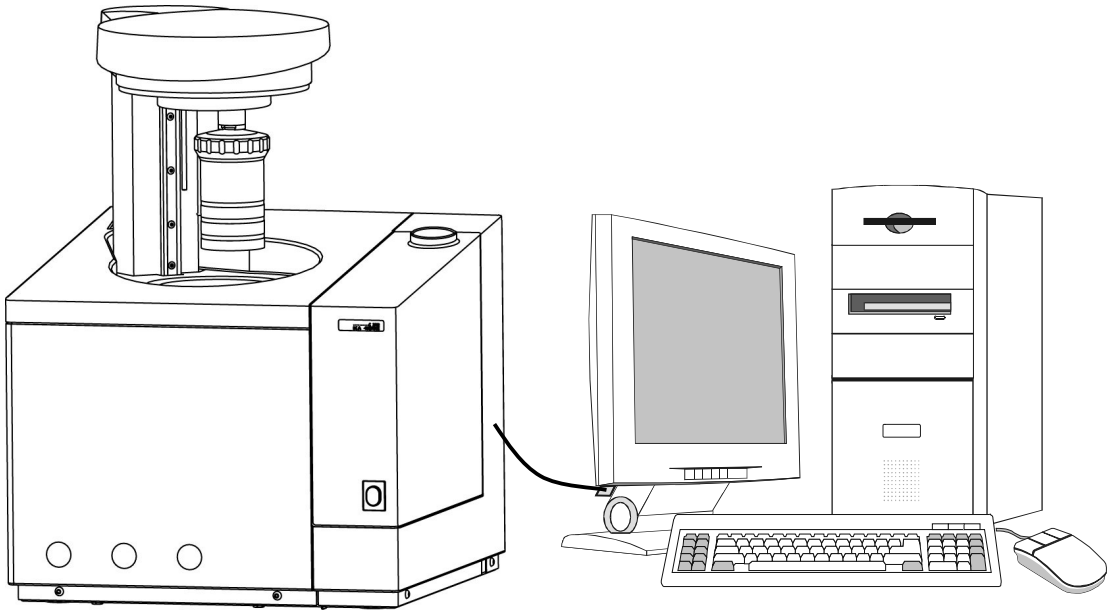
The following processes occur in the measuring cell during an experiment:

experiment procedure

- The measuring cell cover closes automatically and the decomposition vessel is immersed with the fuel sample in the inner vessel.
- Pure oxygen flows into the decomposition vessel through the oxygen filling apparatus until the pressure set by the user has been reached (30 bar).
- Water from an external pressure source (a water faucet, laboratory thermostat or cooler) flows into the device and is heated up to the working temperature (optionally 25 °C / 30 °C).
- The inner vessel is filled with temperature-controlled water (at working temperature).
- A stirrer ensures there will be uniform distribution of heat in the water within the inner vessel.
- The temperature of the water in the insulating outer vessel is controlled.
- Die fuel sample is ignited electrically with the ignition device.
- The increase in temperature of the water in the inner vessel is measured and the gross calorific value is determined.
- At the end of the experiment the inner vessel is emptied and the water in the circuit is cooled off by an external cooler unit or is diverted to the outlet.
- The measuring cell cover opens and the decomposition vessel can be removed.
- Pressure must be released from the decomposition vessel manually (venting button C 5010.6 or venting station C 5030).

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4.2 C 2000 *control*



C 2000 *control*

A measuring cell or control panel (control) is not a unit capable of functionality unless it is combined with a PC and CalWin® control software.

The phases of the measuring process are controlled and monitored by means of the PC software.
The visualization appears on the monitor of the corresponding PC and data input can take place via the interfaces of the peripheral devices or the PC keyboard.
The structure of the measuring cell and the experiment process are similar to devices with a control panel (basic).

If you are using a multiple interface card (optional) up to eight measuring cells can be controlled with this configuration through a single PC.

For more information on operating the C 2000 control, please refer to the Operating Instructions for the C 5040 CalWin® software package.

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4.3 Cooling

The C 2000 calorimeter does not have its own cooler unit.

The system can optionally be operated with a standard commercial cooler or thermostat with water filling (for example the IKA® KV 500) or can be connected to a cooling water line.
For thermostat operating mode, only water with a stabilizer additive (for example Aqua-Pro) should be used.
Do not use distilled or deionized water.

The control range is limited in terms of control quality. The cooling water temperature should therefore not fall below 12°C. The upper temperature limit of the cooling water is 23°C in 25°C mode or 28°C in 30°C mode.
If the temperature falls short of or exceeds the cooling water temperature range, a malfunction message to this effect appears on the display (see Section 12.1).

An internal safety valve is used to adjust the water flow rate on the KV 500 cooling water supply to a value between 60 and 70 litres per hour.
If the device is connected to a cooling water line, the pressure on the input side must not exceed 1.5 bar.
If necessary, you should ensure this by placing a pressure control valve (for example the IKA C 25) far enough forward in the circuit.

amount of filling thermostat

Another important characteristic value for connecting a cooler/thermostat is the switch-off level (the minimum volume).
The inner vessel of the calorimeter is filled with about 1.5 liters of water for measurements. It must be possible to provide this quantity from the cooler/thermostat.

The temperature of the cooling water (on the intake side) determines the working temperature, i. e. the starting temperature of the water in the inner vessel of the calorimeter.
If the cooling water temperature is less than or equal to 23 °C, the calorimeter will work in the 25 °C mode.
For a cooling water temperature greater than 23°C, measurements can now take place in the 30 °C mode. On this subject, see also Section 6.4, Turning on the system.

The calorimeter automatically tests the cooling water temperature each time after the device system is turned on. You can also optionally activate an automatic verification before every measurement.
The system also offers you the possibility of querying the cooling water temperature manually (see Section 11.1, “Maintenance menu”).

The cooling water temperature has no effect on the “isoperibolic” or “dynamic” modes.

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5 Calorimetric measurements

5.1 Determination of gross calorific value

Combustion processes take place in a calorimeter under defined conditions. For this purpose, the decomposition vessel is coated with a weighed out quantity of fuel sample, the fuel sample is ignited, and the increase in temperature of 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 within the inner vessel of the measuring cell

experiment conditions

To optimize the combustion process, the decomposition vessel is filled with pure oxygen (99.95%). The maximum level of pressure of the oxygen atmosphere in the decomposition vessel is 30 bar.
The exact determination of the gross calorific value of a substance requires that the combustion proceed under precisely defined conditions. The applicable standards are based on the following assumptions:

- Depending on the mode that is set, the temperature of the fuel before the combustion is 25 °C or 30 °C.
- The water contained in the fuel before the combustion and the water that is formed when compounds comprising the fuel containing hydrogen undergo combustion is present in a liquid state.
- No oxidation of the atmospheric nitrogen has taken place.
- The gaseous products after combustion consist of oxygen, nitrogen, carbon dioxide and sulfur dioxide.
- Solid substances may be formed (for example ash).

Often, however, the combustion products that form the basis of the standards are not the only products that are formed. In such cases, an analysis of the fuel sample and the products of combustion are necessary to provide data for a correction calculation. The standard gross calorific value is then determined from the measured gross calorific value and the analysis data.

gross calorific value Ho

The gross calorific value Ho is formed as the quotient of the amount of heat liberated upon total combustion of a solid or liquid fuel and the weight of the fuel sample. The compounds comprising the fuel that contain water must be present in liquid state after the combustion.

heat value Hu

The heat value Hu is equal to the gross calorific value less the energy of condensation of the water that was contained in the fuel and was formed by the combustion. The heat value is the more important quantity for technical purposes, since in all important technical applications, the heat value is the only quantity that can be evaluated in terms of energy.

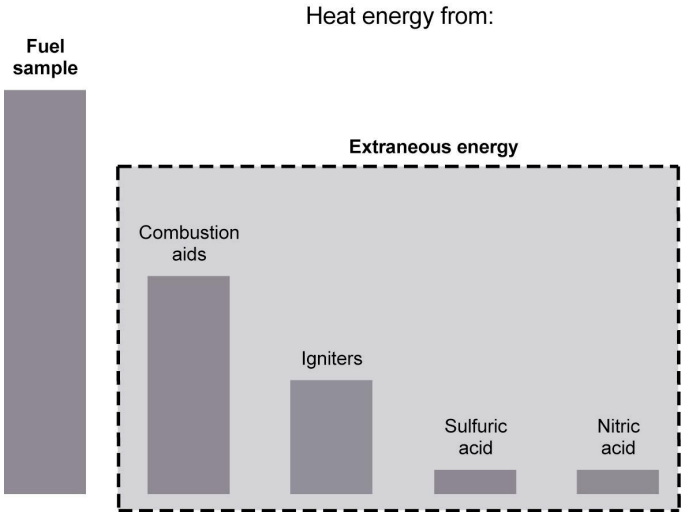
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For information on the fundamental principles of calculation for gross calorific value and heat value, please refer to the applicable standards (for example: DIN 51 900; ASTM D 240; ASTM D 1989).

5.2 Corrections

As determined by the system, during a combustion experiment, not only is heat generated from the combustion of the sample, but heat also occurs in the form of extraneous energy.
This may fluctuate considerably in proportion to the heat energy of the fuel sample.

combustion heat and extraneous energy



The combustion heat of the cotton threat that ignites the sample and the electrical ignition energy would result in distorted values of the measurement. This effect is taken into consideration in the calculation with a correction value.

combustion aids

Substances with low flammability or substances that burn poorly undergo combustion in combination with combustion aids. The combustion aid is first weighed and then added to the crucible with the sample. From the weight of the combustion aid and its known specific gross calorific value, it is then possible to determine the quantity of heat that is introduced thereby. The result of the experiment must be corrected by this amount of heat.

C 14 disposable crucible

The C 14 disposable crucible is a combustible crucible that can be used instead of a traditional crucible. The disposable crucible undergoes total combustion with no residue. If a disposable crucible is used, there is then no need for a cotton thread. The crucible is in direct contact and is ignited by the fixed ignition wire of the decomposition vessel.
The purity of the material used in the disposable crucible prevents any chemical contamination of the sample material from occurring (no blind values).
Decomposition vessels in which the disposable crucible is used must be retrofitted with an additional part (holding surface C 5010.4, see accessories). The sample is weighed into the disposable crucible as usual. In most cases, no additional combustion aid is necessary because the disposable crucible itself acts as the combustion aid.

The disposable crucible cannot be used in combination with the sample rack.

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acid correction

Almost all substances to be analyzed contain sulfur and nitrogen. Under the conditions that prevail during calorimetric measurements, sulfur and nitrogen undergo combustion and form SO_2, SO_3 and NO_x. Sulfuric and nitric acid arise in combination with the water resulting from combustion and humidity. Heat of solution is also generated. To obtain the standard gross calorific value, the effect of the heat of solution on the gross calorific value is corrected.

To obtain a defined final status and to record all acids quantitatively, about 5 ml of distilled water or another suitable solution is placed in the decomposition vessel. With this absorption fluid and the combustion water, the combustion gasses form acids. After the combustion, the decomposition vessel is thoroughly washed with distilled water so as to collect the condensate that has settled on the inner wall of the vessel as well. The solution obtained in this manner can now be examined with a suitable peripheral detection device for aqueous decomposition into the respective acid content.
For more information on this subject, please contact IKA® or your nearest authorized dealer.

5.3 Complete combustion

To correctly determine the gross calorific value, it is of fundamental importance that the sample undergoes complete combustion. After each experiment, the crucible and all solid residues must be examined for any signs that combustion was not complete.

solid substances

Normally, solid substances can be burned directly in powder form. Rapidly burning substances (for example benzoic acid) must not undergo combustion in loose form. These substances tend to squirt, and it would therefore not be possible to ensure complete combustion. In addition, it is possible the decomposition vessel could be damaged. Such substances are pressed into tablets before combustion with a special press (C 21 pelleting press, see accessories).

substances with low flammability

Substances with low flammability (substances with a high content of mineral matter and low-calorific substances) will often undergo complete combustion only with the aid of combustion capsules or combustion bags. It is also possible to use liquid combustion aids such as paraffin oil, for example.

liquid, easily 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 undergo combustion together with the capsules.

The combustion aids as well (for example the cotton thread) must undergo complete combustion. If unburned residues are left over, the experiment must be repeated.

halogens

Substances rich in halogen may cause corrosion to appear on the decomposition vessel. The C 5012 decomposition vessel should be used for these applications.

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5.4 Calibration

To ensure precise, reproducible results of the measurement, the calorimeter system is calibrated after it is initially placed in service, after service jobs, after parts are replaced and at specific time intervals. During calibration, the heat capacity of the calorimeter system is redetermined.

Regular calibration is absolutely essential to obtain accurate measurements. The system must be calibrated in the operating mode that is used (isoperibolic 25 °C or isoperibolic 30 °C or dynamic 25 °C or dynamic 30 °C).

For this purpose, a specific quantity of a reference substance undergoes combustion in the decomposition vessel under the conditions of the experiment. Since the gross calorific value of the reference substance is known, it is possible after combustion has occurred to calculate the heat capacity based on the increase in temperature of the calorimeter system.

The reference substance for calorimetry on an international level is benzoic acid of the National Bureau of Standards (NBS Standard Sample 39) with a guaranteed gross calorific value.

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

For more information on calibration, we refer you to the applicable standards.

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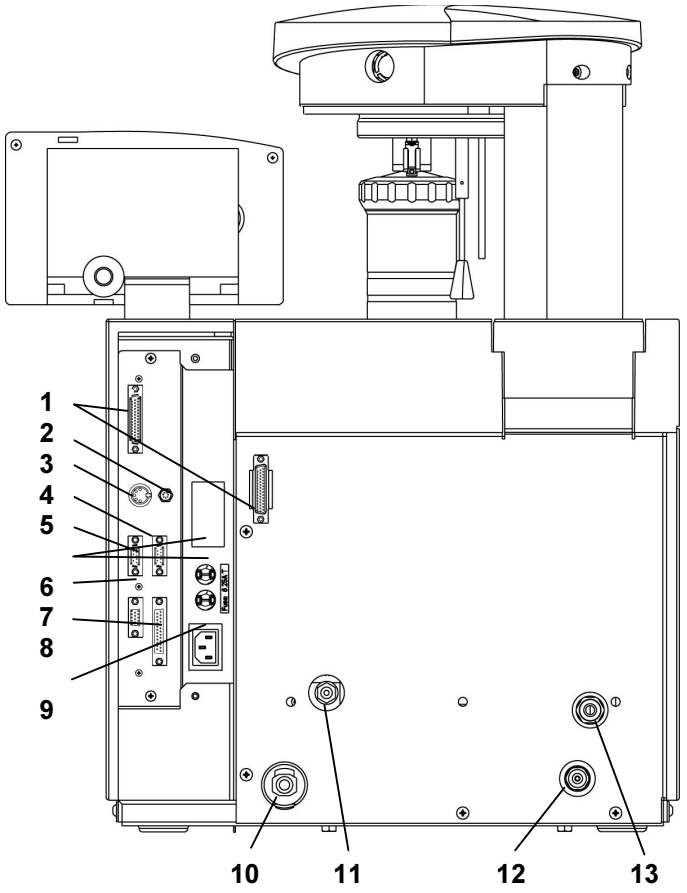
6 Set-up and commissioning

You have unpacked the components of the C 2000 calorimeter system and they are located where they will be set up (see Chapter 3, Section 3.2, “Set-up location”).

Now perform the following steps:

6.1 Set-up

All connections for the supply lines and peripheral devices are located on the rear side of the device.



1	Connecting cable connection
2	Sample rack connection
3	Keyboard connection
4	Scale connection
5	PC connection
6	Fuses
7	Monitor connection
8	Printer connection
9	Mains power connection
10	Water drain adapter
11	Oxygen connection adapter
12	Water outlet adapter
13	Water intake adapter

①

Connecting the oxygen supply line

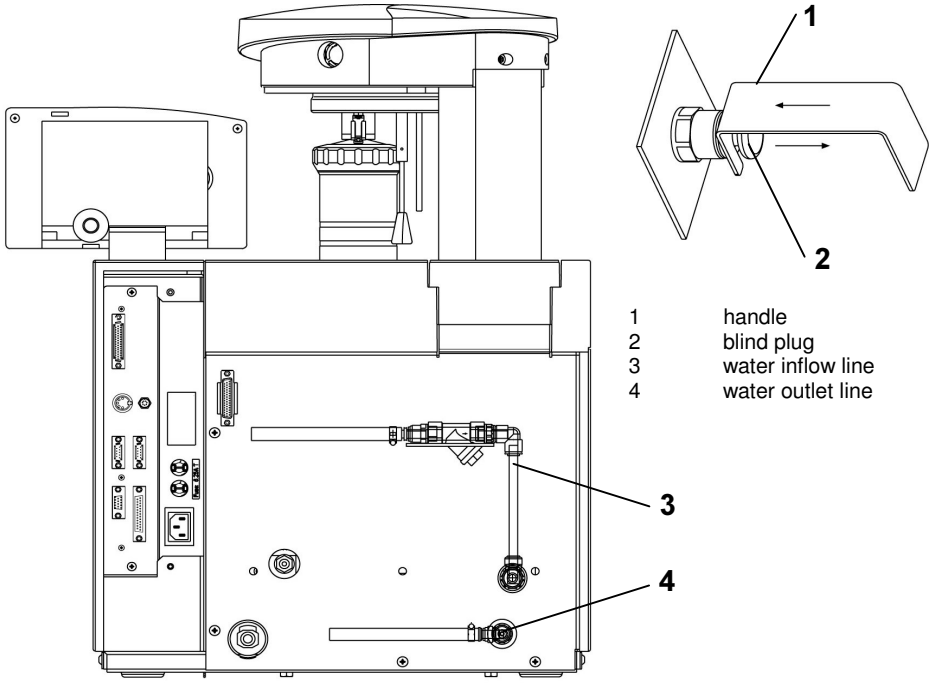
Screw the pressure hose (O_2 line) with the M8x1 cap screw onto the oxygen connection adapter of the measuring cell (fork wrench SW 10, included with delivery) and connect the hose to your oxygen supply on the laboratory side. On the pressure side, the hose is designed with an R ¼” screw thread. A ¼” NPT adapter piece is delivered with all hoses. In some circumstances this must be used to replace the ¼” screwing piece (users in the USA).

Please refer to chapter 6.2 for details how to connect to oxygen supplies.

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②

Connecting the water supply



1 handle
2 blind plug
3 water inflow line
4 water outlet line

Removing the blind plugs

With the aid of the handle (1) (included with delivery of the decomposition vessel), remove the blind plug (2) from the water connections of the measuring cell. Residual water may emerge when the blind plug is removed. This should be captured with an absorbent underlay.

Connect the inflow line (3) to the water intake adapter. The built-in dirt trap should be placed in the holder designed for it.
Now connect the water outlet line (4) to the water outlet adapter.
The lines are correctly connected if you overcome a distinct resistance and plug the line in all the way to the stopper.
Check that the connection is correct by pulling in the opposite direction.



The water inflow and outlet lines (each 1.5 m long) included in the scope of supply must neither be extended nor replaced with longer lines!

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③

Operation on the water line

The cooling water temperature must fall within the range from 12 °C to 28 °C.

The water pressure on your line must not exceed a level of 1.5 bar!

If necessary (if the pressure is higher or if there are fluctuations in pressure) install a suitable pressure control valve (for example the IKA® C 25).
The IKA® C 25 pressure control valve is set to a pre-pressure of 1 - 1.5 bar. Verify this value after the calorimeter is ready for operation. To do this, water must be flowing through the calorimeter! In the maintenance menu, activate the menu item *Cooling water* and check the pre-pressure on the pressure gauge (see Section 6.4 and 11).

Now connect the water inflow line to the connection of the cold water valve.
Secure the connection with the hose clamp (included with the system).
Secure the water outlet line in place in the laboratory wash basin.
Open the fitting valve completely.

If you will be using the device in unsupervised operation, IKA® recommends you use a standard commercial “water stop valve” in the water inflow line.

④

Operation with thermostat

You can place the thermostat wherever convenient, but the above rule regarding the lengths of the water inflow and outlet lines must be observed.
Connect the water inflow line of the calorimeter with the output of the thermostat and connect the water outlet line with the input.
Secure both hoses so that they will not be unintentionally loosened with the hose clamps provided.

Fill the thermostat with water from the line and add Aqua-Pro (to prevent the formation of algae) according to the metering instructions.
Keep a quantity of tap water in reserve, because refilling will be required during commissioning.



Do not use distilled water!

Turn the unit on.

⑤

General notes on cooling water supply

As was already mentioned in Section 4.3, the temperature of the cooling water determines the working temperature of the calorimeter.
When working on a cold water line, the temperature is often unknown or subject to great daily fluctuations.

Below a temperature of the cooling water of 23 °C it is only possible to measure at a working temperature of 25 °C, and above it at a working temperature of 30 °C.
The calorimeter tests the water connection and the current water line temperature every time it is turned on, and (optionally adjustable) before every measurement (see Chapter 6.5 “Configuring the system/System settings/ReInit”).

The following approximate figures apply if you are working with a thermostat or cooling unit:

Cooling water temperature 18 °C to 20 °C:	Working temperature 25 °C
Cooling water temperature 23 °C to 25 °C:	Working temperature 30 °C

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The current line water temperature can be queried manually if the water supply is open through the *Cooling water* menu item (see Section 11.1 “Maintenance menu”) If the line water temperature is subject to large fluctuations, we recommend that you work with a cooler unit.

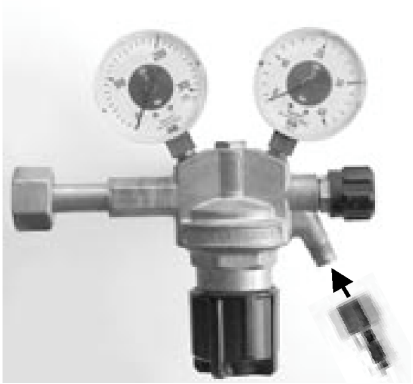
⑥

Connecting the mains power line
Check the voltage information on the rating plate of the calorimeter with the data for your power supply network.

Connect the mains line with the calorimeter or with the power source.

6.2 Connection oxygen supply

Installation
The 2 m long O_2 hose supplied with the system is designed for a maximum pressure of 40 bar at room temperature. This can either be fed directly to the oxygen cylinder or to another oxygen connection point with a pressure reducer (smallest permitted bending radius 80 mm).



The C 29 reducer valve (accessory) for the oxygen cylinder features an R ¼ inch thread for connecting the pressure hose.

The pressure hose is supplied with an adapter for American pressure reducers.

⑦

Make sure that the oxygen supply line is connected to the C 2000 calorimeter as described under Point ③. Then connect the O_2 line to 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).

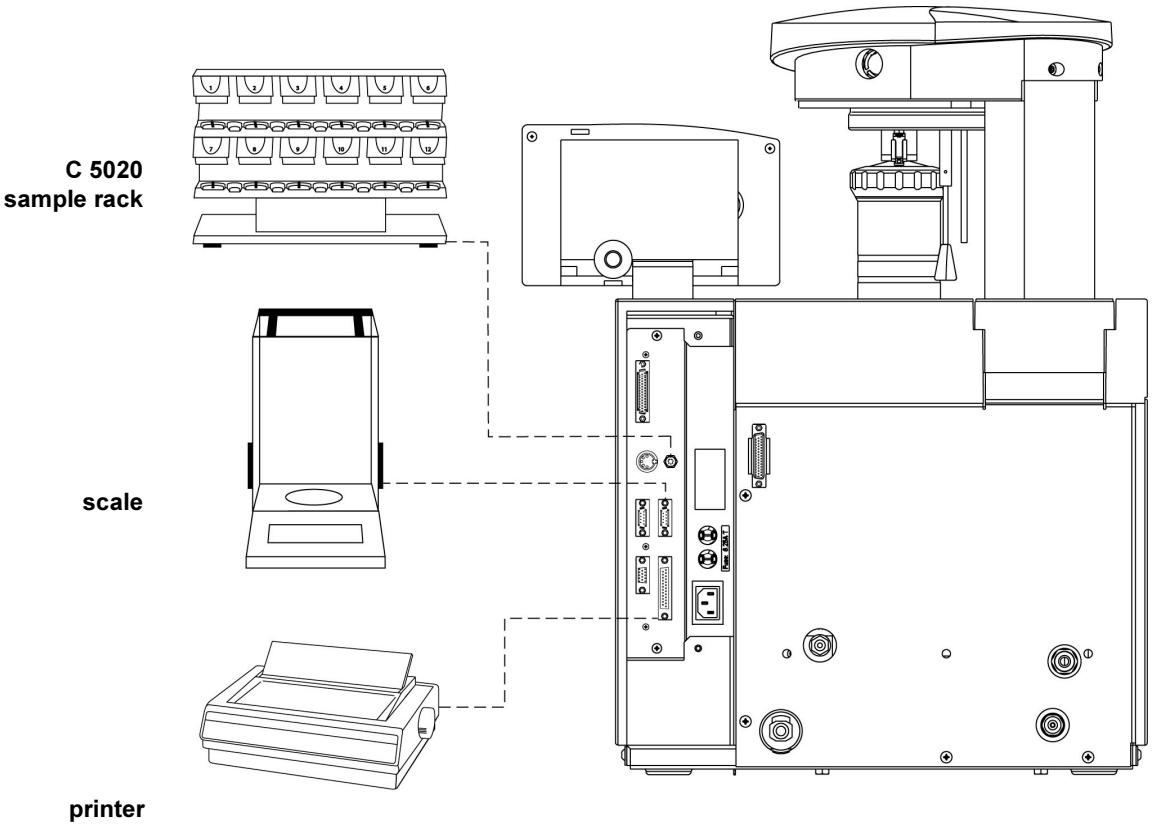
Keep all gas lines and screw connections that carry oxygen free from grease.
Observe the accident prevention requirements applicable to the activity and the work station. Wear personal safety equipment.
Close the main valve on the oxygen supply when work is complete.
Only carry out maintenance work when the system is depressurised.

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6.3 Connecting peripheral devices

While the peripheral devices are being connected, they and the calorimeter must be turned off on the mains power switch.

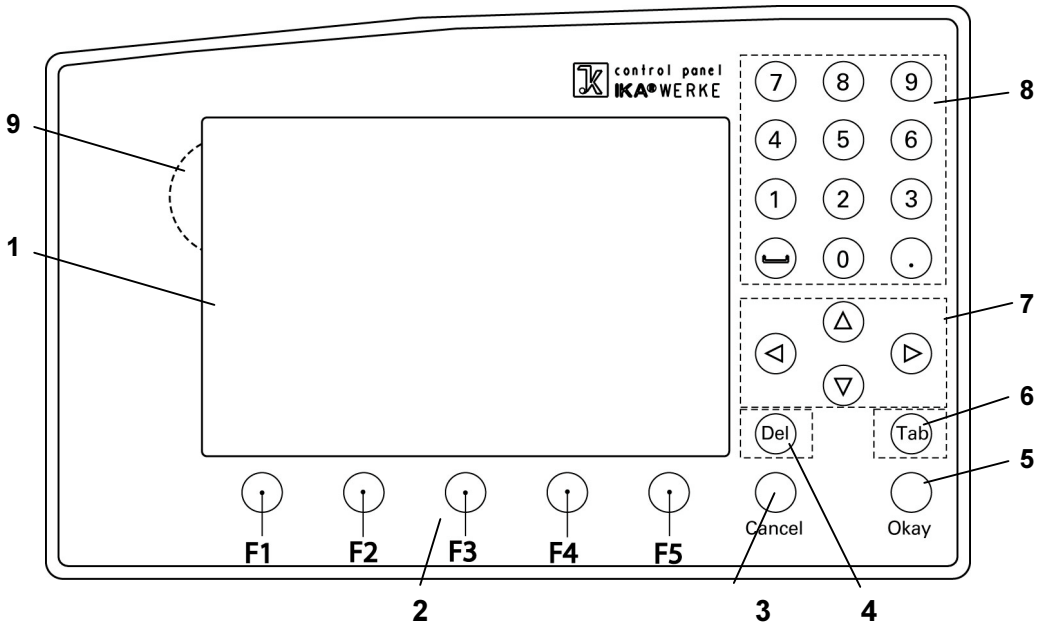
If a sample rack, electronic scale or a printer were delivered with the calorimeter system, they should now be connected.
A screen, keyboard and/or PC can also be connected now.
The connection sockets are located on the rear wall of the device. When connecting the devices, please note the labeling on the connection wires.



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6.4 Display and operating elements

The control console is equipped with the following elements:



1. ***Display:*** used to display system data, experiment data and experiment data menus and dialog boxes for data entry.

2. ***Function keys:*** the assignment of the function keys depends on the current operating status of the system. *F1* calls up a context-related help system. The footer of the display indicates the current assignment of the function key.

3. ***Cancel-key:*** the *Cancel* function is active in the menu and dialog windows. *Cancel* causes you 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 in a dialog box, for example the weight of the combustion sample, you can delete the character to the left of the cursor's position with the *Del* key. As a second function, the menu bar on the upper edge of the screen can be opened outside of a dialog box by pressing the *Del* key.

5. ***OK key:*** with the *OK* key you can activate menu items and close or confirm dialog boxes. In addition, *OK* causes the system to accept data that has been entered in a dialog box.

6. ***Tab key:*** *Tab* moves the cursor in a dialog box from one entry box to another.

7. ***Left, Right, Up and Down arrow key:*** the arrow keys move the cursor within the input lines, menu windows, tables and protocols.

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8. ***Numeric keypad:*** with these keys you can enter number, decimal points and spaces. Outside of a dialog box, you can use the point key to open or close an additional information window for service purposes. The content of this window can be printed out with the space bar ␣. You can use button *1* to go to the maintenance menu when there is no measurement running. Pressing button *2* feeds a page if a printer is connected.

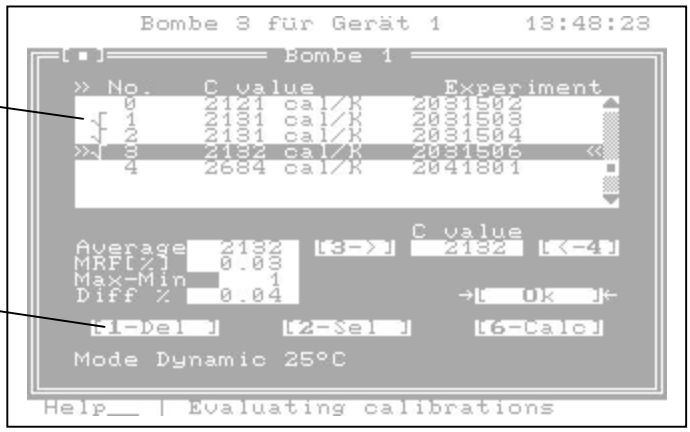
9. ***Contrast control:*** used to control the display contrast.
   Lock screw: by loosening the lock screw you can change the angle of inclination of the display. To lock it in place, the screw must be tightened again.

10. Various dialog elements can be selected on the display within a dialog window.

    The following dialog elements are available:

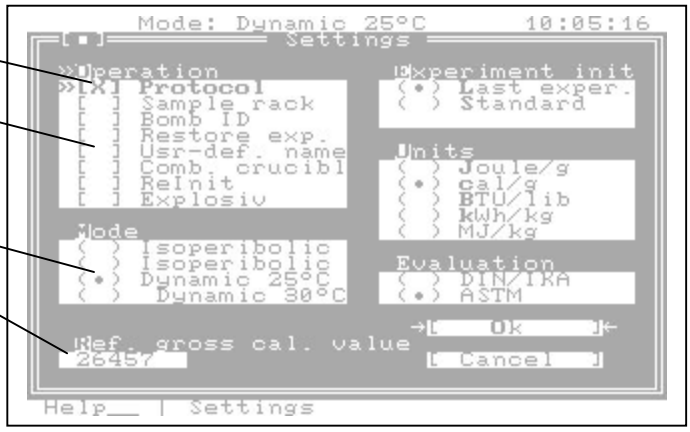
    - input line
    - button
    - simple table
    - selection table
    - options table
    - display elements (cannot be operated)

example of a dialog box



1 table
2 button

example of a dialog box



1 active element
2 options table
3 selection table
4 input line

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active dialog element

All dialog elements are labeled. The labeling is marked by a "**»**" sign for all active dialog elements. Each element can be made active by repeatedly pressing *Tab* for the active dialog element. Only the active dialog element can be operated (exception: Button).

input line

Digits and a decimal point can be entered in an active input line. The last character that was entered can be deleted with the *Del* character. Some input lines offer the possibility of selecting letters and additional characters with the arrow keys from a table of characters that is displayed and then accepting the character into the input text with the "**.**" key.
Tab ends input and activates the next dialog element.
OK ends input and closes the window.

You can select the lines of an active table (including the selection and option table with the *Up arrow/Down arrow* keys. In the case of a selection table, the corresponding option is designated by (●).
For an option table, the option can activated in the selected line (identified by [x]) or can be deactivated again (identified by [ ] with the space bar).
Tab ends 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 can be switched with the *OK* key. If a table is active and the button is labeled with a number, it can also be switched with the corresponding number key.

dialog box

Almost every dialog box has the *OK* and *Cancel* button.
If the *OK* button is marked with → and ← it can also be switched on and off from an active table or input line with the *OK* key. This causes the dialog box to close and the entries and settings in it to be accepted. A button labeled with *Cancel* can always be switched with the *Cancel* key and also causes the window to close, but without the entries and settings being accepted. Actions previously initiated with other switches will not be undone in any case.

A repeat function is provided for all keys.
If the key is held down for more than one second, the character in question is repeated.

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6.5 Turning on the system

When you turn on the calorimeter, the splash screen first appears (the cover of the measuring cell opens automatically and the stirrer starts up for a few seconds).

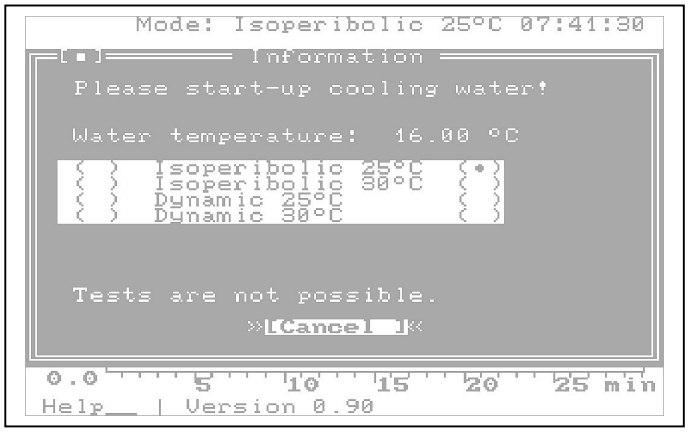


splash screen

The current assignment of the function keys appears in the footer, and the current system message appears in the header.

Make certain that the water connection for the device has been opened and if there is an additional cooler unit that it is turned on.

After you confirm the splash screen (or if you do not then automatically after 10 seconds), the system check is started. The system check verifies whether the cooling water is flowing through the calorimeter (at least 30 seconds).

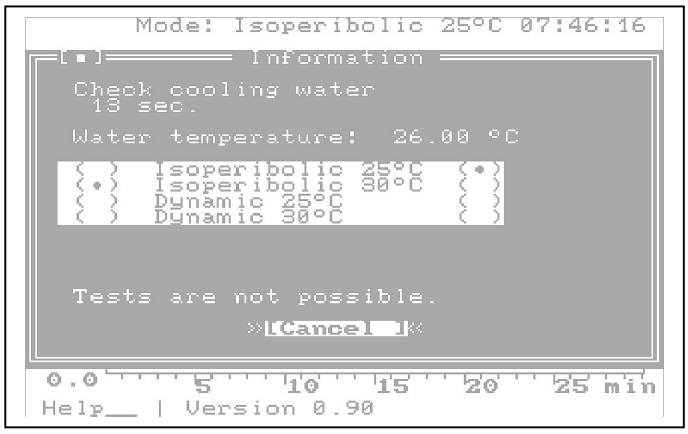


verification of flowthrough

Next the system tests whether the measured cooling water temperature is suitable for stable operation in the selected operating mode (at least 150 seconds). During this time, the measured cooling water temperature that is suitable for the mode is displayed in the left selection table, while the right selection table designates the last operating mode that was used.

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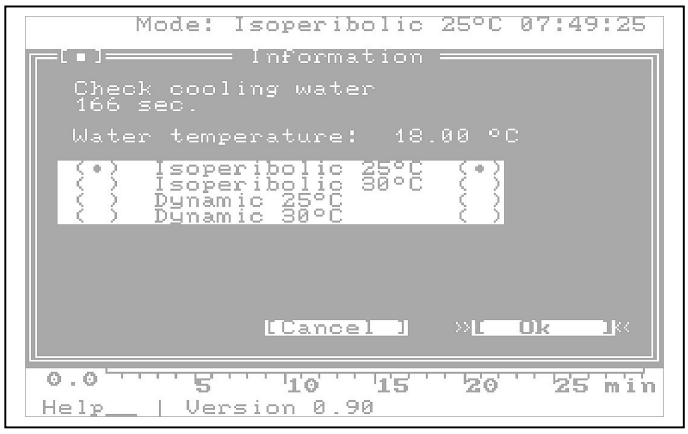
verification of cooling water temperature



When the system is initially placed in service, the vessel system and hose system of the calorimeter are filled with water during this phase. If you are working with a cooler unit, you must now compensate the fill level of the storage tank.
Later losses of water (for example through condensation) must also be compensated for during this operating state of the calorimeter). No measurements may be running and the inner vessel must not be filled.
When using the cooler KV 500, please take care that the bath is filled to the upper level (25 mm below the upper edge of the bath). This ensures that the maximum quantity can be withdrawn.

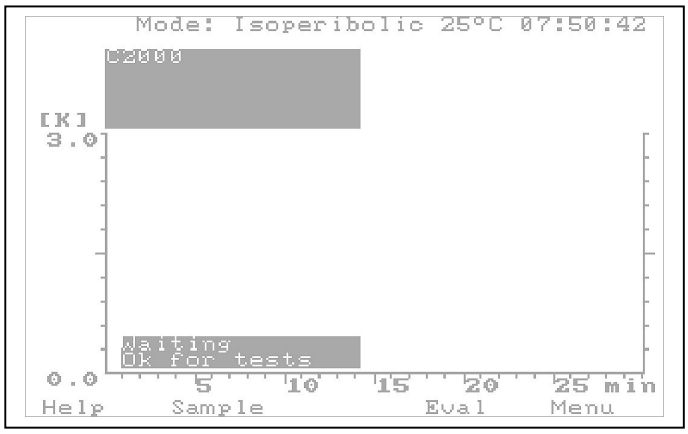
After the system check has been successfully completed, the *OK* button appears, with which the calorimeter can be made ready for operation.

system check successful



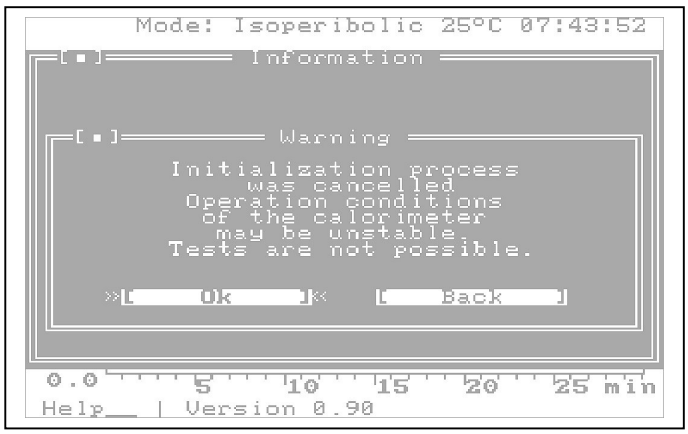
After you confirm with *OK*, the device is ready for operation.

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ready for operation

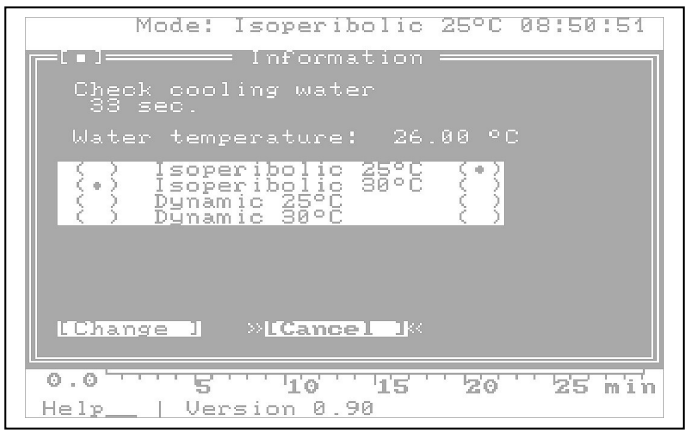
If the system check was interrupted with *Cancel*, no measurements are possible. It is also not possible to reach the maintenance menu. Canceling is useful if only administrative tasks (library, simulation, or evaluation) are to be performed on the calorimeter.



interrupt system check

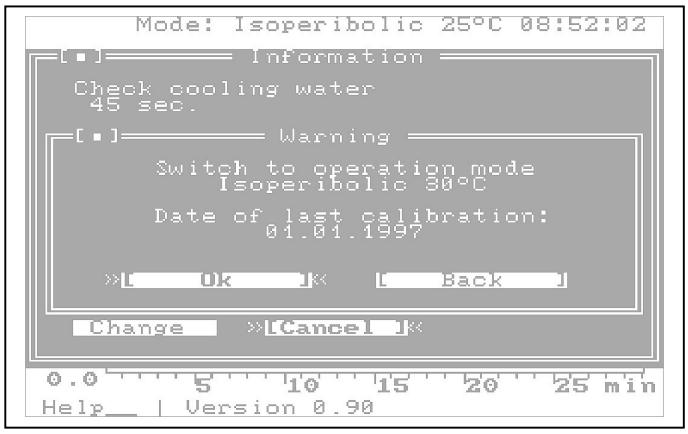
The interrupt is confirmed with *OK*; the system check is continued with *Back*.

If the system check still does not permit operation in the set operating mode after 3 minutes, it is possible to adjust the system to the appropriate mode corresponding to the measured cooling water temperature with the *Change* button.



In this case a reference appears to the effect that there may be no valid calibrations available for the selected operating mode.

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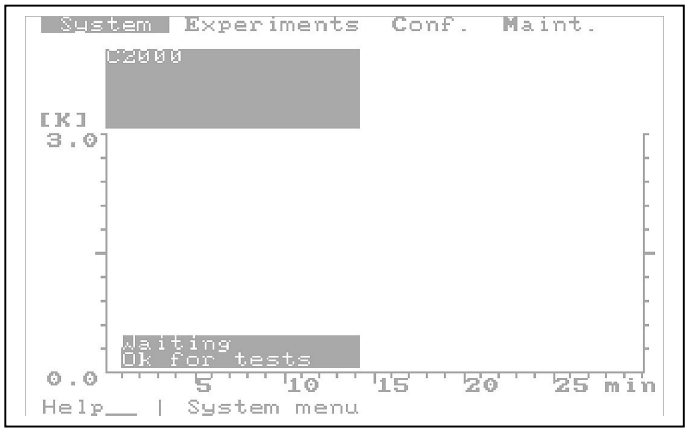


confirmation of the switch to another operating mode

The message is confirmed with *OK*; and the system is made ready for operation. The system check can be continued with *Back*.

6.6 Configuring the system

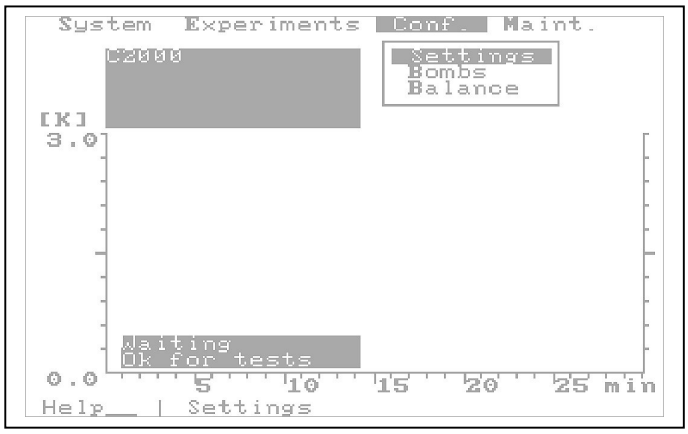
From the main screen you can reach all menu and dialog windows. You can reach part with the menu line that is called with the *Menu* key or the *Del* key.



main screen with activated menu line

With the arrow keys you can move the cursor through the menu line and with the *Down arrow* key or *OK* you can open a menu window and after that a dialog box with *OK.*

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main screen with activated menu line

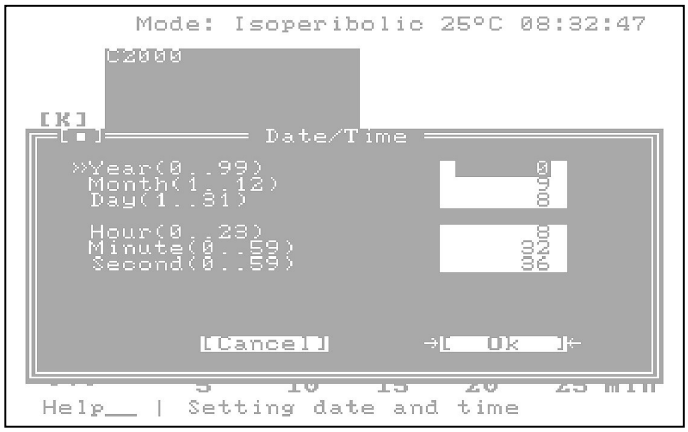
Verifying the date and time

①

Open the *System* menu.

②

Open the *Date/time* dialog box.



dialog box date/time

Meaning of the entries:

Year(0..99)	the number for the year, for example 1997 = 97, 2002 = 02
Month (1..12)	calendar month, for example March = 03
Day (1..31)	day of the month
Hour (0..23)	hour entry, 0 = midnight
Minute (0..59)	minute entry
Second (0..59)	second entry

③

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

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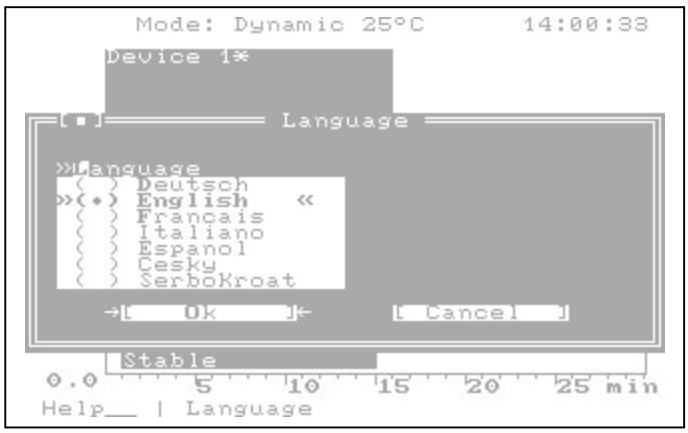
Select language

①

Open the *System* menu.

②

Open the *Language* dialog box. A list of languages in which dialogs can be run with the calorimeter system appears.



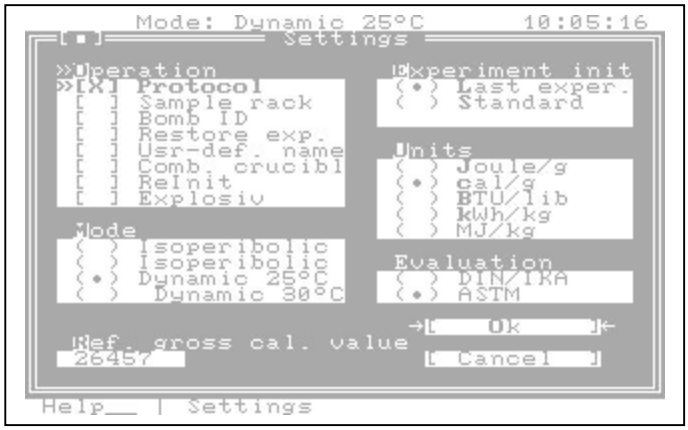
language dialog box

③

Use *Down arrow/Up arrow* to select your language from the list and confirm with *OK*. From now on, text on the screen, the text of the help system and printouts will appear in the new language.

System settings

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



settings dialog box

The window shows the configuration fields of the calorimeter. Use *Tab* to move the cursor to the next configuration field. To make a setting the *Operation* configuration field, you must place the cursor on the desired line with *Down arrow/Up arrow* and press the space bar ␣. Your entry is confirmed with “x“. Pressing the space bar again will delete the “x“ again.

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- *Operation* configuration field
  [ ] *Protocol*
  Marking this option causes a protocol to be printed out for every experiment. This also applies to the individual temperature measurement values during the measurement. In addition, the temperature protocol is displayed in the info-window for test parameters, see Section 9.1, “5-Info”.

  [ ] *Sample rack*
  A sample rack is connected and will be used. *Sample* dialog box can no longer be called manually. Instead it is activated by placing samples in the samples rack or removing them from it. This ensures reliable management of large numbers of samples.
  For more information on working with the sample rack, please refer to the C 5020 Operating Instructions.

  [ ] *Bomb Id*
  Decomposition vessels are automatically identified by their coding (DV detection). There is no manual entry of the code number of decomposition vessels. See Chapter 7, “Preparing and performing measurements”. This selection is deactivated when a device without automatic identification is being run.

  [ ] *Save experiment*
  The experiment can be started again if it is interrupted before the ignition takes place. The experiment parameters remain intact. Even if a test is interrupted after ignition with the error message *No temperature increase*, the test can still be restarted. To restart the experiment, the decomposition vessel must be removed from the measuring cell and suspended again.

  [ ] *Usr-def. name*
  Here you can specify whether you will enter the *Sample name* yourself in the *Sample name* entry field or in the *Sample* dialog box, or whether the system will automatically assign the *Sample name*. If this option is not selected, the system assigns experiment numbers in the *Sample name* box.

  [ ] *Disposable crucible*
  This option reduces the value for extraneous energy by 50 Joules since no cotton thread is used.

  [ ] *ReInit*
  Before every measurement the system tests whether cooling water flowthrough in the calorimeter is ensured and if the cooling water temperature is correct for the current mode. If these conditions are not satisfied, the measurement is not enabled. This option should only be used for major fluctuations in the temperature of the cooling water or with very low frequency of measurements.

  [ ] *Explosiv*
  This option is only valid for the C 2000, which is equipped with the C 60 calorimeter conversion kit. The standard decomposition vessels C 5010 and C 5012 must not be used in this mode. In the *Explosiv* mode, use exclusively the IKA® High-Pressure Decomposition Vessel C 62, or a Peters bomb modified by fitting IKA® Electrode Set C 61.
  The option can only be activated if DV detection is deactivated. The activated option allows the use of up to 20 decomposition vessels per calorimeter and changes the procedure for preparing and performing measurements (see Sections 7.3 and 7.4).

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- *Mode* configuration field
  In this field you can select an option for temperature regulation of the water jacket in the outer vessel. The following options are available:

  ( ) *Isoperibolic at 25 °C* — Measurement and calculation according to the isoperibolic procedure. The temperature of the outer vessel is 25 °C.
  This mode can run with cooling water temperatures of from 12 °C to 23 °C.
  Standard value: 18 °C to 20 °C

  ( ) *Isoperibolic at 30 °C* — Measurement and calculation according to the isoperibolic procedure. The temperature of the outer vessel is 30 °C.
  This mode is selected for cooling water temperatures from 23 °C to 28 °C.
  Standard value: 23 °C to 25 °C

  The isoperibolic measurement procedure is suitable for the most demanding levels of accuracy. The measurement time is 20 - 25 minutes/measurement.

  ( ) *Dynamic at 25 °C* — Measurement and calculation based on a shortened dynamic procedure. The temperature of the outer vessel is 25 °C.
  This mode can run with cooling water temperatures of from 12 °C to 23 °C.

  ( ) *Dynamic at 30 °C* — Measurement and calculation based on a shortened dynamic procedure. The temperature of the outer vessel is 30 °C.
  This mode is selected for cooling water temperatures from 23 to 28 °C.

  The dynamic measuring procedures ensure measuring times of 7 -12 minutes/measurement with sufficient accuracy.

  If the operating mode is changed or the calorimeter was not ready for operation, the initialization dialog box appears after the *Settings* dialog box closes to make certain that this mode can be implemented with the cooling water conditions that have been provided.

- *Ref gross cal. value* [J/g] configuration field
  In most cases, certified benzoic acid is used. The required gross calorific value should be entered. If you are working with different reference fuel, the gross calorific value of this fuel must be entered here.

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- *Experiment init.* configuration field
  With the experiment initialization you can determine how the *User* and *Sample proper* parameters will be set in the *Experiments* dialog box. Chapter 7, “Preparing and performing measurements“, contains further discussion of these setting options. You can select from the following options:

  ( ) *Last exper.* — For a new experiment, the system accepts the *User* and *Sample proper* parameters and the post-experiment parameters that were entered from the last experiment to be evaluated.
  If the option *Usr.-def name* was selected, the sample name will also be accepted. If there is a difference, the sample name must be edited or re-entered completely.

  ( ) *Standard* — For a new experiment, the post-experiment parameters are set to 0, extraneous energy is set to 50 J/0 J (without/with combustible crucible), and the *User* entry field and *Sample proper* in the *Sample* dialog box remain empty.

- *Units* configuration field
  The unit of measure of the caloric results is determined here.
  The following units are available for selection:

  ( ) *Joule/g*
  ( ) *cal/g*
  ( ) *BTU/lib*
  ( ) *kWh/kg*
  ( ) *MJ/kg*

- *Evaluation* configuration field
  ( ) *DIN/IKA* — The calculation modes used previously for IKA® calorimeters are grouped under this evaluation procedure.

  ( ) *ASTM D1989, D240, D5865, D4809, D5468, E711* — This procedure takes into account current US standards for combustion calorimetry of solid and liquid fuels, and wastes.

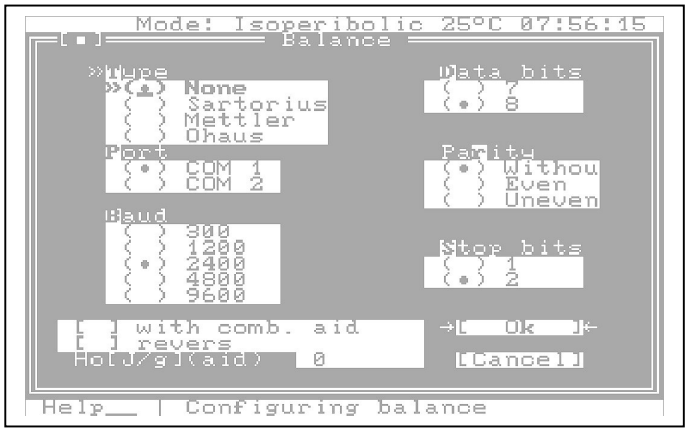
  The evaluation procedure selected will be used for all subsequent evaluations. Measurements already evaluated will not be affected by a change unless they are re-evaluated. In such a case, all the evaluation parameters already entered will be reset to zero.

OK causes the calorimeter system to accept the settings and closes the dialog box.

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Configure scale

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



scale dialog box

The window shows the configuration fields of the scale. The parameters that you select here must agree with the interface parameter of the connected scale. For information on the parameters, please refer to the Scale manual.
Use *Tab* to move the cursor to the next configuration field. *Up arrow/Down arrow* moves the cursor within a configuration field. If you exit a field with *Tab*, the current setting in the field is retained.
You can make the following settings with the configuration fields:

- *Type* configuration field
  Here you can indicate which scale is connected to the system. Either None (no scale) or one of the types indicated may be connected.

- *Port* configuration field
  No entries are possible in the *Port* field. The scale is always connected to COM1.

- *Baud* configuration field
  The data transmission rate between the scale and the calorimeter system can be adjusted in the increments 300, 1200, 2400, 4800, 9600 and 19200 bits/sec.

- *Data bits* configuration field
  Here you can select whether the data will be transferred in 7-bit or 8-bit format.

- *Parity* configuration field
  Enter whether the data that is transferred should be accepted by the calorimeter system *without* testing the parity or whether a test for *even* or *odd* parity should be performed.

- *Stop bits* configuration field
  Select either *1* or *2* stop bits for the data transmission protocol.
  If you are using a combustion aid or a disposable crucible, it is possible to record the weight of the combustion aid by using a special scale mode or disposable crucible and to calculate the resulting extraneous energy automatically.

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- *with comb. aid* configuration field
  If the option *with comb. aid* is marked, the values of the scale are transferred in the following order:
  1. “Weighed in quantity, combustion aid”
  2. “Weighed in quantity, combustion aid + weighed in sample”

- *revers* configuration field
  If the *revers* configuration field is marked in addition to the *With combustion aid* field, the scale values will be transferred in the following order:
  1. “Weighed in sample”
  2. “Weighed in sample + weighed in combustion aid”
  After the second measurement value is transferred, the *New measurement* dialog box appears. The calculated value for extraneous energy is already entered in this dialog box.

- *Ho [J/g] (aid)* configuration field
  In combination with the configuration field *with comb. aid*, the gross calorific value of the combustion aid must be entered in this field for the system to be able to calculate the extraneous energy.

6.7 Turning off the system

When you want to turn off the calorimeter system, open the System menu and bring up Exit.

There must be no decomposition vessel suspended in the measuring cell cover.

Use only the Exit item in the System menu to turn off the device. Do not use the power switch, as this will result in lost data!

When the system has shut down, a message to that effect appears on the display. The message also asks you to turn off the power switch for the calorimeter and cooler unit.

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7 Preparing and performing measurements

The term “measurements” as used in the following presentation includes both measurements for calibration of the calorimeter system (calibration measurements) and actual measurements for determining gross calorific values. The difference consists mainly in the evaluation (see Chapters 8 and 9 on this subject). The preparation and actual execution is nearly identical in both cases.

7.1 Notes on calibration

Before precise measurements are possible with the calorimeter system, it must first be calibrated. This is done by performing combustion on tables of **certified benzoic acid** (see Accessories) with a known gross calorific value. From this process, the heat energy that is required to raise the temperature of the calorimeter system by 1 degree Kelvin is used to determine the heat capacity (C value) of the system. This value is used for the subsequent determinations of gross calorific value.
The heat capacity is determined with the measuring cell and the decomposition vessel (DV). It has a substantial influence on the gross calorific value being determined, and must be re-determined particularly when the device is first placed in service, after maintenance service, and after parts are replaced.



If a measuring cell is operated with more than one decomposition vessel, the heat capacity of the system must be determined by calibration for each decomposition vessel. A decomposition vessel may only be used in the measuring cell in which it has also been calibrated.
The calorimeter system must be calibrated in each operating mode in which measurements will later be performed. Please refer to the applicable standards on this subject.

The calibration must take place under the same conditions that will prevail in the subsequent experiments. If specific amounts of substances are added to the decomposition vessel during combustion experiments (for example distilled water or solutions), exactly the same amount of the substance must be added during calibration.

Note on calibration

- In order to achieve precise results, you should make certain that the temperature does not increase by more than 4 K during the combustion.

- The increase in temperature during the determination of gross calorific value must be of the same magnitude as for the calibration (for example: 2 tablets = about 1g benzoic acid ≅ 3.1 K). It may be necessary to perform several experiments to determine the optimal sample size.

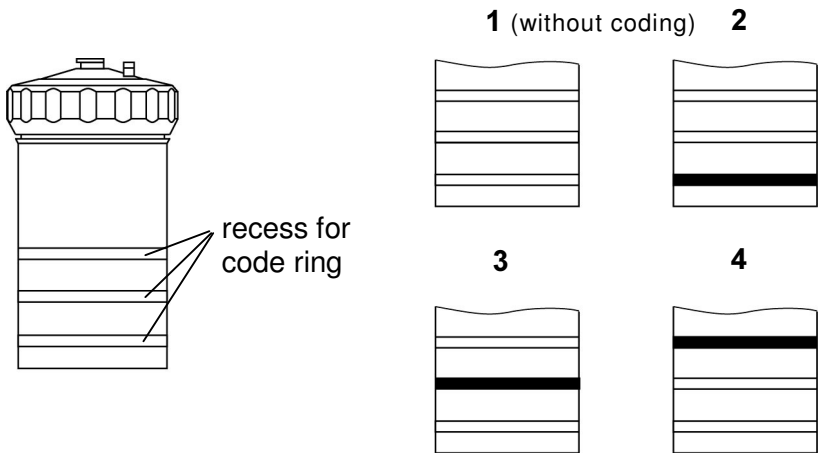
Coding
A maximum of 4 decomposition vessels may be used when working with the calorimeter system. This is possible by coding the decomposition vessels from 1 to 4. For systems with automatic DV detection, the calorimeter recognizes which vessel a measurement is being performed with and assigns its calibration parameters to it.

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

To do this, attach the black code rings into the recesses of the decomposition vessel provided for that purpose.

coding of the decomposition vessels



7.2 Notes on the sample

Decomposition vessels C 5010 and C 5012 are not permitted for experiments on fuel samples capable of exploding! Note in this regard Chapter 1 "For your safety". 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 C 5010 or C 5012.

The C 2000 calorimeter system is a precision measuring instrument for routine determination of gross calorific values of solid and liquid substances. Exact measurements are only possible, however, if the individual steps of the experiment are followed carefully. The procedure as it is described in Chapter 1 "For your safety" and in the following sections must therefore be followed 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).

notes on the sample

Substances of which the combustion behavior is not known must be examined for their combustion behavior before combustion in the decomposition vessel C 5010, C 5012 or C 62 (danger of explosion). If you are burning **unknown samples**, leave the room or **keep a safe distance** between you and the calorimeter.

solid substances

A few points must be observed in reference to the substances to undergo combustion. Normally, solid substances can be burned directly in powder form. Substances that burn rapidly (for example benzoic acid) must not be burned in loose form.
Benzoic acid must only be burned in the form of pellets! Combustible dust and powder must be compressed into pellets before combustion. Oven-dry dust and powder such as wood chips, hay, straw, etc. burn in an explosive manner! They must be moistened first! Readily combustible liquids with a low vapor pressure must not be come in direct contact with the cotton thread (for example tetramethyl dihydrogen disiloxan)!

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Rapidly burning substances have a tendency to squirt or spark. It would therefore not be possible to ensure complete combustion. In addition, these substances could damage the inner wall of the decomposition vessel. Such substances must therefore be pressed into tablets before undergoing combustion.

The IKA® C 21 pelleting press, for example, is suitable for this purpose.

liquid substances

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

highly volatile substances

Gelatin capsules or acetobutyrate capsules (see Accessories) that are filled with the fuel sample are used with highly volatile substances. The gross calorific value of the capsules must be known in order to take into consideration the combustion heat generated by the capsules as extraneous energy.

combustion aids

The capsules described above or combustion bags made of polyethylene (see Accessories) are used for substances with low flammability or low calorific substances. C 14 disposable crucibles can also be used.
Before the capsules or the combustion bag is filled with the substance to be determined, it must be weighed in order to determine the additional extraneous energy introduced into the system from the weight and the gross calorific value (see the *With combustion aid* scale mode). This is taken into consideration with *QExtran1*. The amount of combustion aid should be as little as possible.

acid formation, heat of solution

Almost all substances to be analyzed contain sulfur and nitrogen. Under the conditions that prevail during calorimetric measurements, sulfur and nitrogen undergo combustion and form SO_2, SO_3 and NO_X. Sulfuric and nitric acid arise in combination with the water resulting from combustion. Heat of solution is also generated. This heat of solution is taken into consideration in calculating the gross calorific value. In order to quantitatively record and determine all acids that have been formed, about 5 ml of distilled water or another suitable substance can be added to the decomposition vessel before the experiment.



In this case, the calibration of the system must also be performed with the same amount of water added!

After combustion, the water that was added is collected and the decomposition vessel is rinsed thoroughly with distilled water. The rinsing water and the solution formed from the water that was added are combined and the acid content of the combined solution is examined. If the sulfur content of the combustion aid and the nitric acid correction are known, the water does not need to be analyzed.



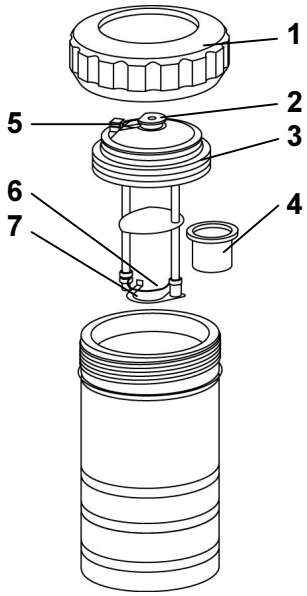
To increase the service life of parts subject to wear and tear (O rings, seals, etc.), we recommend you work with water added to the experiment as a matter of general principle.

substances rich in halogen

The C 5012 decomposition vessel should be used for substances rich in halogen.

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7.3 Preparing the measurement



individual parts of the decomposition vessel

1	cap screw
2	oxygen valve
3	cover
4	crucible
5	electrical ignition contact
6	ignition wire
7	crucible holder



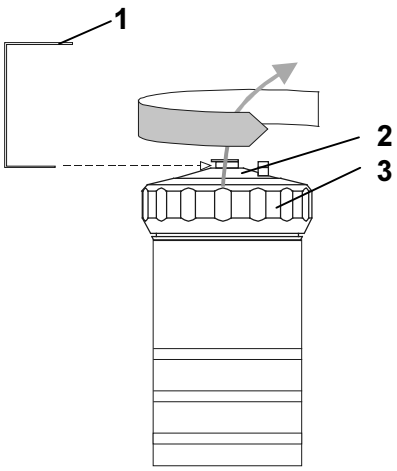
Now you can load the sample into the cleaned decomposition vessel (see 7.5).

If you are using several decomposition vessels, their individual parts must not be exchanged among themselves (see stamping individual parts).

The decomposition vessel should be prepared with the following steps:

①

Unscrew the cap screw and remove the cover by using the handle.



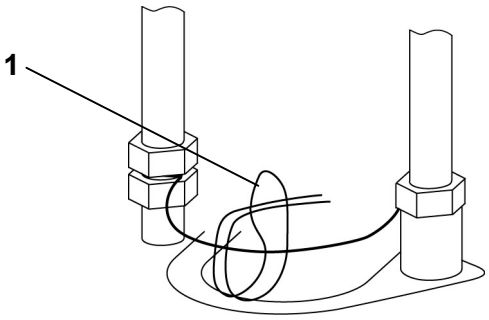
opening the decomposition vessel

1	handle
2	cover
3	cap screw

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②

Fasten a cotton thread onto the middle of the ignition wire with a loop.



fastening a cotton thread

1 cotton thread

③

The substance must be weighed directly into the crucible with an accuracy of 0.1 mg. Distilled water or a solution must also be added to the decomposition vessel.

To increase the service life of parts subject to wear and tear (O ring, seals, etc.), we recommend you work with water added to the experiment as a matter of general principle.

Substances with low flammability should be weighed into the crucible with a combustion aid. The combustion heat of the combustion aid must be known. Note in this regard Section 7.2, “Notes on the sample” and Chapter 1 “For your safety”.

STOP

Generally, the substance weighed in must be selected such that the increase in temperature is less than 4 K during the measurement and comes close to the increase in temperature for the calibration (maximum energy input: 40,000 J).
Otherwise, damage may occur to the decomposition vessel.

Damaged decomposition vessels are at risk of bursting!
Please observe the Operating Instructions for the decomposition vessel.

When working with unknown substances, very small amounts must be weighed in at first in order to determine the energy potential. If you are burning unknown samples, leave the room or keep a safe distance between you and the calorimeter.

If distilled water or solutions are placed in the decomposition vessel during the combustion experiment, calibration must previously have been performed with the same material added.

If you are using a combustion aid, you must add the energy from the combustion aid in the *Sample* dialog box to the energy input in the *QExtran1* field, or use the corresponding scale mode *With combustion aid.*

If you use a scale in *With combustion aid* mode and previously transfer the weight of the combustion aid, the extraneous energy calculated from this information appears in this field.

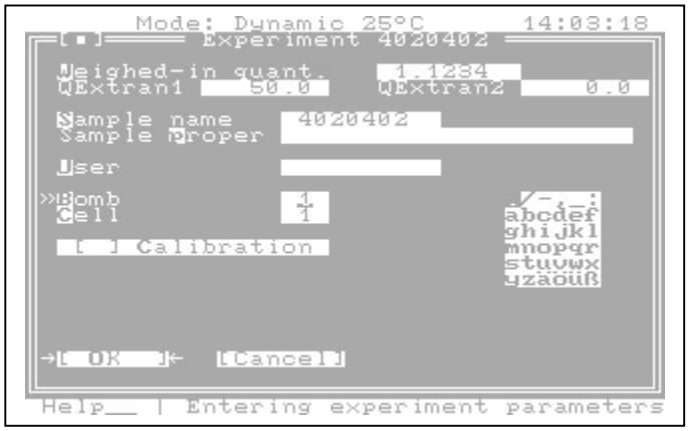
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④

Make certain that the desired operating mode is set (see Chapter 6.6, “Configuring the system). Then open the *Sample* dialog box to enter parameters. If the sample rack is active, parameter input appears automatically when you place a crucible in the rack or remove one (see also the Operating Instructions for the C 5020 sample rack).

Enter the weight of the combustion sample in the *Weighed-in quant.* field. If an electric scale is connected to the calorimeter system, the weight can be transferred automatically. Depending on the type of scale, the *Sample* dialog box can be opened either by the sample key of the calorimeter or with the *Print/Transfer key* of the scale. You can cause the scale value to be accepted again by pressing the *Space bar.*

sample dialog box



When preparing a measurement, if DV detection is not activated, the number of the decomposition vessel is entered. In *Explosiv* mode, the desired decomposition vessel is selected from a list of the available decomposition vessels.

With *Tab* you can move the cursor to the following input fields. The meaning of the remaining input fields:

- *QExtran1*
  Correction for the thermal energy from the cotton thread used as an ignition aid. A preset value of 50 J/0 J (without/with combustible crucible) appears here. If you use a different ignition aid instead of the IKA® cotton thread, change this value as appropriate.

- *QExtran2*
  Correction for the thermal energy from an additional combustion aid. The preset value is 0.
  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 *QExtran2* 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 *QExtran2* box and then press the ↓ arrow. *QExtran2* is calculated according to the formula

  QExtran2 = Weighed in quantity of combustion aid x calorific value of combustion aid

  and is entered in the *QExtran2* box. If a value > 10 J is entered, it is assumed

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that the value already represents all extraneous energy *QExtran2.* In this case, there is no further conversion based on the formula given above.

Note: In all automatic calculations, 70 J is taken into account for the electrical ignition energy.

- *Sample name*
  For each measurement, the software automatically assigns a sample number in the format **ymmddnn,** where **y** is the year, **mm** is the month **dd** is the day and **nn** is the running number, all encoded into the number. With sample names formed in this manner it is very easy to select specific groups of measurements from the library for processing.
  If you select the option *Usr-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 not taken into consideration).
  If you have also selected the *Last* option under *Menu*, *Configuration*, *Settings*, *Experiment Init.,* the number of the last measurement appears as a suggestion for the current measurement. If you do not edit the name, the sample number will stay the same for all measurements!

  Example of a *Sample name* = 6052401

  6 Year digit, 6 = 1996
  05 Month 1 – 12, 05 = May
  24 The day of the month, here May 24
  01 Running experiment number

- *Sample proper*
  Any additional information on the sample. With the *Arrow keys* you can select from the character table of letters and symbols. The *Decimal point* key causes the system to accept the selected character into the input field (max. 40 characters).

- *User*
  Name of the user (maximum of 8 characters, entry as for *Sample properties.*

- *[ ] Calibration*
  Mark this field with the space bar if you want the system to use the experiment for calibration.

With *OK* the system will accept input from the dialog box.

⑤

The message *Bomb* ↓ in the footer of the display, i. e. from this point on the decomposition vessel can be suspended into the measuring cell cover.

⑥

Place the crucible in the crucible container.

⑦

Align the cotton thread with a pair of tweezers so that it hangs down into the crucible and is immersed in the sample. This ensures that during the ignition process the burning thread will ignite the sample.

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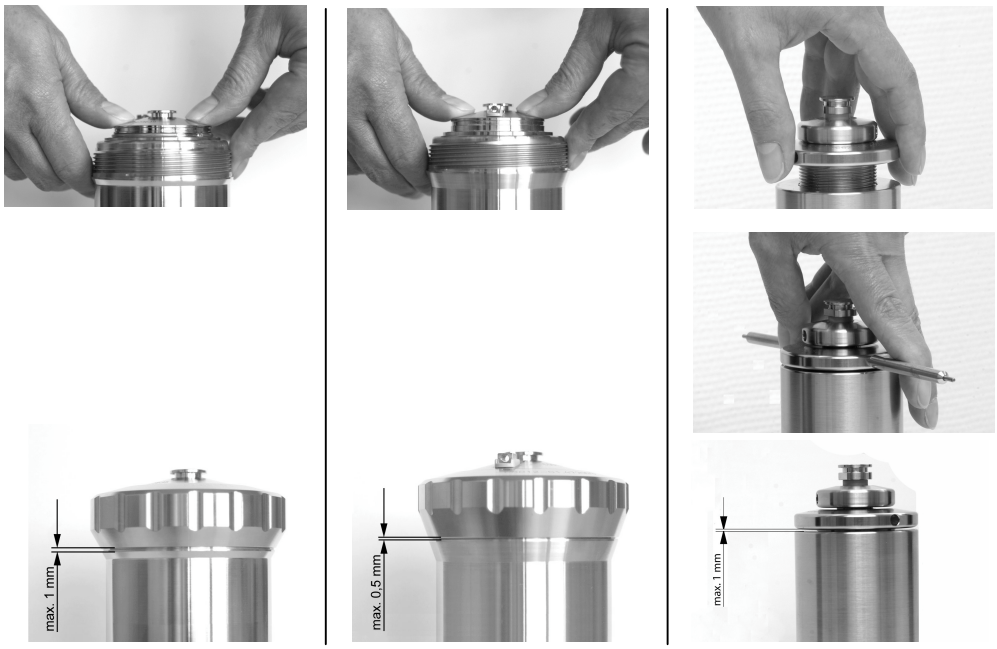
⑧

Close the decomposition vessel (C 5010, C 5012 or C 62).
Place the cover onto the lower section and push down until it presses against the stop piece in the lower section.

C 5010 C 5012 C 62



Place the union nut onto the lower section and tighten by hand.



The decomposition vessel can now be filled.

7.4 Performing the measurement

①

Guide the decomposition vessel into the filler head of the open measuring cell cover until it catches in place.

Always hold the decomposition vessel securely on the top by the cap screw!

The decomposition vessel maintains a defined position in the holder on the fill head by being lowered at the centre of the fill head by 0.8 mm. A spring element then makes contact with the electrical ignition contact on the decomposition vessel.



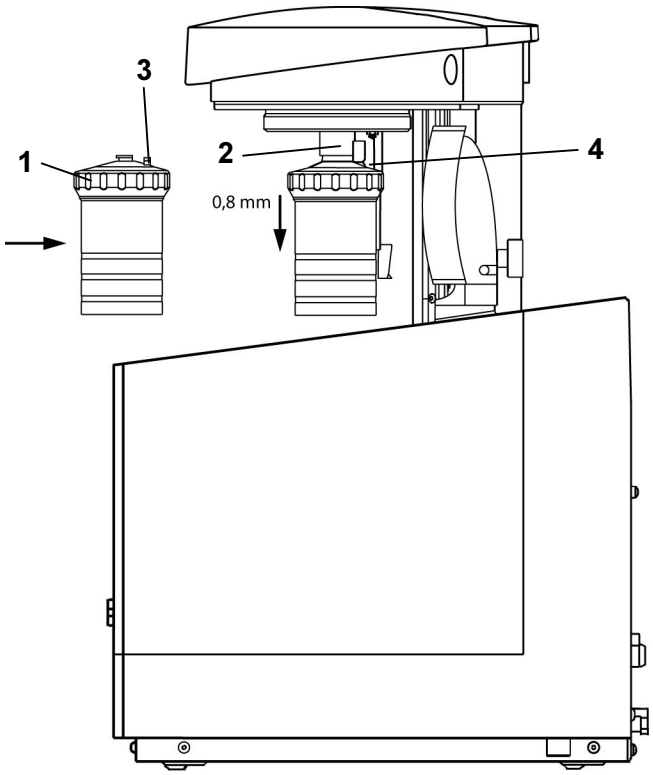
The decomposition vessel is now suspended vertically in the receiving area (check everything one last time).

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The message "Bomb securely closed?" will appear. Ensure that the decomposition vessel is properly closed and confirm with OK.

The ignition wire in the decomposition vessel completes the electrical circuit. The calorimeter is ready to start. The message *Bomb* changes to a display of the function key assignment *Start.* If the *Start* function key does not appear, please check the ignition wire of the decomposition vessel.

suspending the decomposition vessel into the filler head of the measuring cell



1 cap screw
2 filling head
3 ignition contact
4 spring element

②
Push the *Start* button.

Each time 1000 ignitions have been performed using a given decomposition vessel, the following message will appear:

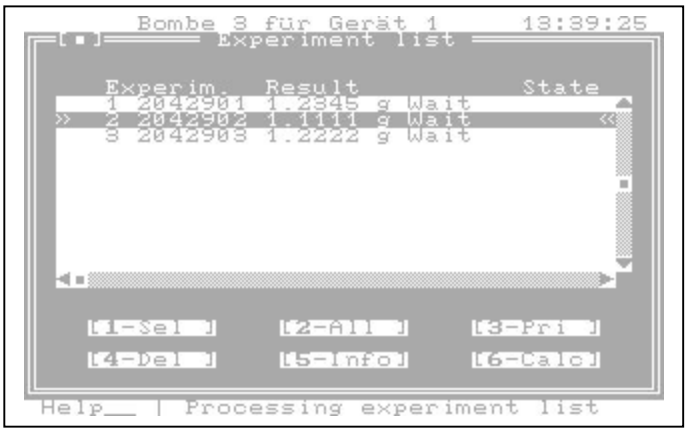
1000 ignitions performed with Bomb x
Inspect decomposition vessel
or contact IKA service

This indicates that the decomposition vessel has reached a maintenance point and that a safety check must be carried out. Confirm this message by pressing *"7", TAB* and *OK* in sequence.
This message does not release the user from the responsibility of also continuously checking the decomposition vessel for wear and carrying out safety inspections as required.

In *Explosive* mode, please observe in addition the following:
As many as 20 decomposition vessels per device can be used. As many measurements can be prepared as there are decomposition vessels available.

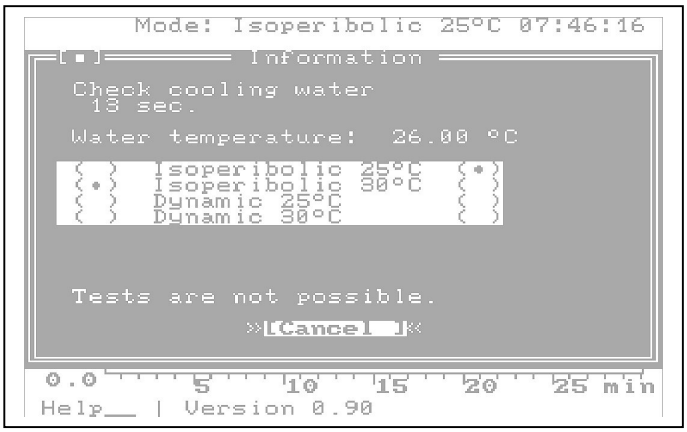
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A window appears at the beginning from which the correct measurement must be selected.



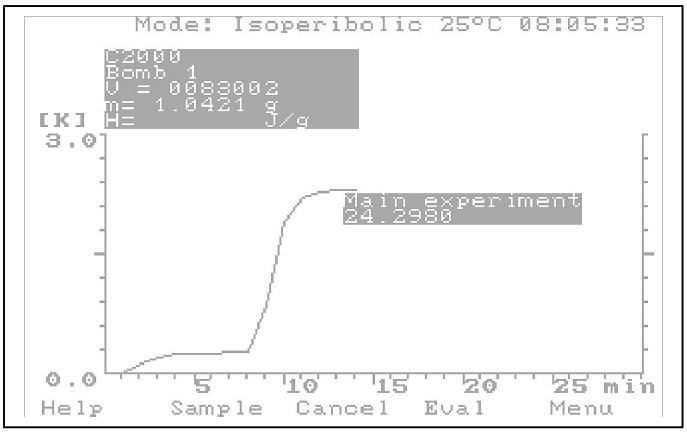
experiment list

If the *ReInit* option is active, a check of the cooling water will first be performed. The remaining course of the measurement is now enabled if stable temperature equalization of the outer vessel is ensured. Otherwise the measurement must be interrupted.



examination cooling water

After this the measuring cell cover closes. The decomposition vessel is filled with oxygen (for about 60 sec.).
After the temperature in the outer vessel has stabilized (90 ... 120 sec.), the inner vessel fills with water. As soon as the system starts the experiment, the display shows the course of the temperature of the inner vessel over time as a graph.



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The measurement proceeds fully automatically until the final result. If the measurement needs to be interrupted, a message window appears that can be acknowledged by the user. (See Section 12, “Troubleshooting”).

③
If necessary:
You can interrupt the experiment at any time with *Cancel*.

④
If the measurement is complete or has been interrupted, the measuring cell cover opens and the inner vessel is emptied. As soon as the message *Bomb* ↑ appears in the footer, you can remove the decomposition vessel.

⑤
Pressure can be released from the decomposition vessel with the venting button by using a laboratory discharge hood or with the C 5030 venting station available as an accessory.

⑥
Open the decomposition vessel and check the crucible for signs of incomplete combustion. If combustion was incomplete, the results of the experiment must be discarded. The experiment must be repeated.

7.5 Cleaning the decomposition vessel



If you suspect that the combustion sample, the gasses generated from it or the residue of combustion could be hazardous to health, protective personal equipment (for example protective gloves, breathing mask, etc.) should be worn when working with these materials. Residue of combustion that is hazardous to health or dangerous to the environment must be disposed of as special waste. We make explicit reference to the applicable requirements.

It is of fundamental importance for precise measurements for the decomposition vessel to be clean and dry. Impurities will change the heat capacity of the decomposition vessel and cause the results of the measurement to be inaccurate. After every combustion experiment the walls of the vessel, the inner fittings (holders, electrodes, etc.) and the combustion crucible (inside and outside!) must be cleaned thoroughly.

vessel inner walls

In most cases, all that is necessary is to remove condensation from the inner walls of the vessel and the inner fittings. It is sufficient to wipe off the parts carefully with an absorbent lint-free cloth.
If it is not possible to clean the decomposition vessel with the measures described (for example singed spots, pitting, corrosion, etc., you should call the Technical Service Department.

crucible

The residue of combustion in the crucible, for example soot or ash, can also be wiped out with an absorbent and lint-free cloth.

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8 Evaluating calibrations

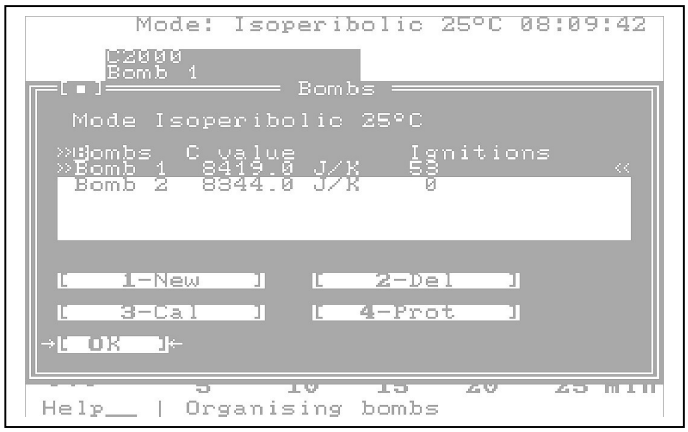
①

Perform several calibration experiments per decomposition vessel according to Chapter 7 “Preparing and performing measurements”. For the number of calibrations required, please refer to the standard you are using.

②

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

configuration of decomposition vessels



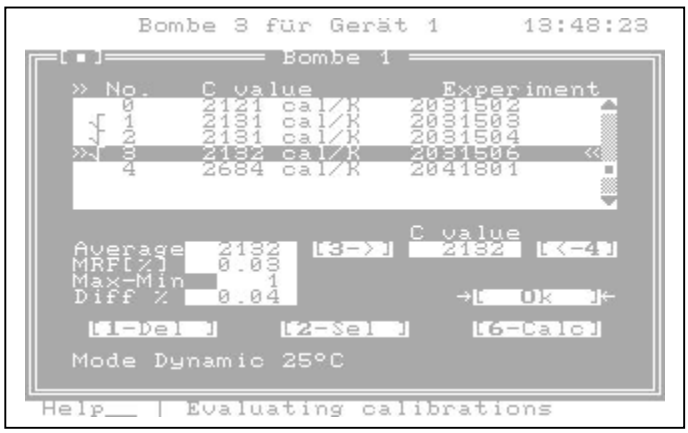
③

Use *Tab* and *Up arrow/Down arrow* to place the cursor on the number of the decomposition vessel with which you wish to perform the upcoming calibration experiments.

④

Open the *3-Cal* dialog box.

list of calibrations



Using the button *6-Calc* in the window *list of calibrations*, carries out the acid correction for the selected calibration. The calculation is dependent on the evaluation procedure selected:

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- Evaluation procedure ASTM D1989, D240, D5865, D4809, D5468, E711
  For calibration, a dialog reduced to the acid-correction fields and a shortened results form are displayed. The comments in Section 9.2 under Point ③ apply here too.

- Evaluation procedure DIN/IKA
  In the DIN/IKA evaluation procedure, the acid correction to ASTM D240 or ASTM D1989 can also be used for calibration. The results form is shortened accordingly. The other calculation modes are not available for calibrations.

Note:

The system saves a maximum of 20 calibrations per decomposition vessel and mode. A message on the display indicates when this number has been reached.
It is then no longer possible to save new calibrations. In this case older calibrations must be deleted manually.

Before decomposition vessels can be deleted from the assignment to the measuring cell, the corresponding calibration values must first be deleted. Otherwise it is no longer possible to assign these calibration values and they will unnecessarily take up space in memory. These calibrations can later be removed from the library only by a service technician.

The calibration experiments are listed in the dialog box. The meaning of the columns in the list of experiments is as follows:

No	The running number of calibration experiments
C value	The heat capacity of the calorimeter system determined with the experiment in question
Experiment	The *Sample name* of the relevant experiment

⑤

Use *Tab* to place the cursor on *2-Sel* and confirm with *OK* or press the *2* key. You have thus selected the experiment for calibration. The experiment is marked on the display with "√".

⑥

Use *Tab* and *Up arrow* to place the cursor on the next experiment and press *2-Sel*. This will select the next experiment for calibration. The average value of the selected experiments, the average relative error by percent, and the deviation range (max/min) absolutely and by percentage are displayed in the corresponding fields.

Average	Calculated mean value
MRF[%]	Average relative error (German abbreviation)
Max-Min	Deviation range
Diff %	The range of deviation as a percentage in reference to the average value

⑦

Repeat Step 6 for all values that are to be selected. *Average* will then display the average C value of these experiments.

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⑧

The following apply to evaluating successful calibrations:

MRF[%]	The average relative error < 0.2 % (according to ISO 1928)
Diff %	The deviation range as a percentage < 0.4 % (according to DIN 51900)

Other criteria may also apply depending on the standard you are using. The values given above, however, will be sufficient to fulfill customary requirements of accuracy for a calorimeter.

⑨

Use *Tab* to place the cursor on the *[3–>]* button and confirm with *OK* or press *3*. The average value of the selected calibration experiments will thus be assigned to the calorimeter system as the system heat capacity or C value. If you place the cursor on *[<–4]* press *OK*, you can enter the system heat capacity manually in the *C value* field.

⑩

Place the cursor on the experiments that were not used to calculate the average value and delete them with *1-Del.*

①①

Exit the dialog box with *OK.* The system calibration is thus complete and you can now continue with the determination of gross calorific values.

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9 Evaluation of gross calorific value determinations

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

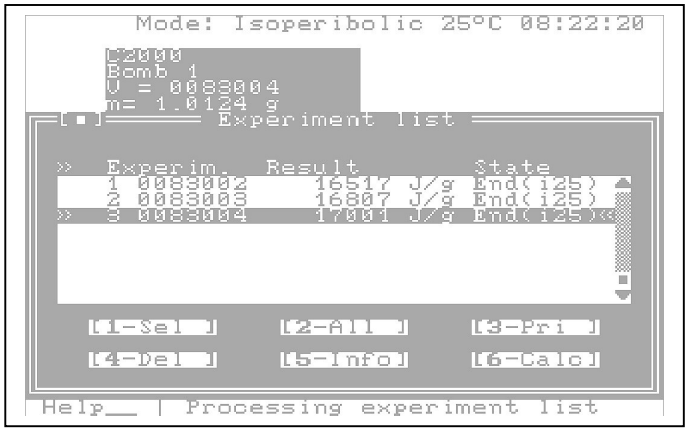
9.1 Post-processing experiments

The calorimeter system categorizes saved experiments into two groups, “Today‘s experiments” and “Library”. Today’s experiments are those experiments that have been performed since the system was turned on. The library is long-term storage.

Processing today’s experiments

①
Activate *Eval.* The *Experiment list* dialog box appears.

experiment list



②
A list appears with today’s experiments. The meanings of the columns in the list field are as follow:

Experiment Sample name, designation of the combustion sample

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

Status *End*
The experiment produced a result.
The operating mode is shown in parentheses. i25 (isoperibolic 25 °C), i30 (isoperibolic 30 °C), d25 (dynamic 25 °C), d30 (dynamic 30 °C)

can
The experiment was stopped.

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+cal
The experiment was performed for calibration.

+Sim
The experiment was a simulation.

Eval
The experiment was evaluated.
- Code Eval: the DIN/IKA evaluation procedure was used.
- Code ASTM: the ASTM D1989, D240, D5865, D4809, D5468, E711 evaluation procedure was used.

wait
The fuel sample is in the crucible, entry of parameters is complete, and the experiment can be started.

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

run
The experiment is currently being processed in the measuring cell.

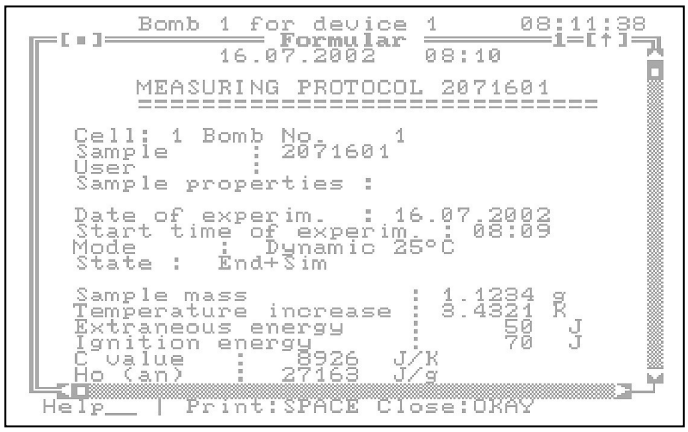
The buttons have the following functions:

1-Sel	Marks an experiment in the list. Exception: calibration
2-All	Marks all experiments (max. 100 experiments) in the list. Exception: Experiments with status *+cal* and *Prep.*
3-Pri	Prints the list of experiments
4-Del	Deletes the experiments that have previously been marked with *1-Sel* or *2-All.* Exception: *+cal*; *Prep.*
5-Info	Opens an information window with the experiment parameters
6-Calc	Opens a dialog box to convert the results of the experiment into different reference states

You can use the *Up arrow/Down arrow* keys to select the experiment from the list that you want to post-process. With *Tab* you can then move the cursor from the list field to the buttons.
To confirm the buttons, either place the cursor on a button with *Tab* and click on *OK* or press the key with the appropriate number button on the numeric keypad.

③
Place the cursor on the experiment that was just completed and confirm with the *5-Info* button. An information window opens with the results of the experiment.

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information window experiment

Post-processing experiments from the library

①

In the *Experiments* menu, open the *Experiment library* dialog box. The header displays the amount of free memory for experiments.



library: search functions

②

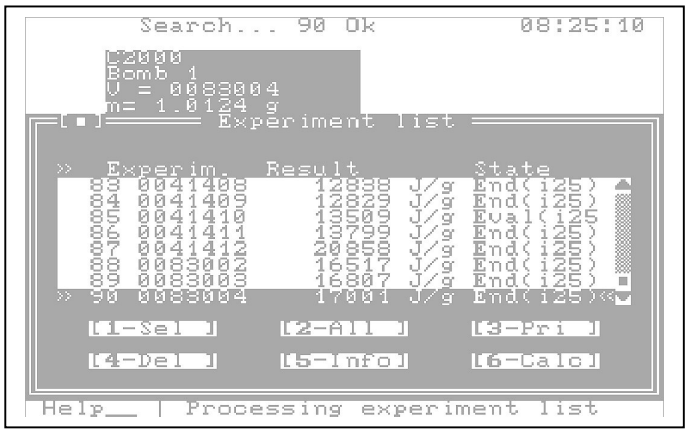
A search mask appears in which you must enter the sample name of the experiment that you want to post-process. If you want to select a whole series of experiments, you must enter the part of the sample name that the whole series of experiments has in common. If you enter a *decimal point* while you are in the *search mask*, the system will list all experiments that are stored in the library. If *search mask* remains empty, the display shows the list of the last search procedure. The *Add* option adds the list of the new search procedures to the list of the last search procedure. Confirm your entry with *OK*.

The search routine will find all measurements that match the search mask. The entries in the display are unsorted. No more than 100 measurements can be displayed. Measurements that are not displayed can only be shown after another search mask with criteria that select more entries. During and after the search procedure, the header line shows the number of measurements found.

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③

A list appears with experiments whose sample name agrees with the search mask.



library: search results

9.2 Calculating reference states / experiment evaluation

The evaluation includes the following points:

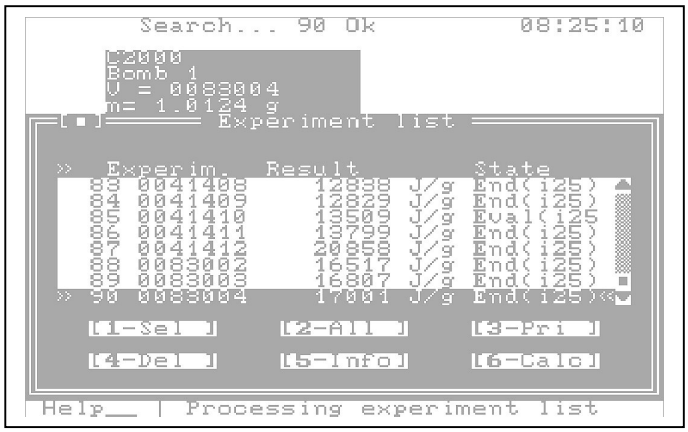
- Acid correction of the gross calorific value
- Calculation of the heat value
- Conversion to other reference states

Calibrations cannot be evaluated. An acid correction will only be carried out for calibrations. This evaluation will be made in the window *list of calibrations*. (See Section 8 “Evaluating calibrations”)

Several input modes are available for these calculations from which you can select the one that corresponds to your existing sample parameters. This will cover many application cases that occur in everyday situations. The formulas used are largely derived from the German standards, DIN. You can find an exact description there or in other applicable standards.

①

Open the *Evaluation* dialog box. You can reach this dialog box either through the *Experiments* menu in the header or through the *Eval*. function key.



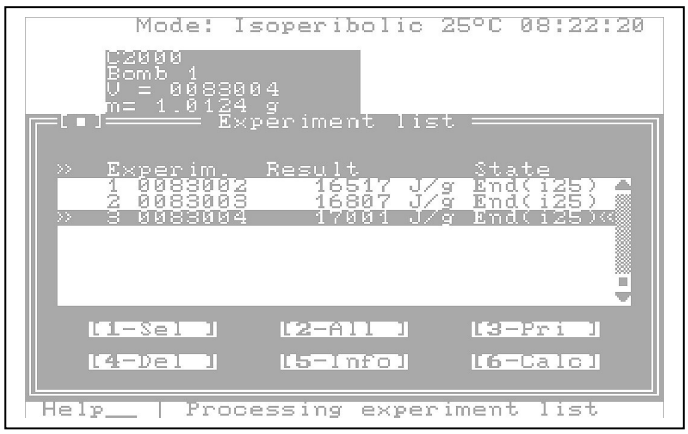
experiment list

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②

The list of today's experiments appears. Use *Up-arrow/Down-arrow* to select the appropriate experiment and press the *6* key or use *Tab* to place the cursor on the *6-Calc* button and then confirm with *OK.*

selection of calculation mode and input of evaluation parameters

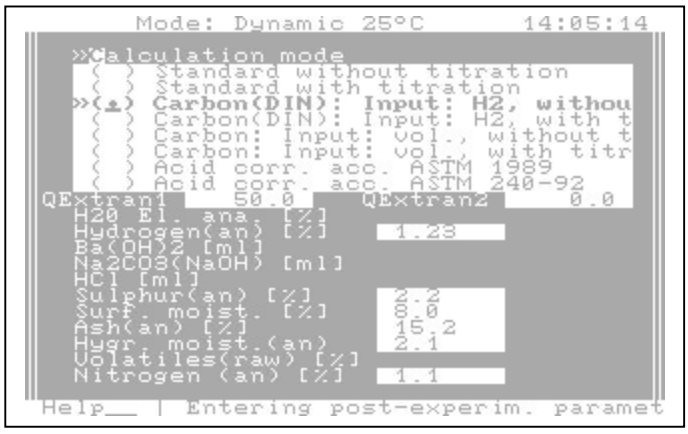


③

There are two different windows, which one is used depends on the evaluation procedure:

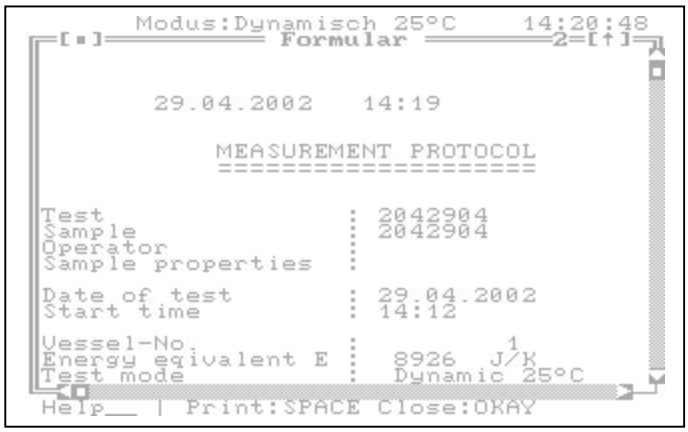
1. Evaluation procedure ASTM D1989, D240, D5865, D4809, D5468, E711
A window opens in which the ASTM standard used can be selected and the necessary evaluation parameters entered. The formulas and designations are those used in the ASTM standard.

dialog box for ASTM evaluation

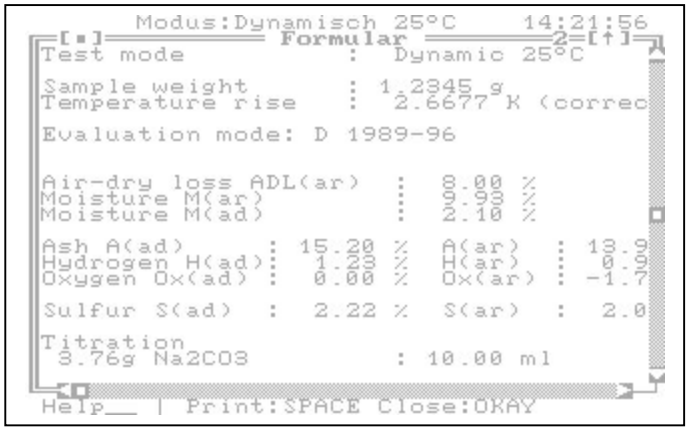


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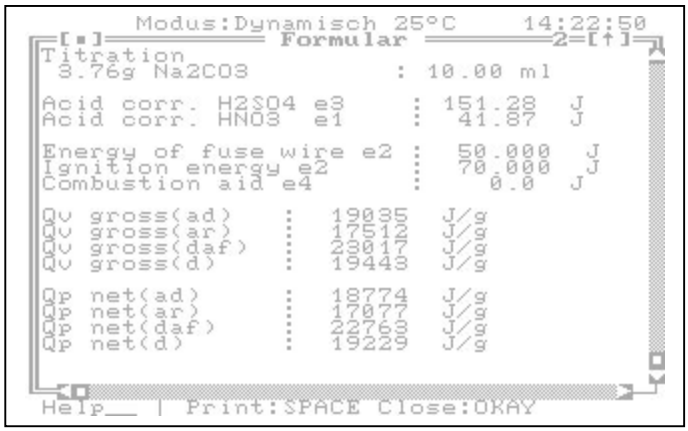
measurement protocol (1)



measurement protocol (2)



measurement protocol (3)



2. Evaluation procedure DIN/IKA

A dialog box for entering the results of analytical examinations of the sample and combustion residues appears. Parameters that were determined in the state in which the sample was delivered are identified with (raw) and parameters from the reference state of analysis moisture are marked with (an). The dialog box provides input boxes for the parameters of the selected calculation mode.

You can select from the following modes:

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standard calculation modes

Standard without titration
Standard with titration

carbon calculation modes

Carbon: H2 input, without titration
Carbon: H2 input, with titration
Carbon: Volatile input, without titration
Carbon: Volatile input, with titration
Acid correction based on ASTM 1989
Acid correction based on ASTM 240

The carbon calculation modes mentioned are used exclusively for examinations of the gross calorific value of carbon.

In addition to the heat of solution from the formation of acid, they also take into consideration the percentage of ash and, depending on the carbon calculation mode selected, calculate the percentage of water from the moisture level of the sample and the percentage of volatile components.

experiment parameters

Meaning of the parameters used:

QExtran1	Extraneous energy from combustion of the cotton thread or other ignition aid; 70 J is automatically taken into account for the electrical ignition energy.
QExtran2	Extraneous energy from the burning of additional combustion aids.
H_2O El. ana.	The percentage of combustion water in the sample
Sulfur (an)	The percentage of sulfur
Nitrogen (an)	The percentage of nitrogen
Hydrogen (an)	The percentage of hydrogen water in the combustion sample
$Ba(OH)_2$	The titrated quantity of 0.1 N barium hydroxide solution (titration of distilled water with which the decomposition vessel was washed out after the experiment)
Na_2CO_3	The quantity of sodium carbonate solution (in accordance with DIN 20 ml 0.05 N) added in the decomposition vessel
HCl	The titrated quantity of 0.1 N hydrochloric acid (titration of distilled water with which the decomposition vessel was washed out after the experiment)
NaOH	The titrated quantity in ml (0.0866 N)
Surf. moisture (raw)	The percentage of water from the approximate moisture
Ash (an)	The percentage of ash
Hygr. moisture (an)	The percentage of water from the hygroscopic moisture
Volatiles (raw)	The percentage of volatile components

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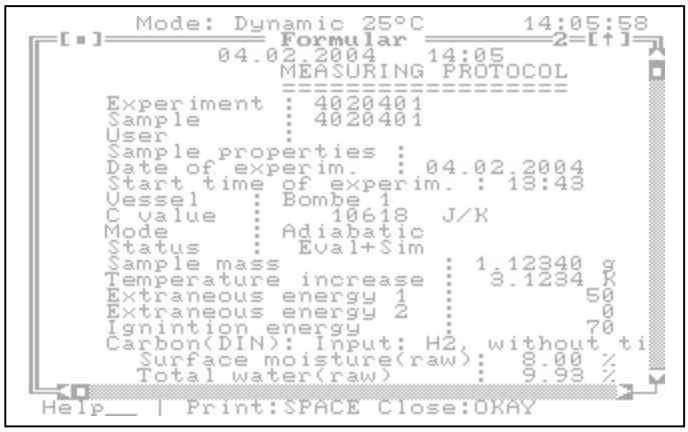
④

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

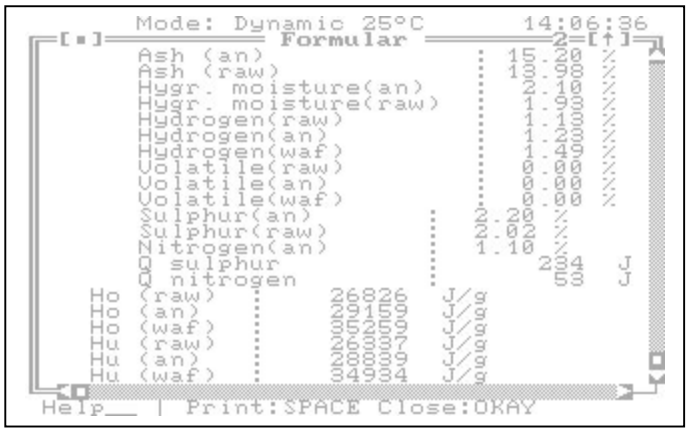
⑤

A new window appears and displays the measurement protocol with the definitive results of the experiment. You can print out the measurement protocol by pressing the *Space bar,* after which you can exit the protocol window with *OK.* You can browse through the protocol with the arrow keys.

measurement protocol (1)



measurement protocol (2)



Meaning of the individual parameters:

Ash (an)	The percentage of ash in the fuel sample in the “analysis moist” reference state
Ash (raw)	The percentage of ash in the fuel sample in delivery state
$Ba(OH)_2$	The titrated quantity of 0.1 N barium hydroxide solution (titration of distilled water with which the decomposition vessel was washed out after the experiment)

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Extraneous energy 1	Extraneous energy from combustion of the cotton thread or other ignition aid; 70 J is automatically taken into account for the electrical ignition energy.
Extraneous energy 2	Extraneous energy from the burning of additional combustion aids.
H_2O elementary analysis	The percentage of water of the fuel sample determined by elementary analysis
HCl	The titrated quantity of 0.1 N hydrochloric acid (titration of distilled water with which the decomposition vessel was washed out after the experiment)
Ho (raw)	The specific gross calorific value of the combustion sample in delivery state
Ho (an)	The specific gross calorific value of the combustion sample in the “analytic moist reference state”
Ho (waf)	The specific gross calorific value of the combustion sample in the “water and ash free” reference state
Hu (raw)	The specific heat value of the combustion sample in delivery state
Hu (an)	The specific heat value of the combustion sample in the “analytic moist reference state”
Hu (waf)	The specific heat value of the combustion sample in the “water and ash free” reference state
Hydrogen (an)	The percentage of hydrogen in the “analysis moist” reference state
Hydrogen (raw)	The percentage of hydrogen in delivery state
Hydrogen (waf)	The percentage of hydrogen in the “water and ash-free” reference state
Hygr. moisture (an)	The percentage of hygroscopic moisture in the “analysis moist” reference state
Hygr. moisture (raw)	The percentage of hygroscopic moisture in the delivery reference state
Na_2CO_3	The quantity of sodium carbonate solution (in accordance with DIN 20 ml 0.05 N) added in the decomposition vessel
NaOH	The titrated quantity in ml (0.0866 N)

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Nitrogen (an)	The percentage of nitrogen in the fuel sample in the “analysis moist” reference state
Q sulfur	Heat of solution from the formation of sulfuric acid
Q nitrogen	Heat of solution from the formation of nitric acid
Sulfur (an)	The percentage of sulfur in the fuel sample in the “analysis moist” reference state
Sulfur (raw)	The percentage of sulfur in the fuel sample in the delivery state
Surf. moisture (raw)	The percentage of approximate moisture in delivery state
Total water (raw)	The percentage of water in the fuel sample in delivery state
Volatiles (raw)	The percentage of volatile components in delivery state
Volatiles (an)	The percentage of volatile components in the “analytic moist” reference state
Volatiles (waf)	The percentage of volatile components in the “water and ash free” reference state

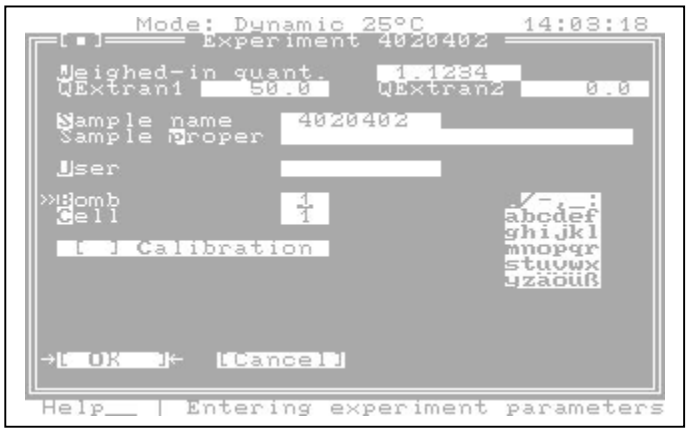
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10 Experiment simulation

In many cases it is helpful to reconstruct gross calorific value experiments or to calculate possible results of experiments without actually performing the combustion experiment. With the aid of the *Simulation* dialog box in the *Experiments* menu, the calorimeter system will simulate experiments based on the data you enter.

This option is especially useful if you accidentally perform a calibration instead of a determination of gross calorific value or vice-versa. By using the increase in temperature of the incorrectly interpreted measurement, it can be corrected by simulation.

①
In the *Experiments* menu, open the *Simulation* dialog box.



entering experiment parameters

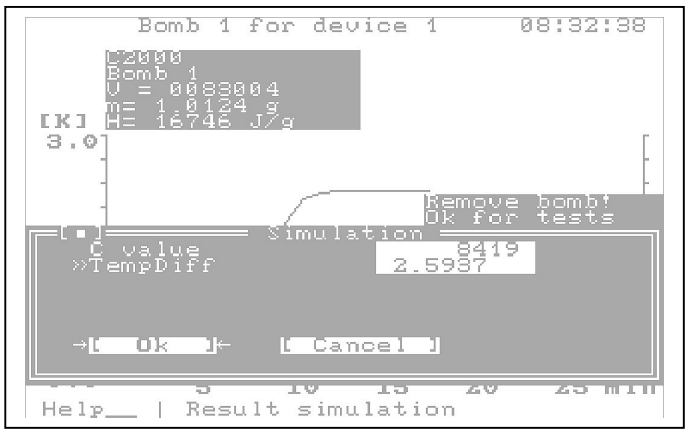
②
Use *Tab* to move the cursor to the input boxes and enter the sample data with which the simulation is to be performed.

③
If you have confirmed the data with *OK*, a dialog box appears in which to enter the simulation parameters.

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④

entering simulation parameters



You must enter the following parameters:

C value	The heat capacity of the calorimeter system
TempDiff	The temperature difference to be used for the simulated combustion

⑤

Confirm the dialog box with *OK*. You can post-process the simulated experiment in the normal manner with the *Evaluation* dialog box (see Chapter 9 “Evaluation of gross calorific value determinations”) or you can convert the result into the desired reference state.

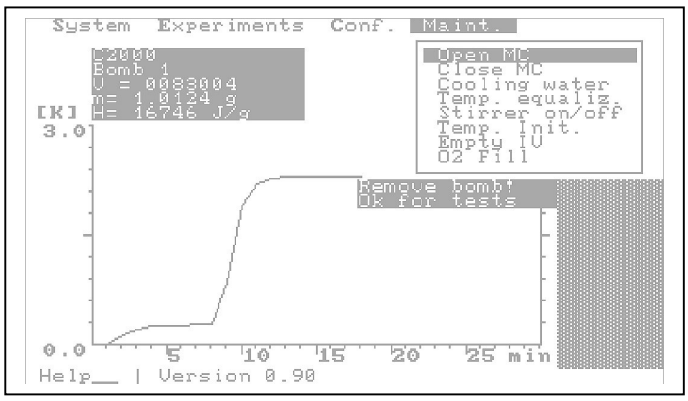
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11 Care and maintenance

11.1 Maintenance menu

The maintenance menu offers you the option of performing a series of system functions manually in the event of a malfunction. The commands of the Maintenance menu can only be performed if the measuring cell is in the Waiting mode.

maintenance menu



The functions *Open MC*, *Close MC*, and *O_2 fill* are activated by the corresponding menu command and are automatically terminated. The menu item in question is locked while the function is being performed.

Open MC	Opens the measuring cell cover
Close MC	Closes the measuring cell cover
Cooling water	Water flows through the outer vessel and the cooling water temperature is displayed in a window. The check can be terminated manually with *OK* or will be terminated automatically after 5 minutes.
Temperature Equalization	Water flows through the outer vessel and regulation is activated. The current target value (25 or 30 °C) is used for this process. The actual value of the temperature is displayed in a window. To end the check, press *OK*.
Stirrer on/off	The stirrer is turned on and off.
Empty IV	Water is pumped out of the inner vessel. Emptying is ended by time control (default: 90 sec).
Temp. Init.	The temperature measurement is reinitialized.

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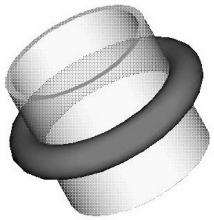
O_2 fill

A decomposition vessel that has been suspended in the measuring cell cover is filled with oxygen. The measuring cell cover will be closed for the duration of the filling process. The procedure ends automatically about 60 seconds after the cover is opened.
If no decomposition vessel is in place, the oxygen valve will be opened for one second.
The procedure is displayed in the process window.

In order to ensure problem-free operation over a long period of time, the maintenance tasks described below must be performed on the calorimeter system.

11.2 Inner vessel sieve

Remove the sieve element from the inner vessel and if there is visible dirt clean the inner vessel sieve in clean water or in an ultrasound bath.





After washing, place the sieve back on the inner vessel adapter.
Operating the device without a sieve element can cause dirt to accumulate in the valves, thus resulting in failure of the device.

11.3 Dirt trap in the water supply line

If the filter in the water feed line is partially blocked, the flow of water in the unit may be reduced. This will reduce the regulation quality of the system.

You can check this by using the maintenance menu item *Temperature* .
If the flow rate is normal (between 60 and 70 litres per hour), the target value currently set (25 °C or 30 °C) will be reached in a short time. The temperature tolerance is ±1 °C. When carrying out this check, it is important that the cooling water temperature (at the input to the unit) is within the guidelines given below. In the info-window, the duration of the last filling operation (FT) and the flow quantity (F) calculated from it are shown.

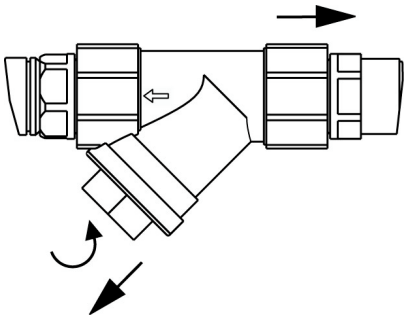
Guidelines
- Cooling water temperature 18 °C to 20 °C: working temperature 25 °C
- Cooling water temperature 23 °C to 25 °C: working temperature 30 °C

In addition, the time to fill the inner vessel with water may be extended. If the upper time limit of 240 s is exceeded, the system will show an error message and the measurement will be broken off. (See also Section 12 „Troubleshooting“)

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The dirt trap in the water supply line can be cleaned by counter-purging.
To do this, suspend the line from the calorimeter and purge it with reverse flow.

counter purging



open the dirt trap

If there is a heavy accumulation of dirt, open the dirt trap and clean the sieve element in an ultrasound bath.

11.4 Maintenance for the water circuit

stabilizer to avoid the formation of algae

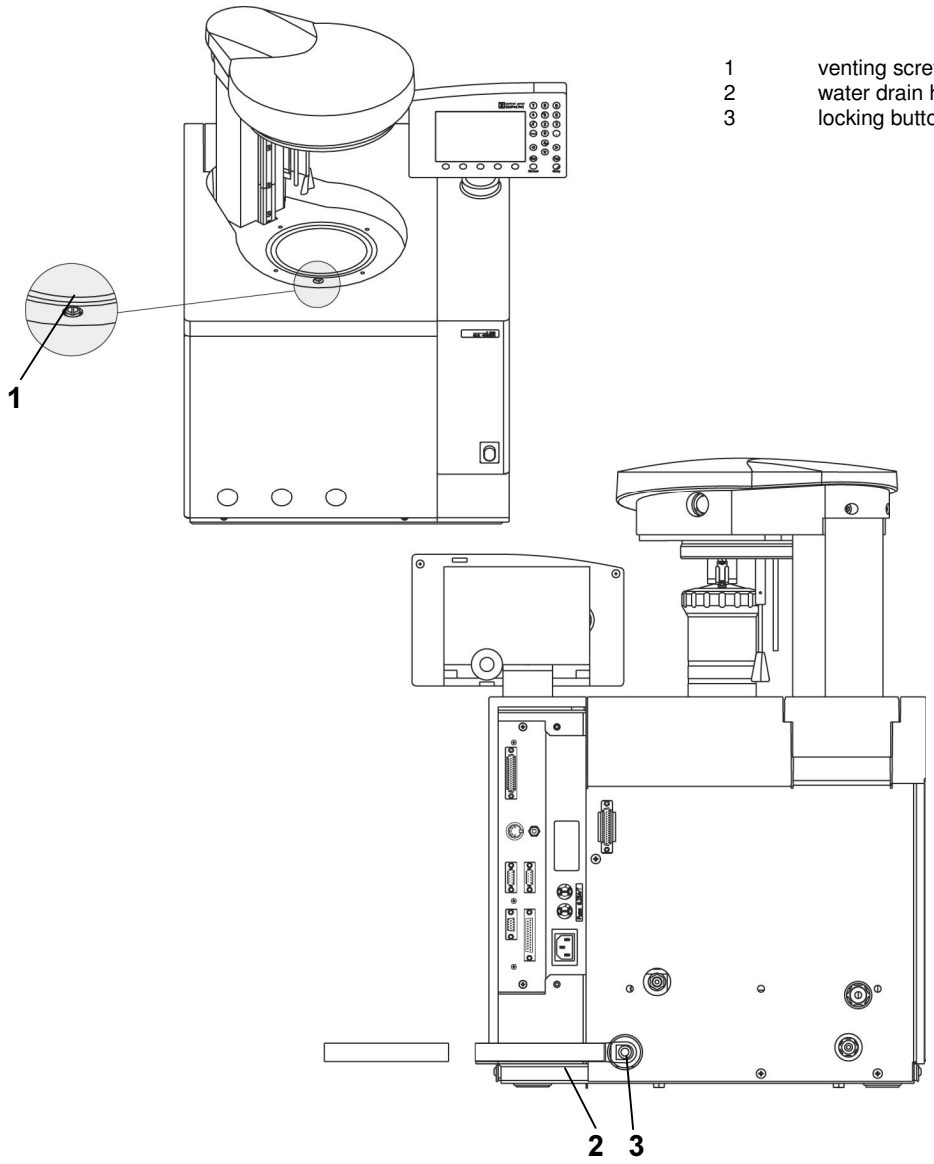
For non-continuous operation with cooling by supply line water (individual measurements separated by relatively long interruptions) a stabilizer must be added to the water circuit to prevent the formation of algae (Aqua-Pro), especially if the device will not be used for long periods of time.
To do this, start a measurement without placing a sample in the decomposition vessel and then manually activate the *Cancel* function key during the preliminary experiment (after filling the inner vessel).
The measuring cell cover opens and the water is pumped in the inner vessel.
While the water is being pumped out, add about 4 ml of Aqua-Pro to the inner vessel.

emptying the device

If the device will not be operated for a relatively long period of time, it is a good idea to completely empty the water circuit of the calorimeter.
The water must also be discharged before the device is transported.

To do this, place the water drain hose on the water drain adapter and open the venting screw completely.
To set the hose in place or to remove it, press the locking button on the water outlet connection.
When emptying a cooler unit, please make note of the corresponding Operating Instructions.

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1 venting screw
2 water drain hose
3 locking button

venting screw used to empty the system

insert the water drain hose into the water drain adapter

If the calorimeter has not been operated for quite a long time and the water circuit was not emptied, you can use the menu item *Cooling water* in the Maintenance menu to purge the system.
The *Temp. equaliz.* menu item results in overall temperature equalization for the calorimeter, which is particularly advantageous for the following messages.

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11.5 Changing the O_2 seal

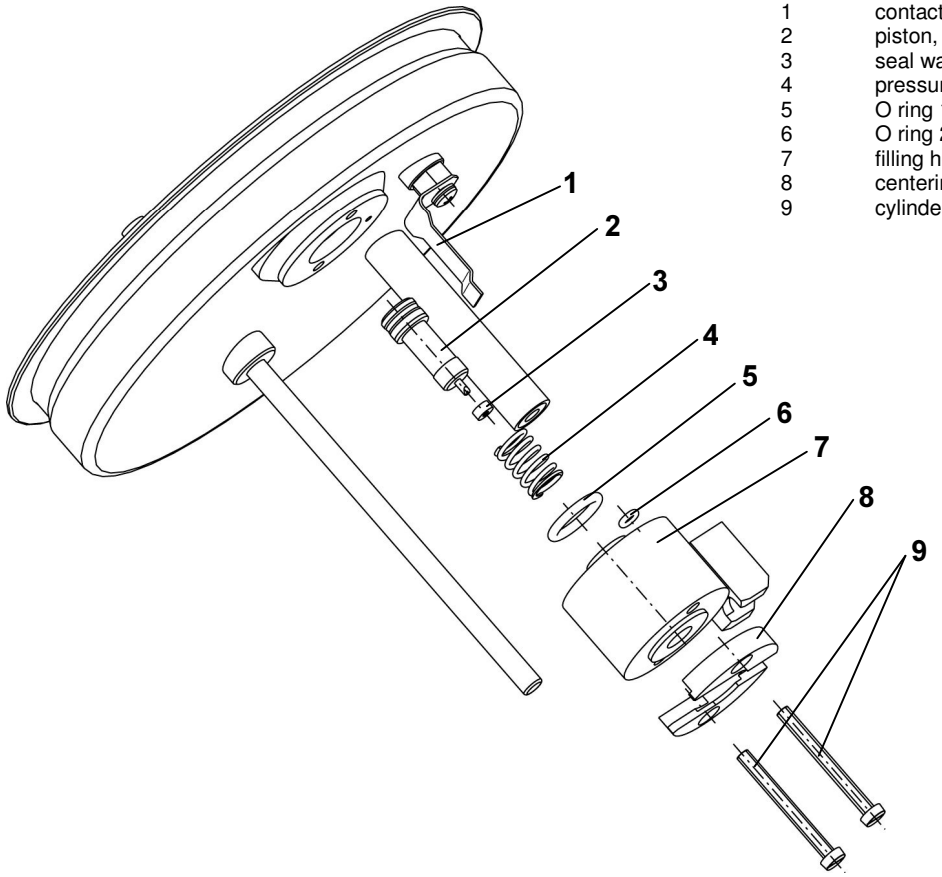
If a faulty seal is noted while the decomposition vessel is being filled with oxygen (loud hissing noise), the O_2 seal washer on the filling piston must be replaced.
Faulty seal during the filling process may also result from the decomposition vessel not being fitted snugly in place. Before checking the seal, make sure the decomposition vessel is snugly in place. (Maintenance menu *O_2 fill*)
You can remove an old badly split seal from the piston with tweezers.

To install a new seal, place it in the depression of the decomposition vessel (the seal surface). Now suspend the decomposition vessel into the filling head of the measuring cell cover and check to make sure it is properly positioned.
In the Maintenance menu, activate the item: *O_2 fill.*
The seal washer is now located on the piston capillary.

At the end of the filling process, remove the decomposition vessel from the filling head and release the pressure on it manually.

11.6 Changing the O_2 filling piston

If it should become necessary to replace the filling piston or seal elements, or if it is difficult to remove the seal ring on the piston, the filling head can be disassembled as follows:



1 contact spring
2 piston, complete
3 seal washer
4 pressure spring
5 O ring 11 x 2
6 O ring 2 x 1.6
7 filling head
8 centering ring
9 cylinder screw

disassembling the filling head

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①
Loosen the cylinder screws with a straight-bladed screwdriver.

②
Remove the centering ring together with the filling head, pressure spring and piston. Be careful with the O rings that are now loose.

③
Push the pressure spring onto the new piston or replace the seal washer and insert both parts into the filling head.

The remainder of the assembly is the same as disassembly in reverse order.

Please note: When reassembling the pieces, make certain the position of the filling head is correct. The seals must be aligned with the opposite side of the inner cover.



After reassembly, check the seal of the system with the *O_2 fill* item in the Maintenance menu.
If the assembly was performed correctly, you will hear only a brief hissing noise.

11.7 Decomposition vessels



On maintenance of decomposition vessels, read the C 5010/C 5012 Operating Instructions.

11.8 Notes on cleaning

When cleaning IKA® devices, use only these cleaning agents that are approved by IKA®:

Type of dirt	Cleaning agent
• Paint	Isopropanol
• Construction materials	Water with detergent, isopropanol
• Cosmetics	Water with detergent, isopropanol
• Food	Water with detergent
• Fuels	Water with detergent
• Substances not named	Please inquire with IKA®

Note:
Electrical devices must not be placed in the cleaning agent for cleaning purposes.
Stainless steel parts can be cleaned with standard commercial stainless steel cleaning agents, but abrasive agents must not be used.
We also recommend that you wear gloves while cleaning.
The operator is responsible for performing appropriate decontamination measures if hazardous materials have been spilled on or in the device.
Before using another cleaning or decontamination method than the one recommended by the manufacturer, the user must verify with the manufacturer that the intended method will not damage or destroy the device.

If the power supply cord is replaced, one of comparable quality should be used.

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12 Troubleshooting

The C 2000 calorimeter system is subject to strict quality control during manufacturing. If improper functionality should nevertheless occur, you will find in the following section a series of malfunction situations and the appropriate measures for remedying the malfunctions. Most malfunctions are shown in the header of the display. In addition to or instead of this, a message window may appear that the user must acknowledge with the appropriate key/button in the control field. If your attempts to remedy errors and malfunctions are unsuccessful, please contact your authorized IKA® Technical Service Department.

12.1 Malfunction with message in the display

Message	Cause	Remedy
No assignment possible	The decomposition vessel cannot be assigned. The system displays the malfunction and interrupts the measurement. Other decomposition vessels have already been assigned to the prepared measurements.	Use a correctly assigned decomposition vessel.
B Bomb “x” not found	A decomposition vessel was detected whose number is not reported. The system displays the malfunction and interrupts the measurement.	Report the decomposition vessel.
	Incorrect number detected.	Check the code ring on the decomposition vessel for incorrect position or damage. In order to continue the measurement series you may have to turn off detection of the decomposition vessel. (*Conf./Settings/Bomb ID*). If the function cannot be restored, please contact the Technical Service Department.

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Message	Cause	Remedy
Bomb “x” already assigned	The decomposition vessel is already assigned to a measurement in progress. The measurement procedure encountered interference. The system displays the malfunction and interrupts the measurement that is starting.	Turn the calorimeter off and back on.
Cover not closed/open	The measurement cell cover is not opening or closing completely. The system displays the malfunction and interrupts the measurement.	Try to open the measuring cell cover manually and then close it again. To do this, in the Maintenance menu execute the commands *Close MC* and *Open MC*. If you cannot perform the function, please get in touch with the Technical Service Department.
Water filling time exceeded	Fill state is not reached in the inner vessel within 240 sec. The system displays the malfunction and interrupts the measurement.	Repeat the experiment. Check • the external water supply for correct functionality • the flowthrough on the device To do this, select the *Cooling water* item in the Maintenance menu and measure the flowthrough per unit of time on the water outlet or check the value F in the info-window (see Section 11.3). **Correct values are between min. 60 l/h and max. 70 l/h** If the setting is not correct, please contact the Service Department. • the dirt trap in the water inflow line
No temp. increase	No increase in the temperature after electrical ignition. The system displays the malfunction and interrupts the measurement.	Check for • correct position of the cotton thread • a neat and tight contact of the ignition wire • the O_2 supply (30 bar) • the combustibility of the sample; it may be necessary to add a combustion aid.

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Message	Cause	Remedy
Preliminary experiment > 13 min. or **main experiment > 16 min.**	The combustion experiment is lasting too long. Isoperibolic measurements are stopped after 13 minutes in the "Stabilize" phase or after 16 minutes in the main experiment.	Check: • the operation of the stirrer drive (Maintenance menu: *Stirrer on/off*). • temperature equalization of the calorimeter to the working temperature with the Maintenance menu: *Temperature equalization* • The seal of the decomposition vessel (see C 5010/C 5012 Operating Instructions).
!! **Temperature: xx.xx °C**	Malfunction while recording the temperature The temperature display does not change any more.	Open the information window and look at the temperature displays *Temp1* and *Temp2*. If Temp1 or Temp2 do not change within 60 seconds, there is an error in temperature recording. Both values are constant: In the Maintenance menu, execute the *Temp. Init.* command. If you cannot reach the Maintenance menu or if you do but this does not provide any help, turn the calorimeter off and back on again. Only Temp2 is constant: In the Maintenance menu, execute the *Cooling water* command. After 60 seconds at the most, the displayed cooling water temperature should no longer be constant. If you cannot reach the Maintenance menu or if you do but this does not provide any help, use another method to check the cooling water temperature. The cooling water temperature must fall within the range of 5.xx°C to 28°C before it is possible to continue working.

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Message	Cause	Remedy
No regulation	Temperature regulation cannot reach a stable condition. The temperature of the cooling water is outside the permissible range (12 °C to 28 °C).	Check cooling water temperature and correct if necessary. Check the flow quantity and correct it if necessary (see Section 11.3).
Error water sensor	The calorimeter is incorrectly detecting a filled inner vessel. The system displays the malfunction and interrupts the measurement.	If there are any drops of water on the water sensor, remove them (see Section 11.6). If the water level in the inner vessel is higher than 3 cm, empty the inner vessel. If this does not resolve the problem, please contact the Technical Service Department.
Error ignition contact	This error message is displayed if ignition capability can no longer be ensured during a measurement. The experiment is stopped.	Check: • the ignition wire • the ignition wire fastening • the electrodes in the decomposition vessel (the electrodes must be tightened) • the contact spring on the filling head (see Section 11.6).
Sample rack invalid	The actual assignment of the sample rack does not agree with the assignment that is administered by the calorimeter.	See the Operating Instructions for the C 5020 sample rack.
Not enough memory	The total number of all measurements that the C 2000 can administer during one run is limited to about 240. This limit will be reached no sooner than after 50 hours of operating time. When it is reached, the message above appears.	Turn the device completely off. (in the System menu, menu item: ***Exit***) **Note:** This number has nothing to do with the storage capacity of the library.

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12.2 Malfunction without message in the display

Error	Cause	Remedy
Calorimeter in undefined state	Power failure	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, the procedure is as follows: • Turn the system off and back on again and start in the normal, proper manner. • If residual water is left in the inner vessel, discharge it with Maintenance menu *Empty IV.* • Remove the decomposition vessel, relieve the pressure and prepare it again.
Loud, permanent hissing discharge during the O_2 filling procedure (60 sec)	O_2 filling is not working	Examine: • the decomposition vessel to be certain it is snugly in place (see Section 7.4) • the condition of the seal on the filling piston (see Section 11.5) • the seal of the decomposition vessel C 5010/ 5012 Operating Instructions)
Incomplete combustion	Insufficient quantity of oxygen in the decomposition vessel.	Check the O_2 supply to the device (30 bar).
	Sample has low flammability	Use combustion aids.
Experiment cannot be started	When the decomposition vessel is suspended into the measuring cell, the Bomb ↓ display does not change to START.	Check the following items: • The decomposition vessel was not removed after the last measurement. • No experiment has been prepared. • The Maintenance menu is open. • There is a problem with the contact spring (see Section 11.6). • The ignition wire is defective.

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Error	Cause	Remedy
Water flow less than 60 l/h or larger than 70 l/h	The pressure output of the external water supply (cooler/ thermostat) is insufficient.	Check the output performance.
	The hose is laid higher than the level of the calorimeter.	Lay the intake or outlet line flat.
	The dirt trap in the intake line is plugged up.	Interrupt the water supply and remove the intake line from the calorimeter; then counter-purge the hose. If there is heavy accumulation of dirt on the sieve element in the dirt trap, clean it mechanically or replace it.
	Operation without the pressure reducer on the water faucet	Install the IKA® C 25 pressure control valve into the water supply line.
	C 25 pressure control valve is set incorrectly.	Adjust the output pressure to 1 - max. 1.5 bar.
	The internal pressure control valve is not working properly.	Contact the Technical Service Department.
No water flowthrough in device states "Stabilize; O_2 filling; Temp. equaliz."	Water supply is interrupted.	Check the water supply.
	The connections of the intake or outlet line on the external water supply may be mixed up.	Check the connections.
	Internal solenoid valve defective.	Go to Maintenance menu: *Cooling water* and check again for proper functionality. Contact the Technical Service Department.
Water flowthrough in the preliminary or main experiment	Venting screw is open.	Tighten the venting screw.
	Internal solenoid valve defective.	Contact the Technical Service Department.
The water level in the inner vessel is higher than about 3 cm.	The water outlet line is higher than the level of the calorimeter.	Check how the hose is laid. Pump out the water in the inner vessel via the Maintenance menu: *Empty IV*.
	Water in the inner vessel cannot be pumped out.	Contact the Technical Service Department.

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13 Accessories and consumables

13.1 Accessories

Ordering description

C 5010	IKA® decomposition vessel, standard
C 5012	IKA® decomposition vessel, halogen resistant
C 5010.4	Holding surface for disposable crucible
C 5010.5	Holding surface large crucible
C 5010.6	Venting button
C 5020	Sample rack
C 5030	Venting station
C 5040	CalWin®, calorimeter software
C 21	Pelleting press
C 25	Pressure control valve
C 29	Reduction valve
C 60	C 2000 calorimeter conversion kit
C 61	Electrode Set for Peters bomb
C 62	IKA® High-Pressure Decomposition Vessel
KV 500	Cooler unit

13.2 Consumables

Ordering description

C 710.4	Cotton thread, cut to length (500 pieces)
C 5010.3	Ignition wire, replacement (5 pieces)
C 5012.3	Pt ignition wire, replacement (2 pieces)
C 5003.1	Aqua-Pro bath stabilizer (30 ml)
C 4	Quartz dish
C 5	Set of VA combustion crucibles (25 pieces)
C 6	Quartz dish, large
C 710.2	Set of VA combustion crucibles, large (25 pieces)
C 9	Gelatin capsules (100 pieces)
C 10	Acetobutyrate capsules (100 pieces)
C 12	Combustion bag, 40 x 35 mm (100 pieces)
C 12A	Combustion bag, 70 x 40 mm (100 pieces)
C 43	Benzoic acid (NBS 39i, 30 g)
C 43A	Benzoic acid (100 g)
C 723	Benzoic acid in tablet form (50 pieces)
C 14	Disposable crucible (100 pieces)
C 15	Paraffin strips (600 pieces)

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14 Technical data

Measurement voltage / frequency	115 V 50 / 60 Hz 230 V 50 / 60 Hz
Power consumption	1.8 kW
Device fuses	2 x 6.25 AT; 230 V 2 x 15 AT; 115 V
Duty cycle	Continuous operation
Protection type according to DIN 40 050	IP 21
Protection class	1 (protective grounding)
Over-voltage category	2
Contamination level	II
Ambient temperature	20°C ... 25°C (constant)
Ambient relative humidity	80 %
Usage above sea level	2000 meters above sea level
Dimensions	440 x 450 x 500 (WxDxH)
Weight	30 kg
Measurement range	40,000 J
Measuring mode	Isoperibolic 25°C Isoperibolic 30°C Dynamic 25°C Dynamic 30°C
Isoperibolic measuring time	About 22 min
Dynamic measuring time	About 10 min
Oxygen operating pressure	30 bar
Oxygen test pressure	40 bar
Cooling medium	Water via line
Flowthrough quantity	min. 60 liters/hour max. 70 liters/hour
Water operating pressure Operation with thermostat (e.g. KV 500)	0,3 bar
Operation connected to water tap with Pressure Reducer C 25	1 ... max. 1,5 bar
Water tap test pressure	6 bar
Interfaces	2 x serial (RS232) 1 x parallel (Centronics) 1 x keyboard (DIN socket) 1 x sample rack 1 x external monitor
Connection for external monitor:	
Image frequency	115.74 Hz
Line frequency	33.333 KHz

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