IKA® Calorimeter System C 7000 with Cooler C 7002

OPERATING INSTRUCTIONS *EN/ USA*

Vers. 07 09.07 Reg.-No. 4343-01

CE – KONFORMITÄTSERKLÄRUNG ***DE***

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.

CE – DECLARATION OF CONFIRMITY ***EN***

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.

DÉCLARATION DE CONFORMITÉ CE ***FR***

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.

DECLARACION DE CONFORMIDAD DE CE ***ES***

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.

CE – DICHIARAZIONE DI CONFORMITÀ ***IT***

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.

Contents

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 7000 calorimeter system may only be used to determine the gross calorific value of solid and liquid materials in accordance with DIN 51900 and ISO 1928. For this purpose, use only original IKA® decomposition vessels C 7010 and C 7012. For further details please see the operating instructions of the decomposition vessels C 7010 and C 7012.

Operating requirements

The maximum amount of energy input into the decomposition vessel must not exceed **30000 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. Perform a check before every combustion to ensure there are no leaks combustion (please observe the operating instructions of the decomposition vessels C 7010 and C 7012, 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! This high-pressure decomposition vessel can only be used with the C 2000 calorimeter system.

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 7010 or C 7012 (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.

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.

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.

2 User notes

2.1 Notes on using the operating instructions

In this section you will learn how to make the most effective use of these Operating Instructions so as to be able to work safely with the calorimeter system.

The instructions given in Section 1 “For your Safety” must be followed without fail.

Studying Sections 1 ... 10

Work through Sections 1 ... 10 in numerical order.
Section 3 "Transport, Storage, Installation Location" is particularly relevant to system reliability and ensuring high accuracy of measurements. Section 4 describes the system components and Section 5 contains the basic principles of calorimetry.

Carrying out tests

Once you have carried out the procedures described in Section 6 "Commissioning", and Section 7 "Preparing and Carrying Out Measurements" the calorimeter is ready for use.
In Section 8 the evaluation of calorific value measurements and the possibility of test simulation are explained.
Section 9 gives important information on care and maintenance, and Section 10 explains the display messages and gives advice on troubleshooting and the correction of simple problems.

Information on accessories, consumables, and the technical data for the unit are given in Sections 11 and 12. Section 13 contains the index.

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.

2.4 System Features

The C 7000 calorimeter is used for the routine determination of the gross calorific value of solid and liquid substances. The system accessories ensure that it can be individually adapted to laboratory tasks (see also Section 11).

The system has the following important features:

- patented double-drying measuring procedure;
- short measurement time, ca. 3 minutes for one determination;
- well over a hundred determinations per day and calorimeter are possible;
- automated measurement reduces routine tasks;
- fully-integrated, independent, microprocessor-controlled calorimeter;
- film key panel provides convenient operation and protection against laboratory conditions;
- integrated fault detection with plain-text display;
- simple, well-proven operation;
- parallel port for direct connection of printer;
- memory for 100 tests;
- automatic acceptance of data from connected balance;
- calculation of calorific value under various conditions;
- four different calculation modes:
  standard, with and without titration,
  carbon, with and without titration;
- online data transfer to an external PC;
- automatic recognition of up to 8 decomposition vessels;
- maximum energy input from decomposition vessel: 30.000 J:
  this corresponds to a temperature increase in the decomposition vessel of about 25 K.

3 Transport, Storage, Installation Location

3.1 Transport and Storage Conditions

During transport and storage, the system must be protected against mechanical shock, vibration, dust deposits, and corrosive atmospheres. In addition, the relative humidity should not exceed 80%.

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 Installation Location

When installing the unit, observe the national and local regulations for the operation of pressure vessels that apply at the site selected.
To ensure high accuracy of measurement, a constant ambient temperature is an important precondition. For this reason, the chosen installation location should fulfil the following conditions:

- not exposed to direct sunlight;
- no drafts (e.g. not beside a window, door or air-conditioning vent);
- sufficiently far from heating radiators and other heat sources;
- room temperature must be between 18 °C and 30 °C: to ensure good measurement quality, there should be no variation in temperature;
- the system must be installed on a horizontal surface;
- If the C 7002 cooler is to be used, a water supply with a supply pressure of less than 9 bar is required near the installation location, or a suitable cold water supply (e.g. IKA® KV 500).

For operation of the system, a power supply corresponding to the typeplates of system components must be available at the installation location. An oxygen supply with a pressure gauge, which can supply 99.95% pure oxygen, quality 3.5 at a pressure of 30 bar is also required. A shut-off valve for the oxygen supply must be installed. Observe the instructions on handling oxygen given in Section 1 "For your safety”.

4 Description of System Components

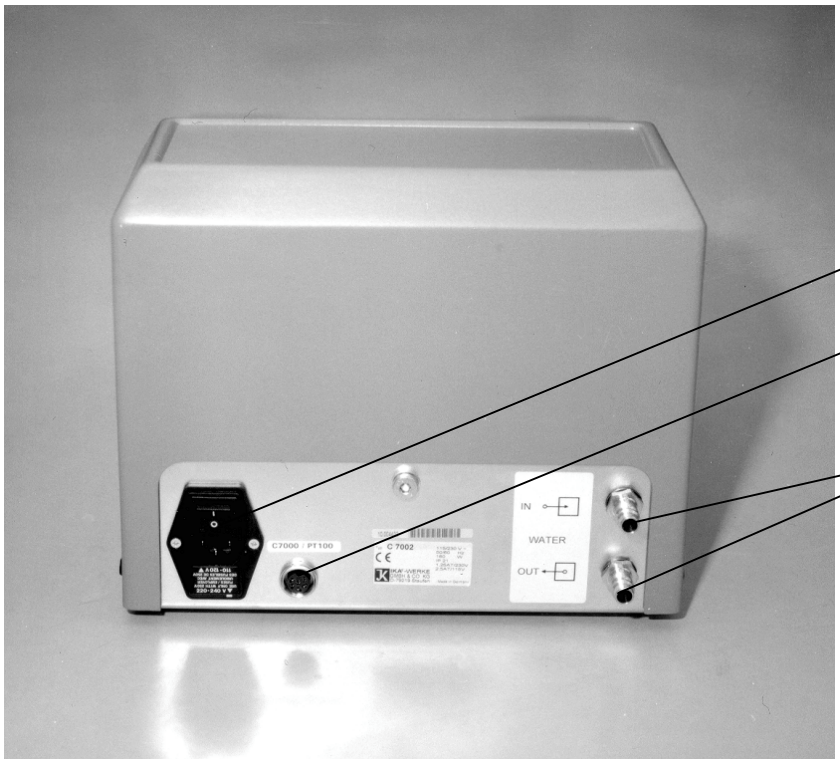
4.1 Calorimeter C 7000

C 7000
Front view

1	Display
2	Key pad
3	Control section

C 7000
Rear view

1	Contrast control for display
2	Typeplate
3	Connection for cooler C 7002
4	PC connection, RS 232
5	Balance connection RS 232
6	Printer connection, Centronics
7	Fuses
8	Socket for mains cable with main fuse



Cooler C 7002
Rear view

1	Mains connection with main fuse
2	Connection to C 7000
3	Cooling water connections with quick-connect couplings

The cooler is used to cool the decomposition vessel quickly after a test. For this purpose, the cooler C 7002 is connected using the cable supplied to the calorimeter C 7000 (see illustrations Cooler C 7002 Rear view, Position 2, and C 7000 Rear view, Position 3). During the cooling operation, the decomposition vessel is cooled to the temperature of the measuring cell. For accurate calibration and measurement, this condition must be fulfilled.

For cooling, the decomposition vessel is placed between the cooling jaws. Peltier elements are used for cooling. They are controlled completely automatically. Water is employed for removal of heat. Internal controls ensure the water consumption is kept especially low. In stand-by mode, the water is switched off automatically. The cooler does not start ist pre-cooling phase until a main test has started.

Please see Section 6.2 "Connection of Peripherals" for more information about installation of the cooler.

5 Principles of Calorimetric Measurement

To make a calorimetric measurement, a sample of a substance is burnt. In order to ignite the sample, energy must be supplied from an external source. An ignition wire, which is heated to glowing point by passing an electric current through it, performs this function. If the measurement is to be accurate, it is essential that the sample is completely burnt. For this reason, combustion takes place in an atmosphere of oxygen at a pressure of 30 bar.

5.1 Determining the Gross Calorific Value

In a calorimeter combustion takes place under defined conditions. A weighed sample of a substance is placed in the decomposition vessel, the sample is ignited, and the increase in temperature of the decomposition vessel is measured. The gross calorific value of a sample is calculated from:

- temperature increase of the decomposition vessel
- heat capacity (C value) of the calorimeter system
- mass of the fuel sample
- heat energy that is released by burning the ignition aid and auxiliary fuel, and also by the formation of sulphuric and nitric acids (external energy).

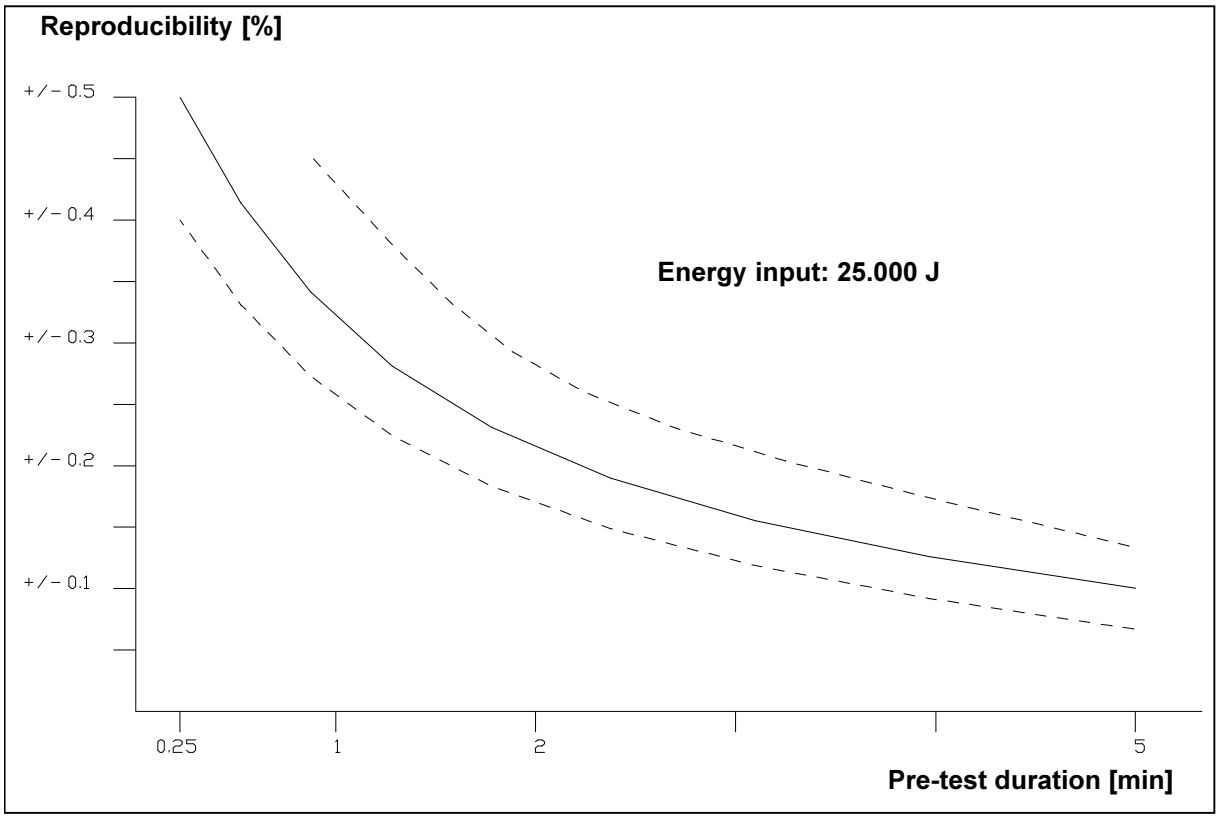
Test conditions

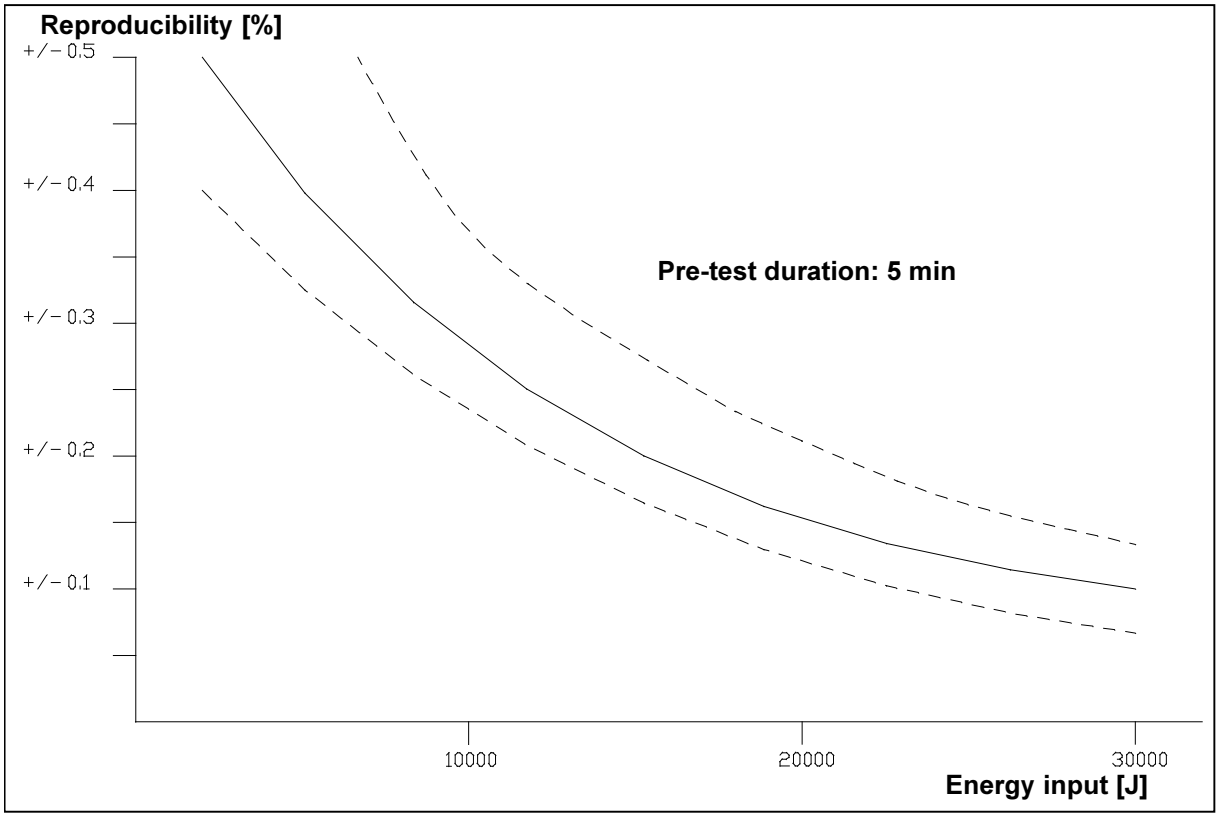
To optimise the combustion process, the decomposition vessel is filled with pure oxygen (99.95%). The pressure of the oxygen atmosphere in the decomposition vessel is 30 bar. The fuel sample is weighed to an accuracy of 0.1 mg using an analytical balance.

Precise determination of the calorific value of a substance demands that combustion takes place under precisely defined conditions. The relevant standards make the following assumptions:

- any water contained in the fuel, and any water formed by the combustion of compounds containing hydrogen in the fuel is present in a liquid state after combustion;
- no oxidation of atmospheric nitrogen has taken place;
- gaseous products present after combustion consist of oxygen, nitrogen, carbon dioxide and sulphur dioxide;
- solids can also be formed (e.g. ash).

Frequently, other combustion products, not foreseen by the standards, are formed. In such cases, analyses of the sample material and the combustion products are required to supply data for correction calculations. The standard gross calorific value is then determined from the measured value and the analysis data.


Reproducibility [%]
+/- 0.5
+/- 0.4
+/- 0.3
+/- 0.2
+/- 0.1
Energy input: 25.000 J
0.25
1
2
5
Pre-test duration [min]


Reproducibility [%]
+/- 0.5
+/- 0.4
+/- 0.3
+/- 0.2
+/- 0.1
Pre-test duration: 5 min
10000
20000
30000
Energy input [J]

Acid correction

Almost all substances that are tested contain some sulphur and nitrogen. Under the conditions employed for calorimetric measurements, sulphur and nitrogen burn to SO_2, SO_3 and NO_x. In combination with water from combustion and moisture from the sample, sulphuric acid and nitric acid are formed and heat of solution is developed. The influence of the heat of solution must be taken into account when calculating the standard calorific value.

To obtain a defined end condition, and to quantitatively record all acids, ca. 5 ml of distilled water or other suitable absorption liquid are added to the decomposition vessel. The gases resulting from combustion form acids with this absorption liquid and any water resulting from combustion. After combustion, the decomposition vessel is rinsed thoroughly with distilled water, to collect any condensate that has settled on the walls of the vessel. The acid content of this solution can be determined using suitable equipment.
You can obtain details of suitable equipment for the purpose from IKA® or from your authorised dealer.

5.3 Complete Combustion

To determine the gross calorific value correctly, it is of fundamental importance that the sample has burnt completely. After a test, the crucible and all solid residues must be examined for signs of incomplete combustion.

Solids

Normally, solids can be burnt directly in powder form. Substances that burn very fast (e.g. benzoic acid) must not be burnt in a loose form. Such substances tend to explosive combustion, and complete combustion cannot then be guaranteed. In addition, the decomposition vessel could be damaged. These substances must be pressed to form tablets using a special press (C 21 pelleting press, see Accessories) before testing.

Substances with low inflammibility

Substances with low flammability (substances with a high mineral content or a low calorific value) can often only be burnt completely with the aid of combustion capsules or combustion bags (C 10/C 12, see Accessories). The use of liquids to aid combustion, for example paraffin, is also possible.

Liquids, highly volatile substances

Most liquids can be weighed directly in the crucible, but highly volatile substances should be filled into combustion capsules (gelatine capsules or acetobutyrate capsules, see Accessories) and burnt together with the capsules.

All combustion and ignition aids (e.g. cotton threads) must be burnt completely. If anything remains unburnt, the test must be repeated.

Halogens

Halogen-containing substances can cause corrosion of the decomposition vessel. For such cases, decomposition vessel C 7012 should be used.

6 Commissioning

Once the components of the C 7000 calorimeter have been unpacked and brought to the location chosen for the instrument (see Chapter 3, Section 3.2 "Installation Location"), the mains lead and peripherals can be connected.

6.1 Connecting the mains lead

Check that the voltage given on the typeplate of the calorimeter corresponds to your mains supply, then plug the mains lead into the socket on the rear of the calorimeter and to the mains socket.

6.2 Connecting peripherals

When connecting peripherals, the mains switch of the calorimeter must be switched off.

For all connections to the calorimeter, use only the interface cables supplied by IKA®.

Connecting a balance, printer or PC
For connection of a balance, a printer or an external PC, there are sockets on the rear of the unit marked **Balance**, **Printer** and **PC** (see Section 4.1, illustration C 7000 Rear view).

Now connect the peripherals you will be using.

Connecting the cooler C 7002
Connect the C 7002 cooler as follows:

①
Check that the voltage given on the typeplate of the cooler corresponds to your mains supply, then plug the mains lead into the socket on the cooler and to the mains socket.

②
There are two hoses with quick-connect couplings included with the cooler. Connect them to the *In* and *Out* connections on the rear of the cooler.

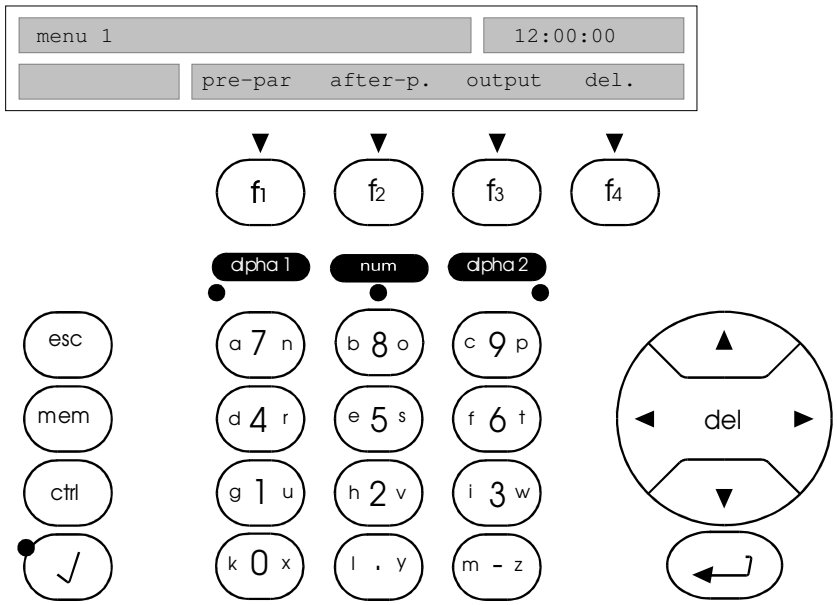
③
Connect the other end of the feed hose (*In*) to a water supply, water tap or to a cooling water supply (e.g. IKA® KV 500) and secure the connection. Take the drain hose (*Out*) to a suitable sink and secure it there.

④
Use the cable provided to connect the cooler and calorimeter at the appropriate sockets on the rear of each unit (see Chapter 4 "Description of System Components").

The C 7002 cooler is now connected to the C 7000 calorimeter.

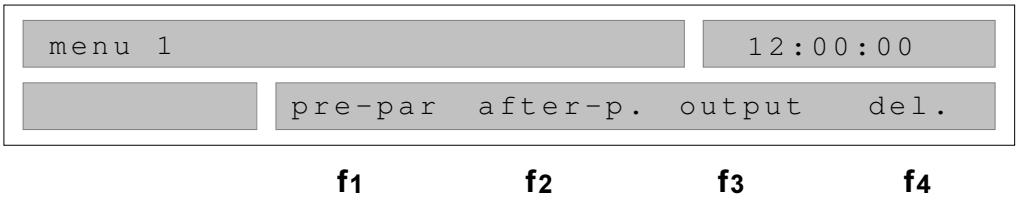
The key pad has the following buttons:

Film key pad and display



1. **Function buttons *f1*, *f2*, *f3*, *f4***: The functions of the buttons *f1, f2, f3, f4* are always indicated by the text that appears in the lower right zone of the LCD display. The relevant text in the display is directly above its button:

Relationship of function buttons to LCD display



With this arrangement, the function buttons can be used to carry out the following actions:

- selection of sub-menus in the three main menus;
- selection of a value from a list of preset values;
- switching the alpha-numeric keypad between letters and figures.

2. ***esc* button**: The *esc* button can be used to exit any menu without storing data. After *esc* has been pressed, the display shows *menu 1*.

3. ***mem* button**: all data for a menu level are stored by pressing the *mem* button.

After completing an entry with the ↵ button (see below), the enquiry *(mem) = save* appears for a few seconds. During this period, operating the *mem* button has no effect. Wait until *(mem) = save* has gone out before pressing *(mem) = save*.

4. ***ctrl* button**: the *ctrl* button is used in conjunction with number keys to enter special functions.

MENU STRUCTURE FOR C 7000 CALORIMETER

- **menu 1**
  - f1 **pre-par**
    Entry of pre-test parameters
  - f2 **after-p.**
    Entry of post-test parameters
  - f3 **output**
    Data output
    - f1 **LCD**
      Shows test data on LCD-display
    - f2 **free**
      Information about number of tests still free
    - f3 **table.**
      Prints tabular summary of tests
    - f4 **samp.**
      Sends test data to printer
  - f4 **del.**
    Erases test data
- **menu 2**
  - f1 **manual**
    For manual data entry
    - f2 **heat-cap.**
      Manual entry of C value
    - f4 **measure**
      Manual measurement (simulation)
  - f2 **edit.**
    For changing post-test parameters
  - f3 **calib.**
    For entering calibration data
- **menu 3**
  - f1 **HDInit**
    Initializes the hardware
    - f1 **ext. PC**
      Interface parameters for PC connection
    - f2 **balance**
      Interface parameters for balance connection
    - f3 **date**
      For changing the date
    - f4 **time**
      For changing the time
  - f2 **STInit**
    Initializes the software
    - f1 **realt.**
      Selection of interval for results output
    - f2 **calcul.**
      For setting calculation mode for gross and net cal. values
    - f3 **unit**
      Selection of units for results
    - f4 **pre. time**
      Selection of pre-test time
  - f3 **IGN time**
    For setting the ignition time

- Sub-menu *STInit*

realt.
The parameter *realt.* (real-time print out) is used to set the time interval in seconds for sending measured values to a printer. The values 0, 3, 12, or 24 seconds can be set. The preset value is 0 seconds . If the value 0 is selected, the transmission of readings to a printer) during a test is switched off. The presentation of data on the LCD-display is unaffected by the setting of this parameter.

☞ **If a printer is not connected, the parameter *realt.* must be set to 0.**

calcul.
In this sub-menu, you determine the calculation mode. Possible settings are:

std - t	standard without titration
std + t	standard with titration
coal - t	carbon without titration
coal + t	carbon with titration

The calculation mode must be set *before* making a measurement, because subsequent conversion to a different calculation mode is not possible.

☞ The presetting is *standard without titration*. The calculation modes *standard* and *coal* differ in that, when *coal* is set, the calculation of gross and net calorific values is carried out under various conditions. If *with titration* is selected, the energy of acid formation is determined by titration.

unit
The results for gross and net calorific values can be calculated and presented in the output protocol in several different units. The corresponding settings for the parameters *unit* are:

joule	J/g
BTU	BTU/lb
kWatt	kWh/kg
cal	cal/g

The preset unit is J/g.

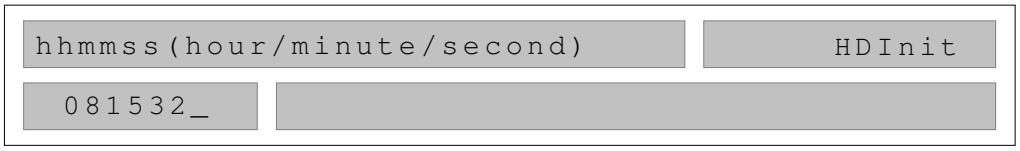
pre. time
In this menu, one of four values can be set for the pre-test time:
15 seconds
60 seconds
120 seconds
300 seconds
The preset value is *120 seconds*.

☞ **The longer the pre-test time, the more accurate the result.**

- *IGN time*
The setting of *IGN time* determines the ignition time in milliseconds for the fixed ignition wire. The works setting is 1000 ms, which is normally entirely adequate to be sure that the cotton thread or the combustible crucible has ignited.
The preset ignition time should only be changed if the cotton thread or the combustible crucible do not burn properly, or if platinum ignition wire is used. In these cases, the ignition time should be increased in successive steps of not more than 100 milliseconds. However, if combustion is incomplete, first check the connections of the ignition wire to the electrodes in the decomposition vessel.

③

Use function key *f4* to call up the sub-menu *time*; by doing so you have reached the lowest Level of the menu structure. The LCD-display is now prepared for entering the time.



④

Press the *del* button six times to erase the time currently entered (here: 8 h: 15 min: 32 sec.), which at the same time moves the cursor to the left. Use the alpha-numeric keys to enter the correct time in the format given in the first line (hhmmss). This format contains only figures and no blanks.

⑤

Confirm with the ↵ button. In the second line, the request *(mem)* = *save* appears. Wait until the request disappears again.

⑥

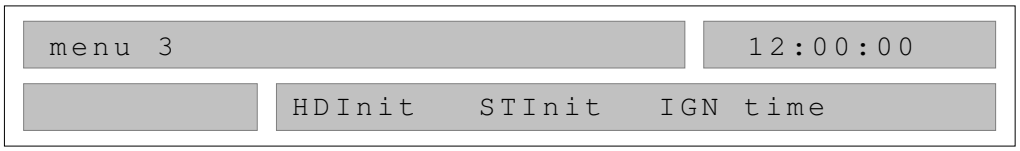
Then press the *mem* button to store your new entry. With the *esc* button, you can now go back to *menu 1* in the main menu. If you do not want to store the new time, press the *esc* button without first using the *mem* button. That will take you back to *menu 1* in the main menu without accepting the newly entered time.

Setting the time interval for output of results

From the menu-structure diagram in Section 6.4, you can see that the time interval for results output (*realt.*) is set in sub-menu *HDInit* of main menu *menu 3*. To set the time interval, proceed as follows:

①

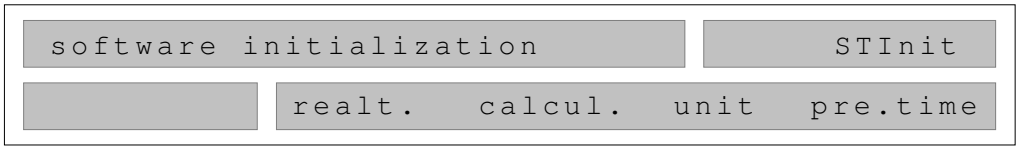
Operate the button ▲ once, or press ▼ twice. *menu 3* appears in the LCD-display.



f1 **f2** **f3**

②

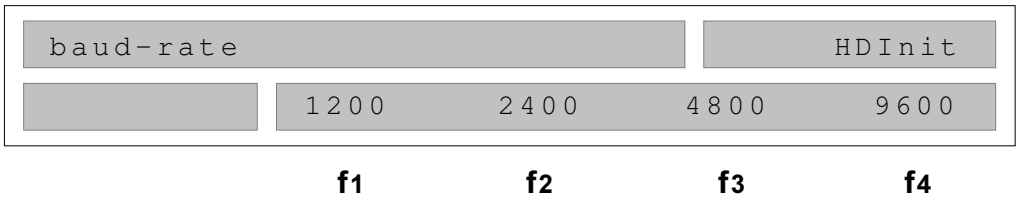
Press the function key *f2* to go to sub-menu *STInit*. The system parameters for software initialization appear in the LCD-display.



f1 **f2** **f3** **f4**

③

Use function key *f2* to call up the sub-menu *balance*; by doing so you have reached the lowest Level of the menu structure. The LCD-display is now prepared for selecting the required *baud-rate*.



④

Use one of the function keys *f1*, *f2*, *f3* or *f4* to select the desired value. Your choice is then shown in the left-hand zone of the second line of the LCD-display.

⑤

Press the ↵ button. The selection possibilities for *data-bits* are shown in the LCD-display.

⑥

In the same way as described above, entered the required values for *data-bits*, *stop-bits*, *parity* and *balan.*

⑦

Confirm with the ↵ button. In the second line, the request *(mem)* = *save* appears. Wait until the request disappears again.

⑧

Then press the *mem* button to store your new entry. With the *esc* button, you can now go back to *menu 1* in the main menu. If you do not want to store the new data, press the *esc* button without first using the *mem* button. That will take you back to *menu 1* in the main menu without accepting the newly entered data.

6.8 Switching off

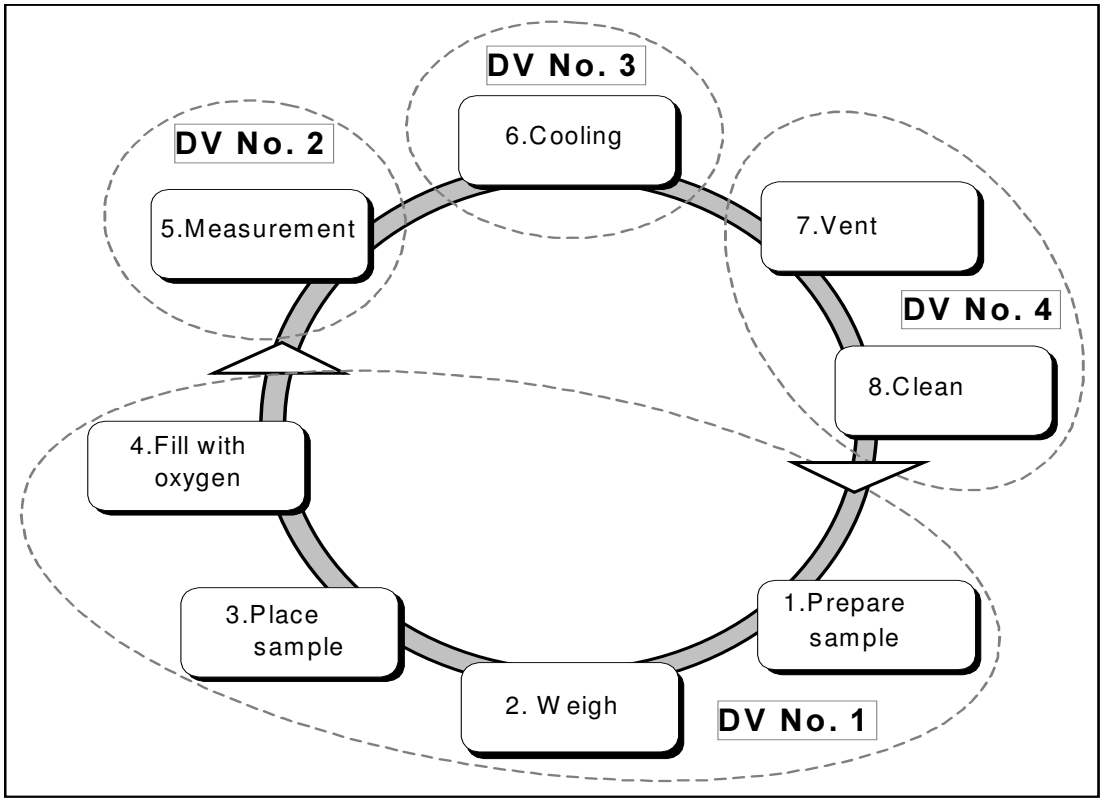
☞ **Only switch the calorimeter off when neither a test nor calibration are taking place. To prevent loss of data, the LCD-display should show one of the main menus, *menu 1*, *menu 2* or *menu 3*.**

Switch off the calorimeter and the cooler by operating their mains switches.

7 Preparation and Carrying Out Measurements

The term "measurements" as used here means both measurements for calibrating the instrument (calibration measurements) and measurements made to determine a gross calorific value. The difference lies mainly in the evaluation of the results, preparation and execution are almost identical for both types of measurement.

A complete test procedure comprises the following operations and procedures (for detailed information about the individual steps, please see further sections in this chapter):



①

Prepare sample
For example, homogenise sample material, press to form pellets, dry

②

Weigh
Weigh the sample in a crucible to an accuracy of 0.1 mg

③

Place sample
Place the sample in the decomposition vessel and close the vessel .

④

Fill with oxygen
Fill the decomposition vessel manually with oxygen at 30 bar, quality 3.5, using the IKA® C 48 oxygen filling station.

If several decomposition vessels are used in a measuring cell, the heat capacity of the system must be determined by calibration with each decomposition vessel. A decomposition vessel must only be used in a measuring cell in which it has been calibrated.

Calibration must be carried out under conditions similar to those that will be used for subsequent tests. If combustion tests are to be carried out with absorption liquid (e.g. distilled water or a solution) in the decomposition vessel, then calibration should be carried out with the same quantity of the same substance.

Recommendations for calibration

- At a starting temperature of 25 °C, the C 7000 calorimeter has a maximum temperature increase of 25 K. For accurate results, a combustion test should yield a temperature increase of approximately 20 Kelvin. If you do not use benzoic acid for calibration, the quantity of material should be adjusted to meet this condition.

- For calibration, use benzoic acid (accessory C 723) compressed to a pellet. Weigh out approximately 1 g, this corresponds to a temperature increase of ca. 20 K.

- Calorific-value determinations must yield approximately the same temperature increase as calibration. The optimum sample quantity must be found by making several tests if necessary.

- The pre-test time should be at least 120 seconds.

- When calibrating to DIN, the C value is specified as the average of at least 5 calibration tests. Individual results should only be used in calculating the average if the difference between the highest and lowest values (scatter) is not more than 0.4% of the average.
  If the scatter is greater, then the result which is farthest from the average is discarded first. A sixth calibration test must now be carried out. The result of this determination must not deviate by more than 0.2% from the average of the four valid results. For further details, please see the relevant standard.

- In the calorimeter, the C value from the last calibration of a decomposition vessel is stored in memory. If several calibration tests are used to determine the C value (e.g. when calibrating to DIN), the calculated average C value for this decomposition vessel must be entered manually before carrying out a calorific value determination.

Notes on the C value

Because a calorimeter can operate in a wide range of ambient temperatures, the C value of a system must be seen as **temperature dependent**. This fact must be taken into account both during calibration and during normal testing. The calibration values printed out use 25 °C as reference temperature. This applies also to manually entered C values. When carrying out combustion tests, the C value must be corrected to the current ambient temperature (temperature of the metal casing at the start of combustion). This also explains why the C value printed after a test does not agree with the value entered manually by the user.

7.2 Notes on samples

Decomposition vessels C 7010 and C 7012 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 7010 or C 7012.

The C 7000 calorimeter is a precision instrument for routine measurement of the gross calorific value of solid and liquid substances. Accurate measurement is only possible if the all the individual steps in a test are carried out with care. The procedure as it is described in Chapter 1 “For your safety” and in the sections below 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 7010 or C 7012 (danger of explosion). If you are burning **unknown samples**, leave the room

Solids

Normally, solid fuels can be burnt directly in powder form. Fast-burning substances (e.g. benzoic acid) must not be burnt in a 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)!

Fast-burning substances tend to explosive combustion. Complete combustion is then no longer certain. In addition, the inner wall of the decomposition vessel may be damaged. Such substances must be pressed to form pellets before they are burnt.

The IKA® C 21 pelleting press is suitable for this task.

Liquids

Most liquids can be weighed directly into the crucible. Liquids that are cloudy or with separable water must be dried or homogenised before weighing. The water content of such samples must be determined.

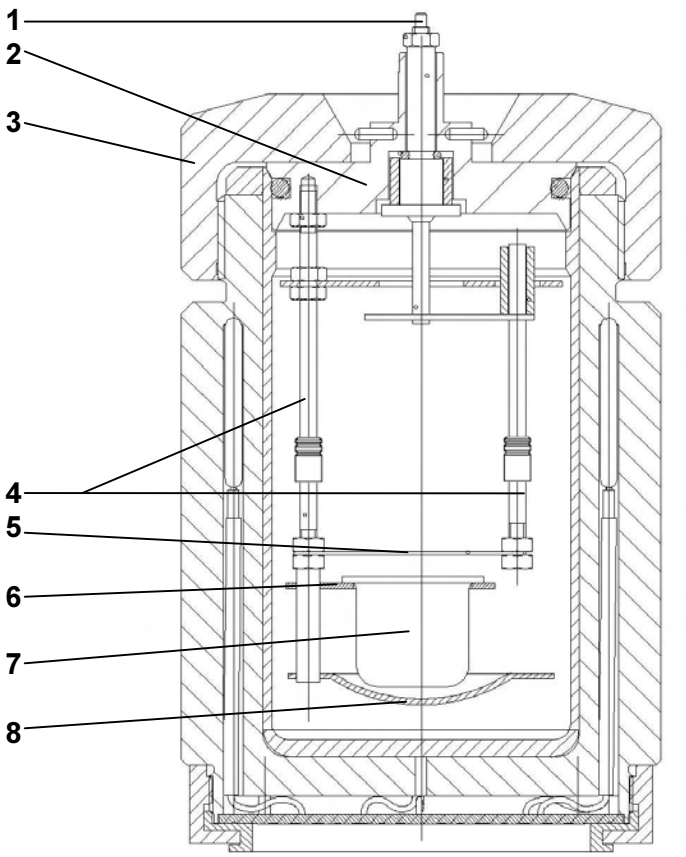
Highly volatile substances

For highly volatile substances, gelatine or acetobutyrate capsules (see accessories), which are filled with the fuel, must be used. The calorific value of the capsules must be known, so that the heat of combustion resulting from them can be taken into account as external energy.

Combustion aids

For low flammability substances and those with low calorific value, the capsules mentioned above, or combustion bags made of polyethylene (see accessories) are used. The C 14 combustible crucibles can also be employed.
Before the capsules or combustion bags are filled with the substance whose calorific value is to be determined, they must be weighed, so that from their mass and calorific value, the amount of external energy they contribute to combustion can be cal-

7.3 Preparation for measurement



Components of the decomposition vessel

1	Connection for filling with oxygen
2	Lid
3	Screw-on cover
4	Electrodes
5	Ignition wire
6	Crucible holder
7	Crucible
8	Support for combustible crucible

For protection against corrosion, the lining of the decomposition vessel is made of stainless steel. The decomposition vessel must be clean and dry, because all foreign materials, in particular water, affect the heat capacity and thus the results.
You can now load the sample into the decomposition vessel.

③

Weigh the sample (with combustible crucible if used).

As a rule, the quantity weighed should be such that the maximum permissible energy input of 30.000 J is not exceeded. Failure to observe this rule may result in damage to the calorimeter system.

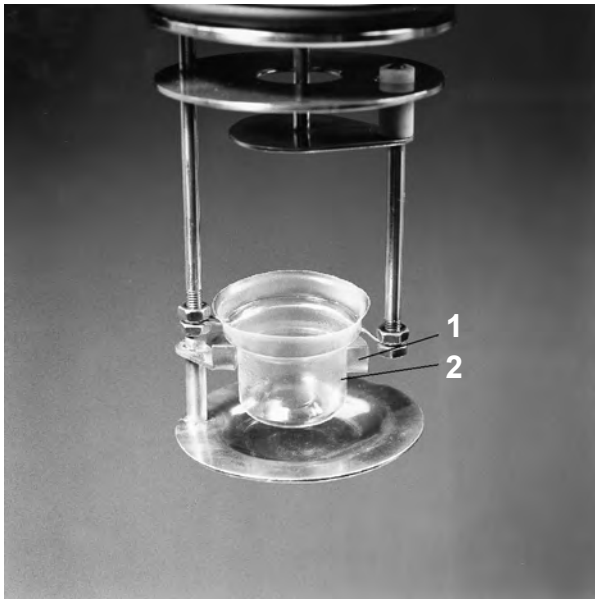
The temperature increase during a test should be as close as possible to the temperature increase during calibration.

If the decomposition vessel is damaged, there is a danger of it bursting! Follow the operating instructions for the decomposition vessel!

When working with unknown substances, start with a very small sample so that you can safely determine its energy potential. If you are burning unknown samples, leave the room or keep a safe distance between you and the calorimeter.

After the sample has been weighed (with combustible crucible if used), the crucible can be placed on the crucible holder.

Placing the combustible crucible



1 Crucible holder (part of C 5010.4) (not in scope of supply)
2 Combustible crucible (not in scope of supply)

If distilled water or other liquid is added to the decomposition vessel for the test, then calibration must be carried out with the same quantity of the same liquid.

④

Positioning the cotton thread

Use a pair of tweezers to arrange the cotton thread so that it hangs in the crucible and as far as possible contacts the sample. This ensures that during the ignition phase the burning thread falls on the sample and ignites it.

①

Starting from *menu 1*, for a calibration, press the ▼ button once to go to *menu 2*. Then press function key *f3*, to enter the sub-menu *calib*. For a calorific value determination, press function key *f1* in *menu 1* to go to sub-menu *pre-par*. In both sub-menus, the enquiry *sample code* appears.

②

The *sample code* identifies a test. Use of this code word will later provide access to the test data. A specified sample code will appear in the upper left zone of the LCD-display for all further entries. The calorimeter generates a sample code automatically when it is switched on, which consists of the date and the number of the current test (00 to 99). The number increases by 1 for every new test. If you do not wish to use these automatic sample codes, use the alpha-numeric keys to enter your own code word for the test. Up to 9 consecutive alpha-numeric characters can be entered. Confirm by pressing the ↵ button. The enquiry *user* appears in the LCD-display.

③

Use the alpha-numeric keys to enter the name or initials of the operator. Here too, up to 9 consecutive alpha-numeric characters can be entered. Confirm by pressing the ↵ button. The enquiry *q-extr. /1/* appears in the LCD-display.

④

q-extr. /1/ is the sum of **all** sources of external energy. The preset entry here is 50 Joule. This is the calorific value of the cotton thread C 710.4, which you can purchase from IKA®. Use the key panel to set this value correctly for the current test.

When using combustible crucibles or other combustion aids, their weight in grams can be entered manually using the key panel, or read automatically from a connected balance. Manual entry is made using the alpha-numeric keys after selecting the menu level *manual* with function key *f3*. The calorimeter accepts only values greater than zero. For automatic input from the balance, use function key *f4* to call up the menu level *balance*. The mass of the sample as determined by the balance will be read in automatically.

It is now necessary to enter the calorific value of the combustion aid. The last value entered always appears here as a default value. If necessary, erase this value by pressing the *del* button repeatedly. Once these two entries are completed, the calorimeter automatically calculates the external energy supplied by the combustion aid.

☞ **If a cotton thread is used in addition to a combustion aid, the external energy supplied by the cotton thread must be added manually.**

Confirm by pressing the ↵ button. The enquiry *sample mass* appears in the LCD-display.

7.4 Carrying out a measurement

When a calibration or a calorific value determination has been prepared as described above, the measuring process can be started. The *sample code* shown on the LCD-display informs you of the test parameters which will be used for the measurement. Pressing the ↵ button starts the calibration or calorific value determination.

From now on, the C 7000 calorimeter takes control. The measurement process is made up of a pre-test and the main test. The current phase of the test is shown on the LCD-display. During both the pre-test and the main test, you can follow the temperature changes in the decomposition vessel on the LCD-display. From the start of the test, they are updated every 1.5 seconds. In the second line of the LCD-display you can also see the duration of pre-test or main test, which is indicated by an extending bar.

End of the test
The end of the test can be seen on the LCD-display or on the print out. As long as the value *0* has not been entered in the sub-menu *realt.* (Real time printout), during the test the printer will print the current temperatures at the set time intervals and the result at the end of the test. In the first line, the LCD-display shows the temperature increase in Kelvin and, for a calibration, the provisional C value in the second line.

The following steps must be carried out when the test has been completed:

①
The green LED blinking near the ✓ button indicates the end of the test. Acknowledge this by pressing the ✓ button. If the button is not pressed, an acoustic signal sounds after about five seconds, as an additional means of making you aware that the test has ended. It sounds for about 30 seconds if it is not acknowledged. After that, the LCD-display returns to *menu 1* in the main menu.

②
Open the cover of the measuring cell by lifting the upper section of the instrument and pushing it backwards. The result of the test just carried out then appears on the LCD-display. Use the carrying and venting handle, C 7010.8, to lift the decomposition vessel out of the measuring cell.

③
So that the next test can take place under the same temperature conditions, the energy absorbed by the decomposition vessel must be removed again. The C 7002 cooler fulfils this function. Place the decomposition vessel vertically in the cooler. The cooling jaws close automatically. The display shows the temperature difference between the decomposition vessel and the calorimeter in percent. During the cooling process, this value progressively approaches zero. When the temperature of the decomposition vessel is the same as the calorimeter temperature, this is indicated by repeated, short acoustic signals. The cooling jaws open at the same time. Remove the decomposition vessel using the carrying and venting handle, C 7010.8.

The cooler can be manually controlled using the *open/close* button. Pressing the *open/close* button during the cooling process opens the cooling jaws. If the button is operated again, the jaws close again and cooling is continued. Cooling only continues as long as the decomposition vessel is at a higher temperature than the calorimeter.

⑦

Then press the *mem* button to store your new entry, and use the *esc* button to return to *menu 1* in the main menu.

The C value for the decomposition vessel you have specified is now stored in the calorimeter, and will be called up automatically when you make a measurement with this vessel.

Repeat the above procedure to enter the C value for all the other decomposition vessels you are using.

7.6 Cleaning and checking decomposition vessels

If there is a suspicion that a sample, or the residues or gaseous products of combustion could present a health hazard, then protective personal equipment (e.g. gloves, breathing mask) must be worn. Combustion residues which are a hazard to health or the environment must be disposed of as hazardous waste. We refer you explicitly to the valid regulations.

For accurate measurements it is absolutely essential that the decomposition vessel is clean and dry. Contaminants modify the heat capacity of a decomposition vessel and so cause inaccurate results. After every test, the inner walls of the vessel, the internal fittings (mountings, electrodes etc.) and the combustion crucible (internally and externally!) must be thoroughly cleaned.

Inner walls of the vessel

In most cases it is only necessary to remove condensate from the inner walls and internal fittings. It is then sufficient to wipe the parts carefully with an absorptive, lint-free cloth.

If the decomposition vessel cannot be properly cleaned using the measures described (e.g. burning, pitting, corrosion etc.), contact your Technical Service Department. Please also note the instructions given in Section 1 "For Your Safety"

Crucible

Combustion residues in the crucible, e.g. carbon or ash, must also be wiped off carefully with an absorptive, lint-free cloth.

8 Evaluation of Calorific Value Tests

When a calorific value test has been completed, you can proceed to evaluating the results. Section 8.1 explains how stored tests can be listed in a table, how you output or erase test results, and how you can list information about free memory for storing new tests. You will find these functions in the sub-menu *output*. The test results are dependent on the post-test parameters; their meaning and how to enter them are described in Section 8.2. Section 8.3 deals with the possibility of test simulation.

8.1 Output and treatment of test data

Before stored data can be issued and processed, the user must first state which tests are to be called up from memory. After the appropriate menu has been activated, you will be requested, in succession, to enter selection criteria for the date, sample code and sample status.

A particular test is called up by the user entering appropriate values for the date, sample code and sample status. With the aid of the two place markers "**.**" and "**-**" it is, however, also possible to select several tests at the same time. The symbol "**.**" serves as a place marker for any desired string of characters, while the "**-**" symbol functions as place marker for any desired single character. Both symbols can be used in the date and sample code fields, whereas sample status only considers one of four fixed, preset values.

When the symbol "**.**" is entered in one of the two fields, the selection criterion is not defined in more detail, all tests in the memory meet this criterion.

On the other hand, the symbol "**-**" stands for just one character within a string. It can, however, be used several times.

The sample status can have the following values:

- *all*
  all stored tests are selected, independent of sample status
- *afterpar*
  selects only tests for which post-test parameters have been entered (see Section 8.2)
- *edit.*
  selects only edited tests
- *printed*
  selects only tests that have already been printed.

The individual data for date, sample code and sample status are logically linked by an AND relationship. This means that only tests that fulfil all three specified criteria will be selected.

Output of test data
It is only possible to calculate net calorific values and publish them by printing or on the LCD-display after all pre-test and post-test parameters have been entered *and* the measurements carried out. The output contains all the parameters entered, the automatically recorded data, and the calculated results. The latter are dependent on the calculation mode selected. In addition, the output contains notes about the test, if appropriate.

To print test results, proceed as follows:

①
Make sure that the printer is connected and switched on.

②
Change from *menu 1* to the sub-menu *output* by pressing the function button *f3*, and then use function button *f4* to select the sub-menu *samp.*

③
Enter selection criteria for the date, sample code and sample status as described above, and close your entries by pressing the *mem* button. The printer will now start to print.

To show test results on the LCD-display, proceed as follows:

If there is no printer connected, or if the printer is not switched on, you can only show test data on the LCD-display if the parameter *realt.* in the sub-menu *STInit* of *menu 3* is set to *0*.

①
Change from *menu 1* to the sub-menu *output* by pressing the function button *f3*, and then use function button *f1* to select sub-menu *LCD*.

②
Enter selection criteria for the date, sample code and sample status as described above, and close your entries by pressing the *mem* button. The parameters appear in the LCD-display in two lines, in the same order as the printed output. The upper line contains the name and units of the parameter, and its value is shown in the lower line. To see the next parameter, use the arrow buttons ▲ or ▼ (in this case, both have the same effect). After all parameters have been shown, the display returns to the main menu.

Parameters can only be displayed in the forwards direction, it is not possible to page backwards. The *esc* button can be used at any time to break off the procedure.

Editing test parameters
The subsequent editing (changing) of parameters is only possible if the test results have been issued at least once by printing or on the LCD-display. This is to prevent values being changed unintentionally.

as delivered condition are marked "raw", and parameters that refer to the analytically moist condition are indicated by "an".

By calling up the sub-menu *after-p.* under *menu 1*, the various parameters can be entered in order in the same way as the pre-test parameters (see Section 7.3). The values entered must be stored by pressing the *mem* button. After they have been stored, the data can only be changed by using the *edit.* menu. However, the post-test parameters can only be changed after the test has been either printed or shown on the LCD-display.

The meanings of the parameters and the calculation modes to which they belong are given in the following list:

Post-test parameters

sample code	The sample code is the means of identifying a test. Use of this code word gives access to the test data. Applies to all calculation modes.
remark	For documentation purposes, a text of up to 10 characters can be entered. Applies to all calculation modes.
q-extr. /2/	For entering any desired external energy for calculation of gross and net calorific values. Applies to all calculation modes.
total H_2O	Percentage of total water in the combustion sample as determined by element analysis. Calculation mode: *std - t*
hydrogen (an)	Percentage of total water in the combustion sample. Calculation modes: *std + t, coal - t, coal + t*
Na_2CO_3 vorg.	Amount of sodium carbonate solution placed in the decomposition vessel (according to DIN: 20 ml; 0.05 N). Calculation modes: *std + t, coal + t*
$Ba(OH)_2$ verbr.	Titrated quantity of 0.1 N barium hydroxide solution (titration of the distilled water used to rinse the decomposition vessel after a test). Calculation modes: *std + t, coal + t*
HCl verbr.	Titrated quantity of 0.1 N hydrochloric acid (titration of the distilled water used to rinse the decomposition vessel after a test). Calculation modes: *std + t, coal + t*
gr. moisture (raw)	Percentage of water from rough moisture. Calculation modes: *coal - t, coal + t*
ash (an)	Percentage of ash. Calculation modes: *coal - t, coal + t*

9 Care and Maintenance

9.1 Maintenance

To ensure proper functioning of our instrument in continuous use, it is essential that it is maintained at regular intervals. We can offer you a special maintenance contract to meet this need. You can obtain further information direct from our Service Department.

For maintenance of the decomposition vessel, see the C 7010/C 7012 operating instructions!

9.2 Recommendations for cleaning

Clean your IKA® instrument only with these IKA®-approved cleaning agents:

Contaminant	Cleaning agent
• Dyes	Isopropanol
• Building materials	Water with detergent, isopropanol
• Cosmetics	Water with detergent, isopropanol
• Foodstuffs	Water with detergent
• Fuels	Water with detergent
• Other substances	Please consult IKA®

Notes

Electrical equipment must not be immersed in a cleaning agent.
Stainless steel can be cleaned with commercially-available stainless steel cleaners, but do not use those containing abrasives.
We recommend the wearing of protective gloves during cleaning.
The operating authority is responsible for ensuring that appropriate decontamination measures are taken if hazardous materials are spilt on or in the instrument.
Before using any method for cleaning or decontamination other than those recommended by the manufacturer, consult the manufacturer to make sure that the intended method will not damage the instrument.

When replacing the mains cable, use a product of equivalent quality and performance.

10 Messages and Troubleshooting

The C 7000 calorimeter is subjected to stringent quality checks during manufacture, should malfunctions occur despite this care, you will find in this section a selection of corrective measures for dealing with a range of fault situations. Most messages and fault indications appear on the LCD-display. They should be acknowledged with the ✓button. If your attempts to correct a fault are not successful, please contact your authorized IKA® Technical Service Department.

10.1 Messages from the C 7000 Calorimeter

Message	Cause	Remedy
printout not complete yet	It is not possible to display a result on the LCD if the unit still has a print order to process.	Check that the printer is online, otherwise wait until the printer has finished.
cover was opened, test aborted	The cover was opened during a measurement.	If the cover was opened during the pre-test period, the same test can be restarted. Opening the cover during the main test invalidates the measurement. Repeat the measurement.
printer timed out	No data can be sent to the printer.	Check that the printer is online. Set the period for a real-time printout (sub-menu *realt.*) to any value other than zero. Check the printer cable.
end of entry [mem] = save	Makes you aware that all parameters in this menu have been entered. To store the entries, you must press the *mem* button as soon as this message has disappeared.	
calibration test	You have attempted to enter post-test parameters after a calibration test. This is not possible.	

Message	Cause	Remedy
test not yet printed	You have attempted to change the parameters of a test that has not yet been printed out.	Print test at least once, and then edit.
loss of power supply	An interruption in the power supply has been detected.	Possibly too many consumers are connected to a single socket, which has caused a brief voltage drop. Connect C 7000 to a separate socket.
PT parameters missing	A test can only be carried out after pre-test parameters have been entered.	Enter pre-test parameters. Check that you have not entered the wrong sample code.
V.24 timed out	Data could not be sent through the V24 interface to the PC.	Check interface parameters and connection cable. Make sure that external PC is ready.
scale timed out	Data could not be sent to the balance, or the balance does not respond.	Balance reading has not yet stabilised. Check interface parameters and connection cable.
memory has been reinitialized	After the program was started, a memory error was detected, and the memory reinitialized. Test data have been erased.	If this problem occurs frequently, the battery on the MC board must be changed (by the Technical Service Department or an instructed specialist).
problem with clock storage	After the program was started, a memory error was detected in the clock unit.	Check date and time, and reset if necessary. If this problem occurs frequently, the clock unit must be changed (by the Technical Service Department or an instructed specialist).
clock battery dead	The clock unit battery is empty.	The clock unit must be changed (by the Technical Service Department or an instructed specialist).

11 Accessories and Consumables

11.1 Accessories

Ordering description

C 7010	IKA® decomposition vessel, standard
C 7012	IKA® decomposition vessel, halogen resistant
C 5010.4	Support for disposable crucible
C 48	Oxygen filling station
C 7002	Cooler
C 7010.8	Venting handle
C 7030	Venting station
C 5040	Calorimeter software for PC (CalWin®)
C 21	Pelleting press
C 29	Pressure reducer for oxygen
KV 500	Cooling water supply

11.2 Consumables

Ordering description

C 710.4	Cotton threads, cut to length (500 pieces)
C 5010.3	Ignition wire (5 pieces)
C 5012.3	Platinum ignition wire(2 pieces)
C 5010.5	Crucible support, large
C 4	Quartz dish
C 5	Set of VA combustible crucibles (25 pieces)
C 6	Quartz dish, large
C 9	Gelatine capsules (100 pieces.)
C 10	Acetobutyrate capsules (100 pieces)
C 12	Combustion bags, 40 x 35 mm (100 pieces)
C 12A	Combustion bags, 70 x 40 mm (100 pieces)
C 43	Benzoic acid (NBS 39i, 30 g)
C 43A	Benzoic acid (100 g)
C 723	Benzoic acid tablets (50 pieces, ca. 25 g)
C 14	Combustible crucibles (100 pieces)
C 15	Paraffin strips (600 pieces)

12 Technical Data

12.1 Technical data for C 7000 Calorimeter

Operating voltage		100 V-120 V / 220 V-240 V
Operating frequency		60 / 50 Hz
Power consumption		100 W
Fuses	for 230 V supply:	2 x 1.0 AT 1 x 630 mAT 1 x 250 mAT
	for 115 V supply:	2 x 2.0 AT 1 x 1.3 AT 1 x 500 mAT
Degree of protection to DIN 40 050		IP 21
Protection class		1 (with protective earth)
Over-voltage category		2
Contamination level		II
Ambient temperature		18°C ... 30°C (constant)
Max. relative humidity of environment		80 %
Use above sea level		max. altitude 2000 m
Dimensions (B x D x H)	unit closed: unit open:	310 x 490 x 395 mm 310 x 490 x 460 mm
Weight		25 kg
Measuring range		30 000 J
Interfaces		2 x serial (RS 232) for PC and balance 1 x parallel for printer 1 x connection for C 7002 cooler

12.2 Technical data for C 7002 Cooler

Operating voltage		100 V-120 V / 220 V-240 V
Operating frequency		60 / 50 Hz
Power consumption		160 W
Fuses	for 230 V supply: for 115 V supply:	2 x 1.25 AT 2 x 2.5 AT
Degree of protection to DIN 40 050		IP 21
Protection class		1 (with protective earth)
Over-voltage category		2
Contamination level		II
Ambient temperature		18°C ... 30°C (constant)
Max. relative humidity of environment		80 %
Use above sea level		max. altitude 2000 m
Dimensions (B x D x H)		360 x 290 x 245 mm
Weight		12.3 kg
Interface		1 x connection for C 7002 calorimeter
Cooling power (only Peltier elements)		80 W (with cooling water supply at 2 l/min)
Water inlet pressure		max. 9 bar

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