# Bomb calorimeter

04403.00

PHYWE Systeme GmbH & Co. KG
Robert-Bosch-Breite 10
D-37079 Göttingen

Phone +49 (0) 551 604-0
Fax +49 (0) 551 604-107

**Operating Instructions**

Fig. 1: Bomb calorimeter

**CONTENTS**



## 1 SAFETY PRECAUTIONS

- Carefully read these operating instructions before operating this instrument. This is necessary to avoid damage to it, as well as for user-safety.
- Only use the instrument for the purpose for which it was designed.

## 2 SAFETY INSTRUCTIONS

Particular care is always necessary when handling pressure containers and experimental autoclaves.
Suitable safety devices must ensure that, should a bomb calorimeter explode, no person suffers harm.
To be certain that the pressure build-up that is to be expected in the bomb calorimeter during reaction does not go above 70 bar, the bomb calorimeter must under no circumstances be filled with oxygen at a pressure higher than the stipulated maximum of 10 bar.
Being a pressure vessel, the bomb calorimeter must be set up and operated according to pressure vessel regulations.
The bomb calorimeter is only to be opened when pressure equalisation with the atmosphere has been achieved. For this, the coupling plug must be fitted into the quick connect coupling of the valve, and the valve then carefully opened. It must be hereby ensured that no hazardous substances are emitted into the ambient air (fume cupboard!).
For the protection of persons, particularly with regard to failure of the autoclave and with pressure reactions for which there is insufficient knowledge of the pressure and temperature that are to be expected, the bomb calorimeter must be set up in a special chamber or behind protective screening. The observation of the measurement and safety devices and the operation must be carried out from a safe place.
After each use, or possibly at the end of a measurement series, the bomb calorimeter must be inspected by a technical expert. Should hereby damage be determined, or should the permitted operating pressure and operating temperature have been exceeded, then the further usability of the bomb calorimeter must be tested by an authorized expert.

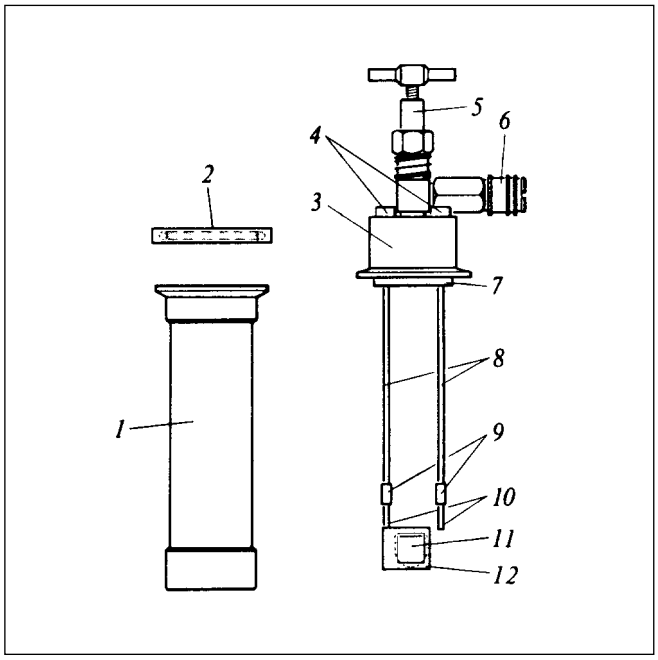

Fig. 2: Individual parts of the bomb calorimeter

## 3 PURPOSE AND CHARACTERISTIC FEATURES

The bomb calorimeter serves to measure the calorific values of solid or liquid subtances. It is usually used in practical work as a complete measurement system by supplementing it with a calorimeter and a temperature measuring device. A holder which enables the bomb calorimeter to be hung in a calorimeter (Art. no.: 04402.00, see List of Materials) is standardly supplied with the bomb calorimeter.

The bomb calorimeter consists of a pressure-resistant container *(1)* (see Figs. 1 and 2), and a lid *(3)* for gas-tight closing of it. Two 4 mm connecting sockets *(4)* on the top of the lid connect through the lid to the nickel electrodes *(8)* at the bottom of the lid. An ignition wire can be attached between these. Sample holder *(12)* at the electrodes serves to hold a combustion crucible *(11)*. Disk *(7)*, made of ceramic-fibre, acts as heat insulator. Regulating valve *(5)* and quick connect coupling *(6)* allow simple connection of the bomb calorimeter to an oxygen cylinder, for filling it with oxygen, by means of a pressure hose with plug (Art. no.: 39299.00). The pressure-resistant container and the lid can be fitted gas-tightly together by means of the externally centered O-ring *(2)* and the bracing ring.

## 4 MEASUREMENT METHOD

An exactly determined amount of sample is combusted in as pure an oxygen atmosphere as possible. The heat evolved is transferred to a calorimeter filled with water and causes an increase in temperature in the bomb calorimeter-calorimeter system that is within the range 1...3 K, and must therefore be measured with the greatest possible accuracy. The formula

$$\Delta U = \frac{(C_{H_2O} + C_{Cal}) \cdot \Delta T \cdot M}{m_E}$$

is used to calculate the change in internal energy $U$.

The dimensions given represent the following values:

| | |
|---|---|
| $(C_{H_2O} + C_{Cal})$ | Heat capacity of the bomb calorimeter-calorimeter system filled with water |
| $\Delta T$ | Temperature difference measured |
| $M$ | Molar mass of the substance under examination |
| $m_E$ | Weight of the sample of the substance |

## 5 HANDLING

Wind an approx. 4 cm long piece of iron wire to form an ignition coil and fit it in the bore holes *(10)* of the nickel electrodes (see Fig. 2); clamp the ends in place by sliding down sleeves *(9)*.

Place the combustion crucible holding the weighed-out sample in the sample holder. Use a pointed object to carefully press the ignition coil into the sample; this is usually only in the range of about 40 mm to 400 mg, so that it is important to avoid loss of substance here. Fit the O-ring on the flange of the pressure-resistant container and carefully place the lid on; use the bracing ring to close the container and lid together as a tight unit. Fill the container with oxygen by first fitting the pressure hose to the oxygen cylinder - with pressure regulating valve!- and then plugging it into quick connect coupling *(6)*. To fill the container, open regulating valve *(5)* and adjust the pressure with the pressure regulator on the cylinder (max. 10 bar). Close the regulating valve and hang the bomb calorimeter in a calorimeter filled with water, whose lid has been replaced by the holder for the bomb calorimeter. Fit a PT100 temperature probe in one of the holes in the holder and connect it to a digital temperature measuring instrument (0.01 K measurement accuracy) so that the temperature of the water can be measured. Connect 4 mm sockets *(4)* to a power supply that supplies a minimum voltage of 15 V/1 A. Place the calorimeter with bomb calorimeter on a magnetic stirrer that is to be operated without heating for the measurement. Now initiate combustion in the bomb calorimeter by applying a voltage to the 4 mm sockets and measure the increase in temperature in the calorimeter under continual stirring (approx. 15 minutes).

When reaction has finished, check that that the bomb calorimeter has cooled to room temperature . Only when this is the case, and with regulating valve *(5)* closed, push the coupling plug into quick connect coupling *(6)* and carefully open the valve. Dangerous gases must not be allowed to escape into the ambient air. It is therefore recommended that bomb calorimeter pressure equalisation be carried out under a fume hood. Do not open the bracing ring until pressure equalisation between the inside of the bomb calorimeter and the surrounding atmosphere has been achieved with certainty.

Note:

In the combustion of solid substances, it has been found that better results are obtained as a rule when the sample is first pelleted in a press. To do this, first grind the solid substance to powder in a mortar and weigh out approx. 400 mg in a weighing dish. Cut a 10 cm length from the iron wire and weigh it exactly to an accuracy of 1 mg. Fit it in the guides of the pressing tool so that a small loop is formed in the middle, which is to give a good hold for the pellet. Use the funnel to fill the weighed amount of substance into the press (Art. no.: 04403.04), insert the stamp and exert moderate pressure in a vice to form a pellet. The ends of the ignition wire must not be cut off during this procedure. Remove projecting edges etc. then weigh the pellet exactly to an accuracy of 1 mg.

Subtract the weight of the iron wire from the weight of the pellet to obtain the weight of the sample. The heat of combustion of the iron wire can generally be disregarded, as it present both in the calibration and in the actual measurement, and is small in value. Fix the two wire ends of the pellet to the lid contacts of the bomb calorimeter so that the pellet is above the middle of the sample holder, so that it can completely combust there when the ignition wire ends have burnt through. Should, at the end of the experiment, the sample holder be very sooty, then the measurement must be repeated, as this shows that combustion was incomplete.

## 6 TECHNICAL SPECIFICATIONS

| | |
|---|---|
| Filling pressure | max. 10 bar |
| Max. operating pressure | 70 bar |
| Max. operating temperature | 100°C |
| Material: | |
| Lid and pressure resistant container | Stainless steel |
| O-Ring | Viton |
| External centered ring | Aluminium |
| Electrodes | Nickel |
| Volume | approx. 120 ml |
| Weight | approx. 1.2 kg |

## 7 LIST OF MATERIALS

### 7.1 Equipment standardly supplied

Bomb calorimeter, consisting of a pressure-resistant container, lid and bracing ring

Holder for hanging the bomb calorimeter in a transparent calorimeter

Coupling plug for quick connect coupling

Expendables:

Sample vessel (combustion crucible), 10 pieces
(Art. no.: 04403.03)

Iron wire, OD 0.2 mm, 100 m
(Art. no.: 06104.00)

### 7.2 Recommended accessory equipment

| | |
|---|---|
| Calorimeter, transparent | 04402.00 |
| Pellet press for calorimeter | 04403.04 |
| Magnetic stirrer | 35720.93 |
| Magnetic stirrer bar oval, 30 mm | 35680.04 |
| Pressure tube with fittings | 39299.00 |
| Reducing valve for oxygen | 33482.00 |
| Steel cylinder, oxygen, 10 l, filled | 41762.00 |
| Temperature meter digital, 4-2 | 13617.93 |
| Temperature probe, immersion type, Pt 100 | 11759.01 |
| Power supply, universal | 13500.93 |

## 8 GUARANTEE

We guarantee the instrument supplied by us for a period of 24 months within the EU, or for 12 months outside of the EC. This guarantee does not cover natural wear nor damage resulting from improper handling.

The manufacturer can only be held responsible for the function and technical safety characteristics of the instrument, when maintenance, repairs and changes to the instrument are only carried out by the manufacturer or by personnel who have been explicitly authorized by him to do so.

## 9 WASTE DISPOSAL

The packaging consists predominately of environmental compatible materials that can be passed on for disposal by the local recycling service.

Please contact your municipal administration for information on the disposal of instruments.