CONTENTS

<u>Laboratory Precision Calorimeter for Liquid Fuels</u>

Laboratory Precision Calorimeter for Fuel Gases (Figure 1)

0. Occupational Health and Safety

The occupational health and safety is referred to in sections 5.2., 5.4., 5.6., 6.1.2., 6.3., 6.4. and 6.5.

1. Purpose

The complete combustion of fuel gases or fuel gas mixtures produces a certain amount of combustion heat, which depends on the gas composition. This amount of combustion heat is called "Gross calorific value". Therefore, a fuel gas is evaluated by diectly analyzing its gross calorific value. For carrying out single analyses, the laboratory precision calorimeter is used providing an accuracy unmatched until now.

2. Application

The laboratory precision calorimeter is to analyze gross calorific values or all industrial combustible gases and may be used

- as a reference or monitoring instrument in gas works or gas producing plants, particularly in conjunction with the recording calorimeters installed

- for controlling test or boundary gases in plants developing or producing burners, thst utilize these gases

- in research institutes or development stations utilizing fuel gases

3. Theory of Operation and Design

The gross calorific value analysis is base on the determination of the quantity of water, gas volume, and temperature difference. The gros calorific value is calculated according to following formula:

$$Q_S = \frac{m_W}{V_G} \Delta \cdot t \cdot C_W$$

where

m_W = Quantity of water in kg

V_G = Gas volume in m^3

$t\Delta$ = Temperature difference in K

Q_S = Gross calorific value

C_W = Specific heat of water (4.187 kJ/kg K)

The gross calorific value is directly analyzed by using the laboratory precision calorimeter. The heat produced by burning the gas is transferred to the water continuously circulated in the calorimeter. Once the heat exchange has entered steady conditions, the heat conducted by the water will be equal to that generated by the burning gas. Calorimetric analyses on gases require several individual devices, which interactions and use of are shown in Figure 1.

The gas enters the water-cooled gas cooler 1 passing inlet socket 2, while the water enters the colling jacket through inlet socket 4. This arrangement causes the gas to take on the temperature of the inflowing water, which is a necessary requirement for a uniform and accurate calorimetric analysis. The gas then passes the precision gas meter 5 being necessary for volumetric determining the quantity of the gas required. Next, the gas enters the gas pressure regulator 6 which provides a continuous gas flow to the burner 7.

The water flows from the outlet socket of the precision gas meter 8 to the overflow 11 passing the tube 9. The overflows included in item 11 ensure that a sufficient amount of water will be directly led into the heat exchanger 10. The remaining water enters the air humidifier 13 after passing the tube 12. The air for combustion required by the burner is saturated with water vapour in the air humidifier taking on the temperature of the water fed into the gas cooler.

After the amount of water required for calorimetric analysis has been adjusted using batching cock 14, it is heated and then led into a measuring vessel passing the drain cup 15 and a three-way cock 16. The temperature difference between the gas cooler (cold) water inlet and (hot) water outlet required for calculating the gross calorific value is obtained from the temperature readings of thermometers 17 or 18, respectively.

The waste gas produced by burning the fuel gas is discharged into the atmosphere through outlet socket 19. The thermometer 20 is to measure the waste gas temperature. The heat exchanger is fitted with a special connector 22 for discharging the condensate. The gas meter is filled using connector 22.

4. Specifications

Site	- A light room, free of draught, with heating facilities. The apparatus must not be exposed to direct solar radiation.
Space requirements	- Flat worksurface, 0.8 x 1.5 m
Total weight	- approx. 23 kg
Environmental temperature range	- +10 °C to +35 °C
Water temperature	- Preferrably 3 to 6 K below room temperature

Quantity of water required	- Approx. 50 litres per analysis
Gas pressure required	- Min. 500 Pa Max. 800 Pa
Gas supply	- PVC tube, 10 mm inner diameter
Water supply	- PVC tube, 10 mm inner diameter
Time response	
Dead time	- 35 secs to 200 secs, depending on measuring range selected
Half value time	- Dead time +30 secs
98 % time	- Dead time +95 secs
Error	- ±0.5 % (when adjusted after test gas analysis)
Measuring ranges	- Refer to Table 1
Class of application	- +10/ +35/ +30/ 80/ 1101

Table 1

Ref. No.	Type of gas	Maximum calorific value (MJ/m^3)	Gas throughput (dm^3/min)	Diameter of burner nozzle (mm)	Burner head for
1	Generator gas	6.5	13.5	4.8	Lean gas
2	Water gas	11	8	2.9	Normal gas
3	Town gas	17	5.5	2.4	Normal gas
4	Remote transmission gas	21	4.4	2.1	Normal gas
5	Coke oven gas	23	4.0	2.0	Normal gas
6	Natural gas	42	2.2	1.7	Normal gas
7	Rich gas/lean gas mixtures	55	1.7	1.6	Rich gas
8	Rich gas mixtures	69	1.3	1.4	Rich gas
9	Rich gas mixtures	126	0.8	1.0	Rich gas

5. Setting Up

5.1. Unpacking and Inspection

The apparatus is unpacked and inspected for any demages. All parts and components delivered are checked for completeness as specified by the acknowledgement of order.

5.2. Installing the Laboratory Precision Calorimeter

For installing the complete apparatus a flat worksurface of about 0.8 by 1.5 m is required. The site should be a light and draught-free room with heating facilities available. Note the calorimeter not to be exposed to direct solar radiation. Users are advised to set up the individual calorimeter components in the order shown in Figure 1.

A sufficient air reversal must be guaranteed in the room without any draught. The bench surface must not consist of flammable material. Otherwise it has to be covered with a protective sheet made from fire-resistant material. No inflammable objects should be placed on the bench.

5.3. Heat Exchanger

Insert the feet in the appropriate places as well as the T-shaped connector 23 accomodating both the air humidifier connector and the burner. One hose each is connected to both the three-way cock 16 outlets (refer to Figure 1) providing water drain.

5.4. Gas and Water Connections

Hoses are used to supply gas or water to the unit as well as to connect the analyzing apparatus and the heat exchanger to each other. All hose connections are secured by hose clips.

In order to keep both pressure and temperature of the inflowing water as constant as possible a storage tank having a capacity of about 120 litres can be placed approximately 3 m above the calorimeter and the water fed from there. A float valve can be inserted providing a mean of controlling both water level and feed. If necessary, a thermostat can be installed to ensure a constant temperature of the water entering the calorimeter.

5.5. Mounting the Air Humidifier

The air humidifier is connected to the heat exchanger overflow pipe using the pipe bend 24 (Figure 1).

5.6. Inserting the Burner

The burner is inserted into the T-shaped connector. Note the appropriate burner and burner nozzle to be installed as required by the gross calorific value. Table 1 shows the various gases and gross calorific value ranges as well as the Reference Numbers. The reference number corresponding to the nozzle diameter is impresses on the surface of the burner nozzle.

Figures 2 through 4 illustrate the three types of burners available.

5.7. Fitting the Thermometers

The thermometers 17 and 18 are fitted into the apertures provided in the heat exchanger cover using the rubber stoppers supplied. The thermometer 20 is inserted into the waste gas outlet in the same way. The thermometers are advised to be greased slightly in order to facilitate pushing the rubber stoppers onto the thermometers.

Make sure that the mercury column is not disrupted before inserting the thermometers into the heat exchanger.

6. Preliminaries prior to Setting the Calorimeter into Operation

Following checking procedures have to be carried out on all devices included in the analyzing apparatus before setting the laboratory precision calorimeter into operation.

6.1. Gas meter

6.1.1. Levelling (Figure 5)

The precision gas meter (Figure 5) is levelled at its site using the setting screw 1 at the base plate until the bubble of the level will be centred.

6.1.2. Leakage Test and Filling the Manometer Pipe

Now the instrument is tested for leakage. Coloured water is introduced through filler neck 3 using a pipette until the water level will have reached the zero mark on the manometer pipe 4. Thereafter the water level is adjusted to zero using setting screw 5.

The gas flow is stopped by a hose clip placed near gas outlet 6. The hose connection at the gas cooler gas inlet is disconnected and replaced with a mouthpiece fitted with a hose clip. A pressure of 1 kPa above the atmosphere is produced in the gas meter by blowing and the gas inlet immediately closed using the laboratory hose clip. The manometer reading must not drop by more than 30 Pa within 5 minutes time.

6.1.3. Sealing Liquid

After the leakage test has been carried out successfully, all hose connections are restored and the sealing liquid is filled into the precision gas meter. For this purpose, the protective cap 10 is removed from the overflow at the rated level and the cock 11 set to "Prüfen" /Testing/ position (refer to Figure 6).

Any grease or lubricant must be removed from the measuring tip and the edge of the overflow using petrol or soap solutions. The scale front ring 7 is removed by twisting off and the instrument pointer moved carefully up to the triangular red scale mark.

Water is used as sealing liquid. For filling the water, the screw cap on the filler is unscrewed and the water having a temperature of 3 to 6 K below the room temperature fed into the gas meter. The filling operation will be completed as soon as the sealing liquid level in the rated value overflow vessel is slightly bulging(convex liquid meniscus) and the index 12 in the overflow just touching the liquid surface.

Now the cock 11 is set from the "Prüfen" /Testing/ to the "Betrieb" /Operation/ positions, screw cap 10 fitted, scale front ring 7 attached to the precision gas meter, and filler 3 closed by cap 10.

Check the sealing liquid in the overflow once again and, if necessary, adjust after the gas meter has rotated several times.

6.2. Filling the Heat Exchanger with Water

The three-way cock 16 on the heat exchanger 10 (Figure 1) is set to the "Abwasser" /Drain/ position. In this case, the batching cock is put to "0". Once the shut-off valve in the water supply line is opened the batching cock is set to scale mark 4 in order to prevent air pockets from developing by slow water influx into the heat exchanger, while it is being filled. The water flow should not be stopped as long as bubbles are seen forming on the surface of the water in the drain vessel 15. Now, the shut-off valve in the water supply line is closed again. Water must not come out of outlet 21, otherwise there will be a leakage which must be eliminated immediately.

6.3. Setting the Gas Meter

The burner is removed from the heat exchanger. Then, the main gas valve in the gas supply line is opened. After the gas meter drum has rotated two or three times the burner is extinguished by use of the hose clip thus getting the air displaced which is trapped in the gas meter. At the same time, a pressure balance will occur in the gas meter indicated by the pointer continueing to rotate slowly for a short time after the hose clip has been closed and then coming to a standstill.

In case this will not happen, the gas system must be leaky. Refer to Section 9.2. for eliminating leakages.

THE PRECISION GAS METER IS NOW READY FOR OPERATION.

6.4. Adjusting the Burner and Pressure Regulator

The hose clip at the burner is opened again and the gas lighted escaping from the burner head. The admission of air is adjusted so as to obtain a roaring flame with a blue-green cone. The gas consumption is controlled using the setting screw 25 of the gas pressure regulator 6. Readjust the flame as described above, if necessary. The burner continues to operate outside the heat exchanger.

6.5. Preliminaries prior to Calorimetric Analysis

The shut-off valve included in the water supply line is opened again. Then the batching cock 14 is adjusted so that a continuous flow of water of 1.5 ± litres per minute is obtained. The water throughput is determined by collecting the water into the measuring vessel (2000 cm^3) in one minutes's time. The measuring vessel is placed below the three-way cock 16, which must be set to the appropriate position. Thereafter the burner is inserted in the heat exchanger and the gas flow readjusted at the gas pressure controller 6 so that a temperature difference Δt between the cold water inlet (T_{WE}) and the hot water outlet (t_{WA}) of

$$\Delta t = t_{WA} - t_{WE} = 10 \ldots 11 \text{ K is obtained.}$$

Here the temperature reading t_{WE} (approximately the same as that of the water contained in the storage tank as per Section 5.4) should be 3 to 6 K below the calorimeter environmental temperature.

In case the flame is extinguished after inserting the burner, refer to the troubleshooting guide (Section 110.1.).

ATTENTION

When unburned gas escaping from the burner happened to get into the heat exchanger, it has to be blown by air before inserting the burner again.

7. Calorimetric Analysis on Gases

7.1. Procedures to be observed

The temperature difference found in the way as described under Section 6.5. is continuously observed. The heat exchange will enter steady conditions after approximately 8 to 10 minutes have elapsed. At this time, condensate must steadily drip off the outlet 21 (Figure 1), while the threeway cock 16 (Figure 1) is still remaining in "Abwasser" /Drain/ position.

Subsequent analysis and readings are advised to be performed in the order given below.

7.1.1. All values being quantifiable in advance (e.g. barometric pressure, room temperature etc.) should be entered into the calculating sheet (refer to Section 7.3.6.) prior to commencing an analysis.

7.1.2. Any analysis or reading are started at the moment the gas meter pointer is passing the full litre red mark.

7.1.3. Determining the Quantity of Condensate

Read off the actual gas meter reading V_{GA1} and simultaneously place the measuring vessel 100 cm^3 below the drain outlet 21. After more than 40 cm^3 of condensate have been collected, remove the measuring vessel from below the outlet and read off the gas meter reading V_{GE1} at the moment the gas meter pointer is passing the litre mark for the next time. Enter V_{GE1} and V_{GA1} readings into the calculating sheet. Determine the quantity of collected condensate in grams and enter the value found into the calculating sheet.

7.1.4. Following gas volumes $V_{G2} = V_{GA2}$ are burnt in the course of an analyzing period:

Ref. No.	1	2	3 through 5	6 through 9
V_{G2}	20	15	10	5

7.1.5. Place the collecting vessel for the flow water below the outlet of the three-way cock 16.

For determining V_{GA2}, set the three-way cock to "Messen" /Measuring/ and immediately start reading off the temperatures shown by thermometers 17 and 18 as soon as the pointer will pass the litre mark. 10 thermometer readings are recorded and entered into the calculating sheet in each measuring period. Reset the three-way cock at the end of each measuring period when the pointer is passing the litre mark.

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Ref. No.	1	2	3 through 5	6 through 9
V_{G2}	20	15	10	5

7.1.5. Place the collecting vessel for the flow water below the outlet of the three-way cock 16.

For determining V_{GA2}, set the three-way cock to "Messen" /Measuring/ and immediately start reading off the temperatures shown by thermometers 17 and 18 as soon as the pointer will pass the litre mark. 10 thermometer readings are recorded and entered into the calculating sheet in each measuring period. Reset the three-way cock at the end of each measuring period when the pointer is passing the litre mark.

Note the gas consumption $V_{G2} = V_{GE2} - V_{GA2}$ to be kept according to Section 7.2.1. and enter it into the calculating sheet.

7.1.6. Next, determine the corresponding amount of the collected flow water in grams by accurately weighing it (the maximum error must not exceed ± 1 g) and enter the value found into the calculating sheet.

7.1.7. The procedures described above cause the heat produced by burning the gas to be transferred to the flow water getting hot via the waste gas, which is almost completely cooled down to the inlet temperature of the test gas, air or water. The heat of condensation of the steam generated by the combustion is transferred to the flow water in the same way, thus allowing a complete gross calorific value analysis.

7.2. Calculating the Gross and Net Calorific Values

7.2.1. Both gross and net calorific values are obtained by use of the formulae mentioned on the calculating sheet (page 29).

7.2.2. The values calculated this way include an absolute error of ±2 %, which is loaded with a reproducability error of up to 0.5 % provided calibrated thermometers and gas meters are used and corrections made as specified by the instrument test certificates.

7.3. Corrections to be made after Analysis on Test Gas

7.3.1. After the gross calorific value of a test gas has been analyzed which the gross calorific value of is exactly known, the test gas gross calorific value Q_{SP} to the analyzed value Q_{SM} ratio is the correction factor.

$$f_b = \frac{Q_{SP}}{Q_{SM}}$$

7.3.2. When values of the gas to be analyzed are determined in the same way and multiplied by f_b, errors caused by each individual component of the apparatus (gas meter, thermometer, heat exchanger) will be summarized and, therefore, corrected so that a little subjective error only will remain related to operating the calorimeter.

The total error can be reduced up to ± 0.5 % (perhaps with the test gas value included) by this method.

However, this result in accuracy will be obtained only if both water throughput and heating are adjusted according to Section 6.5 and, furthermore, a constant difference between the average temperatures of water and environment is maintained.

7.3.3. High-grade methane gas CH_4 contained in a gas cylinder is used as test gas which the contaminants of must be known from the accompanying certificate in order to allow the tabular value of G_S = 39820 kJ/m^3 to be converted to the standard condition.

The corrective factor determined this way is applicable to the measuring ranges from no. 3 on, since the very little gas meter error may be included in the above mentioned error.

7.3.4. An additional error will arise when the average water temperature difference ($t_{Wm} = t_{WE} + \frac{\Delta t}{2}$) t_R obtained while analyzing the gross calorific value $\left[(t_{Wm} - t_R)_M \right]$ differs from that observed while determining the corrective factor $\left[(t_{Wm} - t_R)_B \right]$. The gross calorific value analysed can be adjusted using the corrective factor f_2.

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$f_2 = 1 + [(t_{Wm} - t_R)_M - (t_{Wm} - t_R)_B] \cdot 0.0005\ K^{-1}$

3.5. Examples

a) Test gas CH_4, contaminants (N_2, CO_2, O_2) = 0.25 %

Tabular value	Q_S	= 39,820 kJ/m³	in standard condition
	−0.25 %	= 55 kJ/m³	
Test gas value	Q_{SF}	= 39,765 kJ/m³	
After analysis on above mentioned test gas	Q_{SM}	= 39,525 kJ/m³	

Corrective factor $f_b = \frac{39,765}{39,525} \cdot 1.0061$ (= 0.61 %)

where:

Room temperature t_R = 23 °C

Water inlet temperature t_{WE} = 19.00 °C

Water outlet temperature t_{WA} = 29.60 °C

$\Delta t = t_{WE} - t_{WA}$ = 10.6 K

$t_{Wm} = t_{WE} + \frac{\Delta t}{2}$ = 24.3 °C

$(t_{Wm} - t_R)_B$ = 1.3 K

b) Analysis on Town gas

$Q_{S,n}$ = 16,950 kJ/m³ (in standard condition)
where

t_R = 22.30 °C

t_{WE} = 19.50 °C

t_{WA} = 30.00 °C

$\Delta t = t_{WA} - t_{WE}$ = 10.50 K, t_{Wm} = 24,75 °C

$(t_{Wm} - t_R)_M$ = 24.75 °C − 22.30 °C = 2.45 K

$f_2 = 1 + [(t_{Wm} - t_R)_M - (t_{Wm} - t_R)_B] \cdot 0.0005\ K^{-1}$

$= 1 + (2.45\ K - 1.3\ K) \cdot 0.0005\ K^{-1}$ = 1.000,575

Corrections to be made:

$$Q_{S,nk} = Q_{S,n} \cdot f_b \cdot f_2$$

$$Q_{S,nk} = 16{,}950 \cdot 1.0061 \cdot 1.000575 \text{ kJ/m}^3$$

$$Q_{S,nk} = 17{,}065 \text{ kJ/m}^3$$ adjusted gross calorific value under standard condition

Error:

± 0.5 % = ± 85 kJ/m^3

7.3.6. Calculating Sheet for Laboratory Calorimeter Serial No.

Corrected barometric pressure	b	= .... kPa
Room temperature	t_R	= .... °C
Pressure in gas meter	P_G	= .... kPa
Temperature in gas meter	t_G	= .... °C
Saturation pressure at t_G	P_D	= .... kPa

Factor for converting to 0 °C: 101.325 kPa

$$F_R = \frac{101.325 \text{ kPa } (273\ °C + t_G)}{273\ °C\ (b + P_G - P_D)}$$

Calculating the heat of condensation

Gas meter reading at start	V_{GA1}	= l
Gas meter reading at end	V_{GE1}	= l
Mass of condensate collected	m_K	= ....g
Volume of burnt gas	V_{G1}	= $V_{GE1} - V_{GA1}$ = ...l

Heat of condensation per 1 m^3 of burnt gas

$$Q_K = \frac{2.454 \text{ kJ/g} \cdot m_K}{V_{G1} \cdot f_1}$$

= .... kJ/m^3

Gas meter corrective factor f_1 = ...

Calculating the net calorific value

Gas volume measured V_{G2} = ...l

Temperature readings

	I		II		III	
	t_{WE}	t_{WA}	t_{WE}	t_{WA}	t_{WE}	t_{WA}
	in °C		in °C		in °C	
1	......	......	......	......	......	....
2	......	......	......	......	......	....
3	......	......	......	......	......	....
Date:.......... 4	......	......	......	......	......	....
5	......	......	......	......	......	....
Name:.......... 6	......	......	......	......	......	....
7	......	......	......	......	......	....
8	......	......	......	......	......	....
9	......	......	......	......	......	....
10	......	......	......	......	......	....
Arithmetic mean °C	......	......	......	......	......	....
Corrected °C	......	......	......	......	......	....

Water heating

$\Delta t = t_{WA} - t_{WE}$ ...... K ...... K ......K

Water mass m_W .... g ....g ......g

Gross calorific value

$$Q_s = \frac{m_W \cdot \Delta t \cdot C_W}{V_{G2} \cdot f_1}$$ ....

(unreduced)

Arithmetic mean .... kJ/m³

Net calorific value

(unreduced)

$Q_i = Q_S - Q_K$ .... kJ/m³

reduced

$Q_{s,n} = Q_S \cdot F_R$ .... kJ/m³

Convert to 0 °C, 101.325 kPa

$Q_{i,n} = Q_i \cdot F_R$ .... kJ/m^3

Corrective factor f_b

$Q_{S,nk} = Q_{s,o} \cdot f_b \cdot f_2$ .... kJ/m^3

Corrective factor f_2

$Q_{i,nk} = Q_{i,o} \cdot f_b \cdot f_2$ .... kJ/m^3

8. Accessories Supplied

1 pc of thermometer, +5 °C to +20 °C, graduated in 1/10 °C, with manufacturer's test certificate
1 pc of thermometer, +15 °C to +30 °C, graduated in 1/10 °C, with manufacturer's test certificate (water temperature)
1 pc of thermometer, +20 °C to +40 °C, graduated in 1/10 °C, with manufacturer's test certificate
1 pc of thermometer, 0 °C to 100 °C, (waste gas temperature)
4 pcs of rubber stoppers, fitted with hole
1 pc component part for lean gas burner
1 pc rich gas burner head
1 kit of burner nozzles (9 pieces)
1 pc of measuring vessel 2000 cm^3
1 pc of measuring vessel 100 cm^3
3 m PVC hose, 10 mm inner diameter
2 pcs of gaskets 14 x 18
1 pc operating instructions

9. Inspections

9.1. Daily Inspection

Check the level of the sealing liquid in the overflow at the rated level according to Section 6.1.3.

9.2. Monthly Inspection

Check the packing of the gas meter drum shaft. In case of leakage, top up the special lubricant supplied with the gas meter. The lubricant may be topped up also while running the gas meter (Figure 7).

9.3. Quarterly Inspection

The gas meter sealing liquid and, therefore, the interior of the apparatus will be contaminated to a varying extent, depending on the calorimeter use. As a consequence, the sealing liquid should be replaced in a three months interval. To this end, following procedures have to be performed.

9.3.1. Emptying the Manometer

The manometer is emptied by unscrewing the screw 26 (Figure 1).

9.3.2. Replacing the Sealing Liquid and Cleaning the Gas Meter

Both gas and water shut-off valves are closed and the gas and water screw joints loosened. Discharge the gas meter sealing liquid through both the drain cocks. Next, the gas meter is rinsed for several times using a soda solution. Caution should be used when cleaning the gas meter in order to prevent the drum from getting damaged.

10. Trobleshooting Guide

10.1. Incomplete Combustion

Check burner flame and pressure regulator adjustments first. Then, check the burner nozzle being used for the appropriate size (i.e. the Reference Number), which must correspond to the gross calorific value (Section 6.4.). Inspect the gas passages in the heat exchanger tubes for any obstructions caused by deposites.

10.2. Variations in Gross Calorific Value

Excessively high or low water level in the gas meter; remedy is by checking the sealing liquid level at the overflow at the water rated level.

Leaky burner head; remedy is by inserting a new burner or burner head.

10.3. The Measuring Accuracy Certified is Not Available

The three-way cock 16 is changed over either too soon or too late. Thermometers are not read off exactly. Auxiliary instruments are loaded with errors (e.g. thermometer does not work accurately, leaky gas meter).

Remedy: The procedures specified by the operating instructions should be strictly followed when operating the calorimeter. Replace faulty components.

10.4. Unsteady Thermometer Readings at the Water Outlet

Cause: Irregular water inflow or poor mixing of the heated water; heavy variations in pressure.

Remedy: Insert a new water mixer. Mount a water storage tank (Section 5.4.). Remove deposites caused by water (e.g. scale) in the inner nest of tubes (Section 10.3.).

11. Setting the Calorimeter Out of Operation

When the unit is intended to be out of use for a longer period of time, the parts having a metallic luster should be covered using an acid-free oil and both heat exchanger and gas meter emptied. The thermometers are stored in the cases supplied. Connecting sockets are closed by stoppers after disconnecting the hoses.

Table 3. Partial pressure of steam p_D in the state of saturation

t_g (°C)	p_D (kPa)	t_g (°C)	p_D (kPa)	t_g (°C)	p_D (kPa)
0	0,61	10	1,23	20	2,33
1	0,66	11	1,31	21	2,48
2	0,71	12	1,40	22	2,64
3	0,76	13	1,50	23	2,81
4	0,81	14	1,60	24	2,99
5	0,87	15	1,71	25	3,17
6	0,93	16	1,81	26	3,36
7	1,00	17	1,93	27	3,56
8	1,07	18	2,06	28	3,77
9	1,15	19	2,20	29	3,00

Laboratory Precision Calorimeter for Liquid Fuels (Figure 8)

0. Occupational Health and Safety

The occupational health and safety is referred to in sections 5.2., 6.2., 6.3., 7.3., and 9.

1. Purpose

Liquid fuels have a varying heat of combustion depending on their types and compositions. This heat of combustion is analyzed as the gross calorific value. For analyzing this value, the laboratory precision calorimeter is used offering an extremely high accuracy.

2. Application

The net calorific value analysis is necessary in

- petroleum processing plants
- oil refineries
- chemical plants, paraffin plants, laboratories
- methylated spirits plants
- light or heavy engine plants, motor vehicle industries
- research and development stations

However, only those vegetable, ammimal, or mineral oils can be analyzed which completely vaporize at a boiling point of 250 °C without leaving any carbon or other residues. The laboratory precision calorimeter cannot be used to analyze the gross calorific values of refined colza oil, tung oil, bone oil, or mazut oil.

3. Theory of Operation and Design (Figure 8)

The gross calorific value of a liquid fuel is analyzed by determining the mass of water, the mass of the fuel required, and the temperature difference. The gross calorific value is calculated according to following formula:

$$Q_S = \frac{m_W}{m_B} \cdot \Delta t \cdot C_W$$

where

Q_S = Gross calorific value
m_W = Quantity of water in kg
m_B = Quantity of fuel burnt in kg
Δt = Temperature difference in K
C_W = Specific heat of water (4.1868 kJ/kg K)

The gross calorific value if a liquid fuel is directly analyzed by using the laboratory precision calorimeter in the same way as it is in analysis on gases. The heat produced by burning the fuel is transferred to the water continuously circulated in the calorimeter. Once the heat exchange has entered steady conditions, the heat conducted by the water will be equal to that generated by the burnt liquid fuel.

The calorimeter for liquid fuels includes the heat exchanger, pressure vessel with burner, and precision balance with accessories. Their arrangement is illustrated in Figure 8. The water enters the collecting vessel 3 passing the pipe 2. The overflows included in the collecting vessel ensure that the heat exchanger is supplied with a sufficient amount of water. The remaining water is drained via pipe 4.

The liquid fuel required to heat the flow water is stored in the pressure vessel 5 and moved to the burner, where it will be gasified and burnt, by a certain overpressure produced by a pressure pump.

The quantity of water to be heated in the heat exchanger is adjusted through batching cock 6 and passed into the discharging vessel 7 as well as via the three-way cock 8 into a measuring vessel. For determining the temperature difference between the cold water inlet and hot water outlet, temperatures indicated by thermometers 9 and 10 are read off.

The waste gas produced by burning the fuel is discharged into the atmosphere through the waste gas outlet. The thermometer 11 is to measure the waste gas temperature. The outlet 12 is provided for discharging the condensate.

The precision balance is to determine the amount of liquid fuel used by differential weighing. The value found is put into the equation.

4. Specifications

Site	- A light room, free of draught, with heating facilities. The apparatus must not be exposed to direct solar radiation.
Space requirements	- Flat worksurface, 0.8 x 1.5 m
Total weight	- approx. 17 kg
Environmental temperature range	- +10 °C to +35 °C
Water temperature	- Preferrably 3 to 6 K below room temperature
Quantity of water required	- Aprox. 35 litres per analysis
Quantity of liquid fuel	- 10 g per analysis
Water supply	- PVC tube, 10 mm inner diameter
Error	- ±1 %
Measuring ranges	- Refer to Table 5
Class of application	- +10/ +35/ +30/ 80/ 1101

Table 5.

Type of fuel	Calorific value (about, MJ/m^3)	Diameter of burner nozzle (mm)
Benzene	42	
Benzine	42	
Kerosene	42	
Toluene	43	0.3
Benzene/benzine mixture	44	
Petroleum	46	
Gasoline	47	
Oil of turpentine	42	0.4
Methylated spirit/methanol mixture	19 - 25	1.0
Methylated spirit	21 - 28	
Methanol	17 - 23	
Ethanol (alcohol)	27	1.3

5. Setting Up

5.1. Unpacking and Inspection

The apparatus is unpacked and inspected for any damages. All parts and components delivered are checked for completeness as specified by the acknowledgement of order.

5.2. Installing the Laboratory Precision Calorimeter

For installing the complete apparatus a flat worsurface of about 0.8 by 1.5 m is required. The site should be a light and draught-free room with heating facilities available.

Note the calorimeter not to be exposed to direct solar radiation. Users are advised to set up the individual calorimeter components in the order shown in Figure 1.

A sufficient air reversal must be guaranteed in the room without any draught. The bench surface must not consist of flammable material. Otherwise it has to be covered with a protective sheet made from fire-resistant material. No inflammable objects should be placed on the bench.

5.3. Heat Exchanger

Insert the feet in the appropriate places. For water drain, one hose each is connected to both the outlets of the three-way cock 8.

5.4. Water Connections

The water supply is connected to the heat exchanger 1 by fitting a hose to the pipe 2. A storage tank having a capacity of about 120 litres can be placed approximately 3 m above the calorimeter and the water fed from there in order to keep both pressure and temperature of the inflowing water as constant as possible. A float valve can be inserted providing a mean of controlling both water level and feed. If necessary, a thermostat can be installed to ensure a constant temperature of the water entering the calorimeter.

5.5. Fitting the Thermometers

Make sure that the mercury column is not disrupted before inserting the thermometers into the heat exchanger. The thermometers 9 and 10 are fitted into the apertures provided in the heat exchanger cover using the rubber stoppers supplied. The thermometers are advised to be greased slightly in order to facilitate pushing the rubber stobbers onto the thermometers. The thermometer 11 is inserted in the same way.

5.6. Precision Balance with Pressure Vessel

For gross calorific value analysis, the precision balance 13 (Figure 8) is prepared accordingly. The left scale pan is replaced by the pressure vessel using the suspension 14. The counterweight 15 is put on the right scale pan to balance the masses. The precision balance is tared by use of the smaller weights in order to cause the index to come to zero.

6. Preliminaries prior to Setting the Calorimeter into Operation

Following checking procedures have to be carried out on all devices included in the analyzing apparatus before setting the laboratory precision calorimeter into operation.

6.1. Filling the Heat Exchanger with Water

The three-way cock 8 on the heat exchanger 10 (Figure 6) is set to the "Abwasser" /Drain/ position. The batching cock is put to "O". Once the shut-off valve in the water supply line is opened the batching cock 6 is set to scale mark 4 in order to prevent air pockets from developing by slowly pouring the water into the heat exchanger.

The water flow should not be stopped as long as bubbles are seen forming on the surface of the water in the drain vessel 7. Water must not come out of outlet 12, otherwise there will be a leakage at the heat exchanger which must be eliminated immediately. Close the shut-off valve included in the water supply line after the heat exchanger has been filled.

6.2. Installing the Precision Balance with Pressure

Vessel (Figure 9)

The pressure vessel is removed from the precision balance set up according to Section 5.6. and filled with the liquid fuel intended to be analyzed. For this purpose, the screw plug 1 is unscrewed and about 150 to 200 cm^3 of filtered fuel poured in.

Attention

Do not pour in the fuel with a naked flame immediately nearby!

Avoid overflows while pouring in fuels or spirits (Section 6.3.). Completely remove any fuel or spirit in case an overflow has occured. Carefully dry the pressure vessel prior to the ignition.

The filler neck is then tightly closed using the appropriate cap and the pressure vessel suspended in the precision balance as described under Section 5.6. The precision balance is tared by using the smaller weights until the index will be adjusted to zero. The precision balance with the pressure vessel suspended is set up at the right of the heat exchanger so as to dead centrally extend the burner 2 (Figure 9) into the heat exchanger combustion chamber 1 (Figure 8). The precision balance has to be accurately levelled using the adjustable feet. The exact central position of the burner is checked by holding a mirror below the heat exchanger. Verify the burner nozzle used to meet the gross calorific value requirements (Section 7.3.) before commencing the analysis.

6.3. Setting the Pressure Vessel into Operation

The pressure vessel is again removed from the precision balance. The heater collar 3 (Figure 9) below the burner head is filled by half with methylated spirit and the spirit then ignited. When the spirit has got almost completely burned an overpressure is generated in the pressure vessel by means of the pneumatic pump thus forcing the liquid fuel to move to the burner surface which has been heated by the spirit already burnt. Here the spirits is vaporized and burnt. Air is pumped into the pressure vessel until a steady and roaring flame can be seen, having a blue-green cone (maximum manometer reading 100 kPa).

6.4. Preliminaries

The shut-off valve in the water supply line is opened again and the three-way cock set to "Abwasser" /Drain/ position. Now the pressure vessel set into operation according to Section 6.3. is suspended in the precision balance and the burner dead central position in the heat exchanger combustion chamber checked by using the mirror as described. This is a necessary precaution, since a non-centric burner position would cause the flame to incidentally touch the combustion chamber wall. Consequently, the calorific value analysis would be erroneous.

The water outlet temperature reading indicated on thermometer 10 (Figure 8) will increase and enter steady conditions after a few minutes time. Now the continuous water flow accepts as much heat as generated by the burning flame. The water throughput is adjusted by use of the batching cock 6 so as to obtain a temperature difference of about 10 K between the cold water inlet and the hot water outlet.

7. Gross Calorific Value Analysis on Liquid Fuels

7.1. Procedure to be observed

The temperature difference stated according to Section 6.4. is monitored and, if necessary, adjusted using batching cock 6 (Figure 8), until the temperature difference is about 10 K and keeps to be the same reading. The condensate must steadily drip off from outlet 12 (Figure 8). The three-way cock 8 still remains in position "Abwasser" /Drain/. The pan 4 (Figure 9) is loaded with a number of small weights having a few grams each to cause the pressure vessel to have a slight excess weight during this period of combustion and, therefore, the left balance arm to drop a little. When the amount of fuel corresponding to these weights is used up the balance will return to its equilibrium position and the index coincide with the zero mark.

NOW THE ANALYSIS CAN BE STARTED!

The pan on the pressure vessel is loaded with 10 g (Figure 9) and the three-way cock 8 (Figure 8) set into position "Meßwasser" /Collected water/ and the water collected in a measuring vessel simultaneously. At the same time, the dripping condensate required for the gross calorific value analysis is collected in the measuring vessel 100 cm^3 supplied and reading off the temperatures indicated on both thermometers started.

Reading off the temperatures and collecting the condensate is maintained up to the moment the balance will have reached its equilibrium condition and the index coincided with the zero mark. At that time, the three-way cock is set to the "Abwasser" /Drain/ position and the measuring vessel removed from below the condensate outlet.

Both temperature readings and water collections are carried out for two or three times and recorded in appropriate manner, which an example for is given below. However, the quantity of condensate is determined only once while burning 10 g of fuel.

Quantity of flow water collected:	m_W = 10.500 g
Quantity of fuel used:	m_B = 10 g
Quantity of condensate collected while burning 10 g of fuel	m_K = 12.7 g
Water inlet temperature (arithmetic mean)	t_{WE} = 15.3 °C
Water outlet temperature (arithmetic mean)	t_{WA} = 25.3 °C
Temperature difference (arithmetic mean)	$t = t_{WA} - t_{WE}$ = 10 K

7.2. Calculating the Gross and Net Calorific Values

The laboratory precision calorimeter is used to analyze the gross calorific value of liquid fuel. The waste gas is cooled down to the temperature of the gaseous fuel and air for combustion pouring in. The water vapour contained in the combustion products is completely condensed so that the heat of condensation of the water vapour is transferred to the flow water and, therefore, included in the gross calorific value.

The gross calorific is the calculated by means of the equation and the values found according to Section 7.1. as follows:

Gross calorific value:

$$Q_S = \frac{m_W \cdot t \cdot c_W}{m_B} =$$

$$Q_S = \frac{10.5\ kg \cdot 10\ K \cdot 4.1868\ kJ}{10\ g \cdot kg \cdot K}$$

$$Q_S = 43{,}961\ kJ/kg$$

At first, the heat of evaporation Q_K is calculated from the quantity of condensate collected in the first analysis in order to determine the net calorific value.

Heat of evaporation:

$$Q_K = \frac{r \cdot m_K}{m_B}$$

where

r = Heat of condensation of water (2.454 kJ/g at 20 °C)

m_K = Mass of condensate collected in grams

The net calorific value is calculated as follows:

$$Q_i = Q_S + Q_K$$

Following result is obtained:

$$Q_i = 43{,}961\ kJ/kg = \frac{2.454\ kJ/kg \cdot g^{-1} \cdot 12.7\ g}{10\ g} = 40{,}844\ kJ/kg$$

7.3. Selecting the Appropriate Burner Nozzle and Replacement

The burner nozzles supplied have inner diameters of 0.3 or 0.4 m and from 1.0 through 1.3 mm. The appropriate burner nozzle is selected depending on the fuel or fuel mixture as indicated by Table 5. Generally, for fuels inculded in the heavy hydrocarbons series, e.g. petroleum, petrol etc., the nozzles being smaller in diameter should be used. On the other hand, for fuels containing water, e.g. methylated spirit, nozzles having a larger diameter are selected. With the nozzle properly sized according to the fuel intended to be analyzed, the exit velocity generated will draw as much air as is necessary to get the fuel burned with a roaring flame. In case an inappropriate burner nozzle is selected, either too much or too little air will be drawn causing a luminous or flaring flame, respectively. If this occurs, the combustion will be insufficient and can be readjusted by replacing the nozzle only.

Care should be taken when replacing the burner nozzle in order to avoid any leakage, as this may result in erroneous analysis.

7.4. Test on Fuel Mixtures

An additional error may occur while analyzing fuel mixtures if the mixture shows a tendency to separation.

It is advisable to check the mixture in question for this tendency. For this purpose, allow the mixture to rest for 24 hours. Then, take samples from the surface, centre and bottom of the liquid and analyze their densities.

In case the densities found coincide it can be assumed that there is no tendency to separation.

7.5. Precautions when Analyzing Different Fuels

When the calorific values of different fuels are intended to be analyzed successively the pressure vessel should be carefully rinsed using the fuel subjected to analysis next and filled for analysis thereafter.

8. Accessories Supplied

1 pc of thermometer, 5 °C to 20 °C, graduated in 1/10 °C, with manufacturer's test certificate (for water temperature measurement)
1 pc of thermometer, 15 °C to 30 °C, graduated in 1/10 °C, with manufacturer's test certificate (for water temperature measurement)
1 pc of thermometer, 25 °C to 40 °C, graduated in 1/10 °C, with manufacturer's test certificate (for water temperature measurement)
1 pc of thermometer, 0 °C to 100 °C (for waste gas temperature measurement)

1 pc of pneumatic pump
1 pc of measuring vessel of 2000 cm^3 capacity
1 pc of measuring vessel of 100 cm^3 capacity
4 pcs of burner nozzle (0.3; 0.4; 1.0; 1.3 mm in diameter)
3 pcs of cleaning wire (0.3; 0.4; 1.00 mm in diameter)
4 pcs of rubber stoppers, fitted with hole
3 m of PVC hose, 10 mm inner diameter
2 pcs of gasket A 14x18
1 pc of gasket C 14x20
1 pc of test certificate
1 pc of operating instructions

9. Setting the Calorimeter Out of Operation

The pressure vessel screw cap must not be loosened unless the flame is extinguished due to the fuel being completely used up. Under no circumstances the screw cap is removed with a naked flame nearby.

The heat exchanger is advised to the emptied when the calorimeter is intended to be out of operation for a longer period of time. Store the thermometers in the cases supplied. The hose connections are closed using the stoppers supplied after having removed the hoses. In any case, the pressure vessel is decompressed. Thereafter, all parts having a metallic luster are covered using an acid-free oil.

10. Inspections on the Calorimeter Pripr to Setting It Into Operation or While Running

10.1. Daily Inspection

When the laboratory precision calorimeter is continuously being used for calorific value analysis, both burner head and nozzle will have to be cleaned daily. The nozzle is cleaned using the appropriate cleaning wire. A foul burner head, particularly fouled by deposits which are caused by the combustion of heavy oil, is removed from the pressure vessel and acrewed onto a pipe, heated up to red heat and blown with compressed air thereafter. The deposits will get burned in most of the vases. The burner nozzle must be unscrewed prior to this cleaning operation. If this cleaning operation will be completed unsuccessfully, the burner head in question is replaced by a new one.

10.2. Annual Inspection

Deposits caused by the water passing the heat exchanger will be often found on the nest of tubes after the calorimeter has been continuously ron for a longer period of time. These deposits adversely affect the heat transfer from the waste gas to the flow water. Therefore, the nest of tubes is required to be cleaned thoroughly.

10.3. Cleaning the Heat Exchanger (Figure 1)

Following instructions are applicable to the laboratory precision calorimeter for liquid fuels and gases.

10.3.1. Set the laboratory precision calorimeter out of operation, empty the heat exchanger and unscrew the cock 27 (Figure 1).

10.3.2. Lift off thermometers 17 and 18.

10.3.3. Unscrew connections at batching cock 14 and drain vessel 15 as well as the screw at the support.

10.3.4. Remove the tree screws on the cover and remove the cover.

10.3.5. Lift off the nest of tubes from the outer casing.

10.3.6. Unscrew the screws holding the flange at the bottom of the nest of tubes and draw out the nest of tubes.

10.3.7. Again rinse the nest of tubes with soda solution for several times and pass pure water through thereafter. On completion, reinsert the nest of tubes and mount the heat exchanger in following order.

10.3.8. Place the gasket on the flange. If necessary, use new gasket.

10.3.9. Replace the nest of tubes. Use the same power when tightening each of the screws in order to firmly connect the flange to its counterflange.

10.3.10. Fill the interior of the heat exchanger with water and check the seal at the flange.

10.3.11. After having completed the leakage test, empty the heat exchanger.

10.3.12. Insert the inner assembly of the heat exchanger and not the combustion chamber to firmly rest on the condensate groove located in the heat exchanger bottom.

10.3.13. Again attach both batching cock and drain vessel to the heat exchanger using the screwing provided. Mount the overflow and outlet to the support with the screw.

10.3.14. Replace the cover and fasten to the outer casing by means of the three screws, reinsert the thermometers and restore the hoses to the appropriate outlets.

11. Troubleshooting Guide

11.1. Incomplete Combustion

There are several causes.

11.1.1. The flame is luminous, insufficient air is drawn.
Remedy: Replace the burner nozzle with that of a smaller diameter in order to get more air drawn and, therefore, to adjust the appropriate gas/air ratio.

11.1.2. Unsteady flame, air is drawn in excess.
Remedy: Insert a larger nozzle. Perhaps reduce pressure in pressure vessel.

11.1.3. Intermittent flame due to insufficient preheating or to excessively high pressure in the pressure vessel.
Remedy: Reduce pressure in pressure vessel as indicated under Section 11.1.2. or preheat again.

11.2. Variations of Errors in Calorific Value Analyses

Insufficient heat transfer from the waste gas to the flow water is caused by residues from the water, e.g. boiler scale, deposited in the nest of tubes of the heat exchanger.

Remedy: Clean the heat exchanger as indicated under Section 10.3.

11.3. The Measuring Accuracy Certified is Not Available

This may be caused by changing-over the three-way cock too soon or too late or by reading off the thermometers incorrectly. Furthermore, the precision balance is checked for proper operation and the precision gas meter for leakage.

11.4. Unsteady Water Outlet Thermometer Reading

This may be caused by an irregular water supply or a faulty heat exchanger.

Remedy: The heat exchanger is required to be repaired.

Laborator Precision Calorimeter for Liquid and Gaseous Fuels

1. Purpose

The laboratory precision calorimeter is often required to carry out gross calorific value analyses on gaseous or liquid fuels. Therefore, the laboratory precision calorimeter for gases is provided with additional equipment in order to extend the range application to liquid fuels also.

2. Accessories Supplied

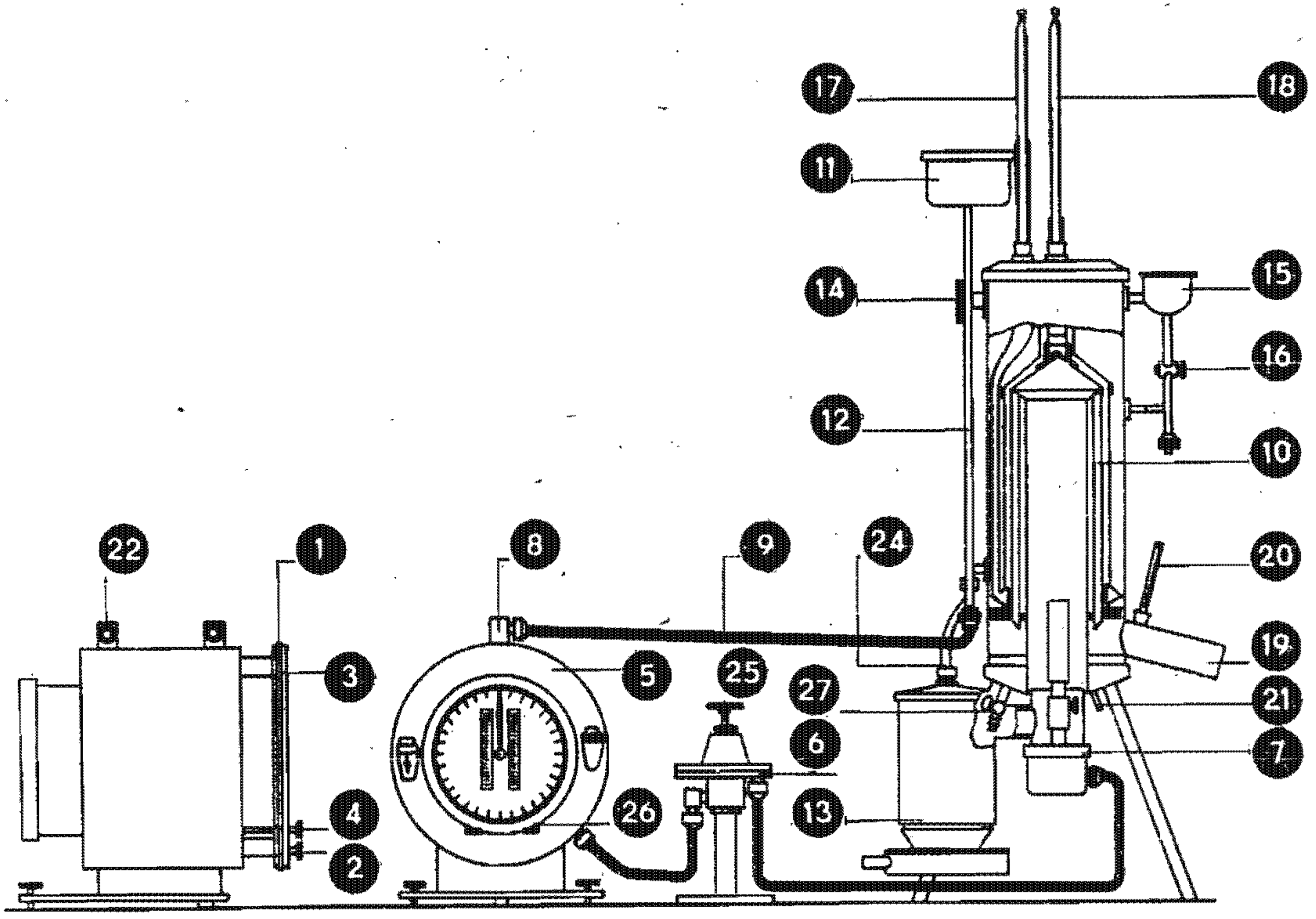
1 pc of laboratory precision calorimeter for gases
1 pc of pressure vessel fitted with burner

1 pc of precision balance
1 pc of counterweight
1 pc of suspension for pressure vessel
1 pc of hose liner
1 pc of gasket C 14x20
3 pcs of burner nozzle (0.3; 0.4; 1.0 mm in diameter)
3 pcs of cleaning wire (0.3; 0.4; 1.0 mm in diameter)
1 pc of pneumatic pump for pressure vessel

3. Installing the Laboratory Precision Calorimeters

The laboratory precision calorimeter is reset by simply replacing the appropriate components according to the medium intended to be analyzed. In any case, refer to the operating instructions before resetting the calorimeter.

Bild 1



Laboraty Precision Calorimeter for Fuel Gases

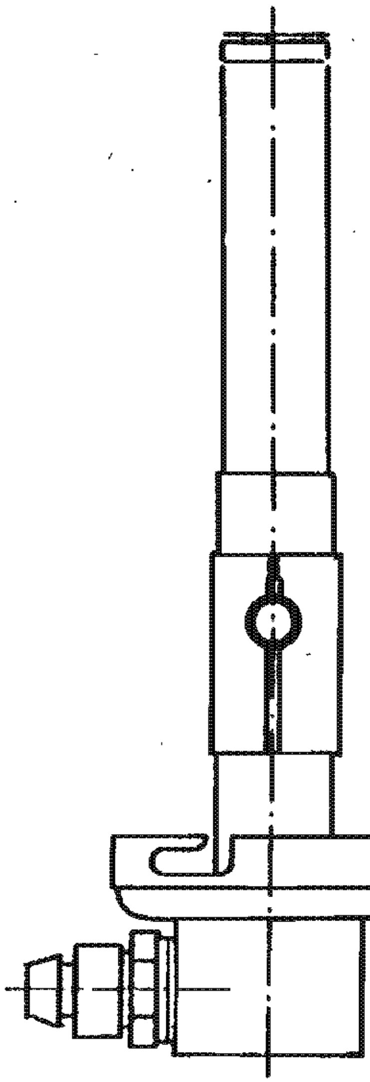


Bild 2

Burner for normal gases

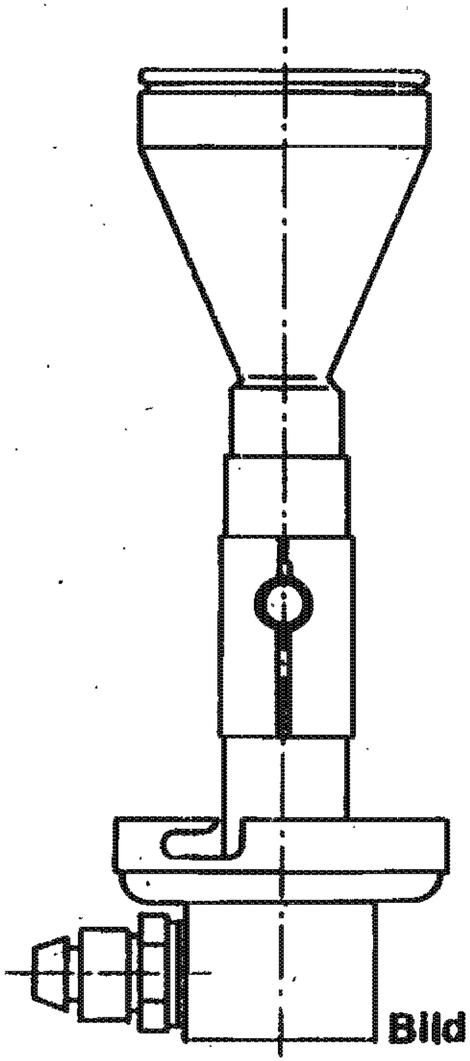
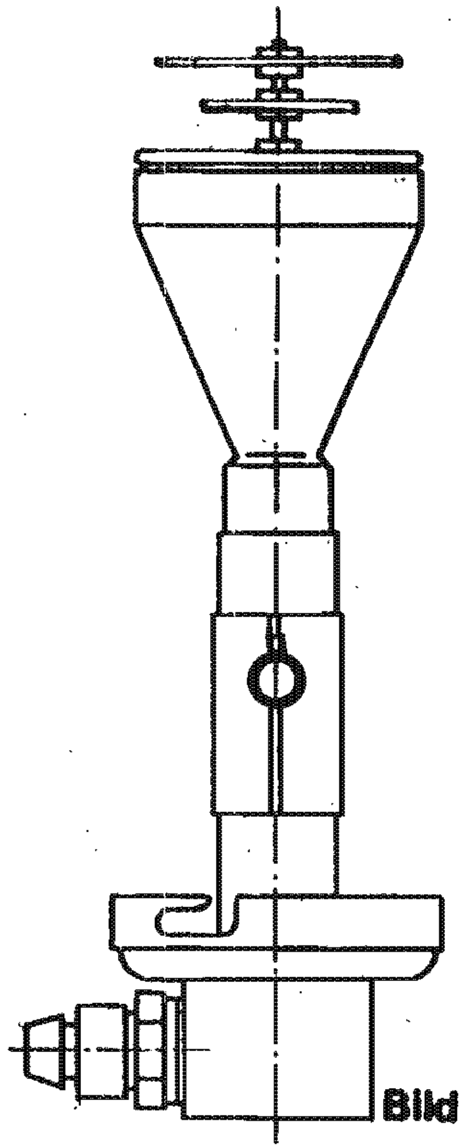


Bild 3

Burner for rich gases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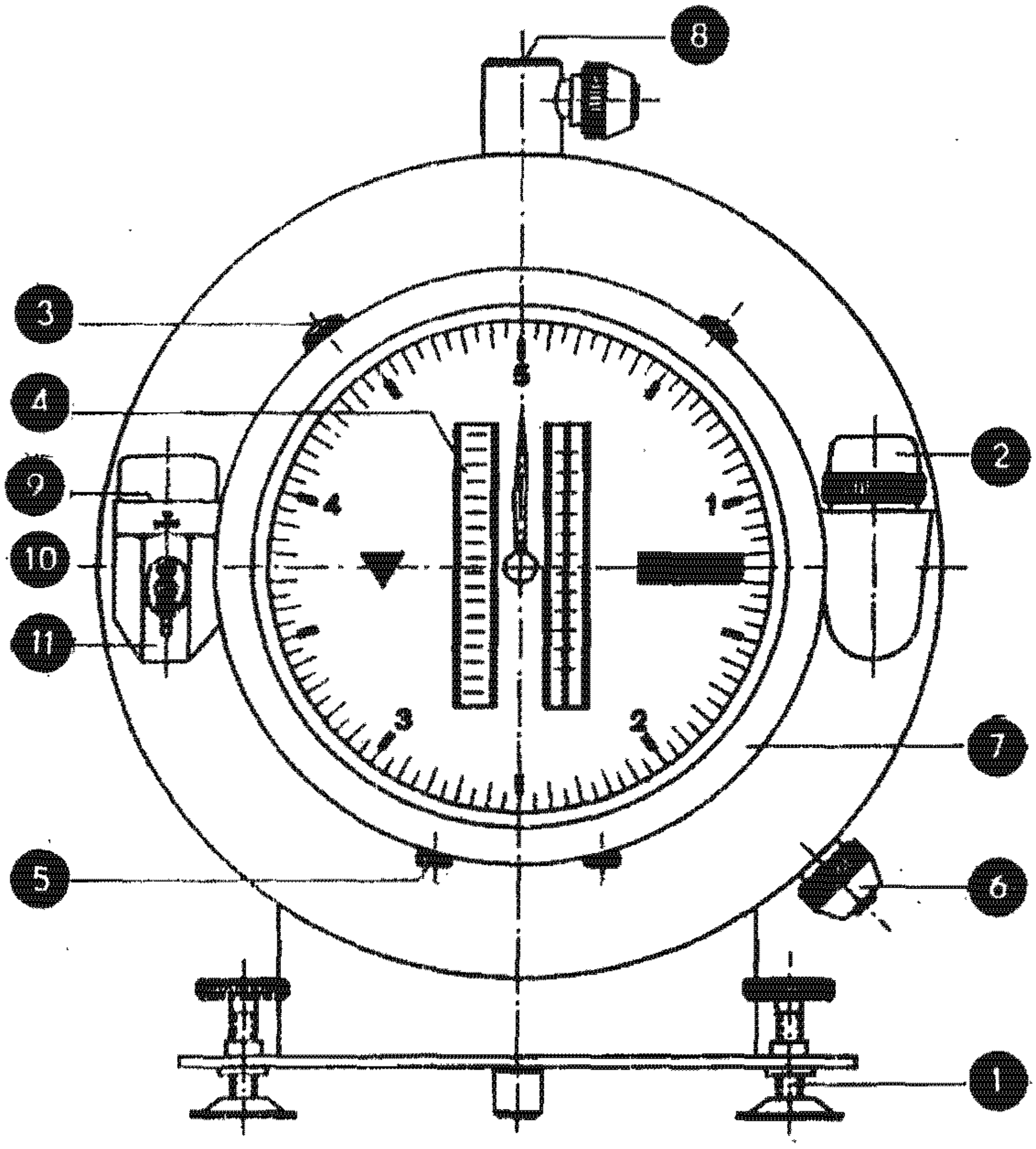
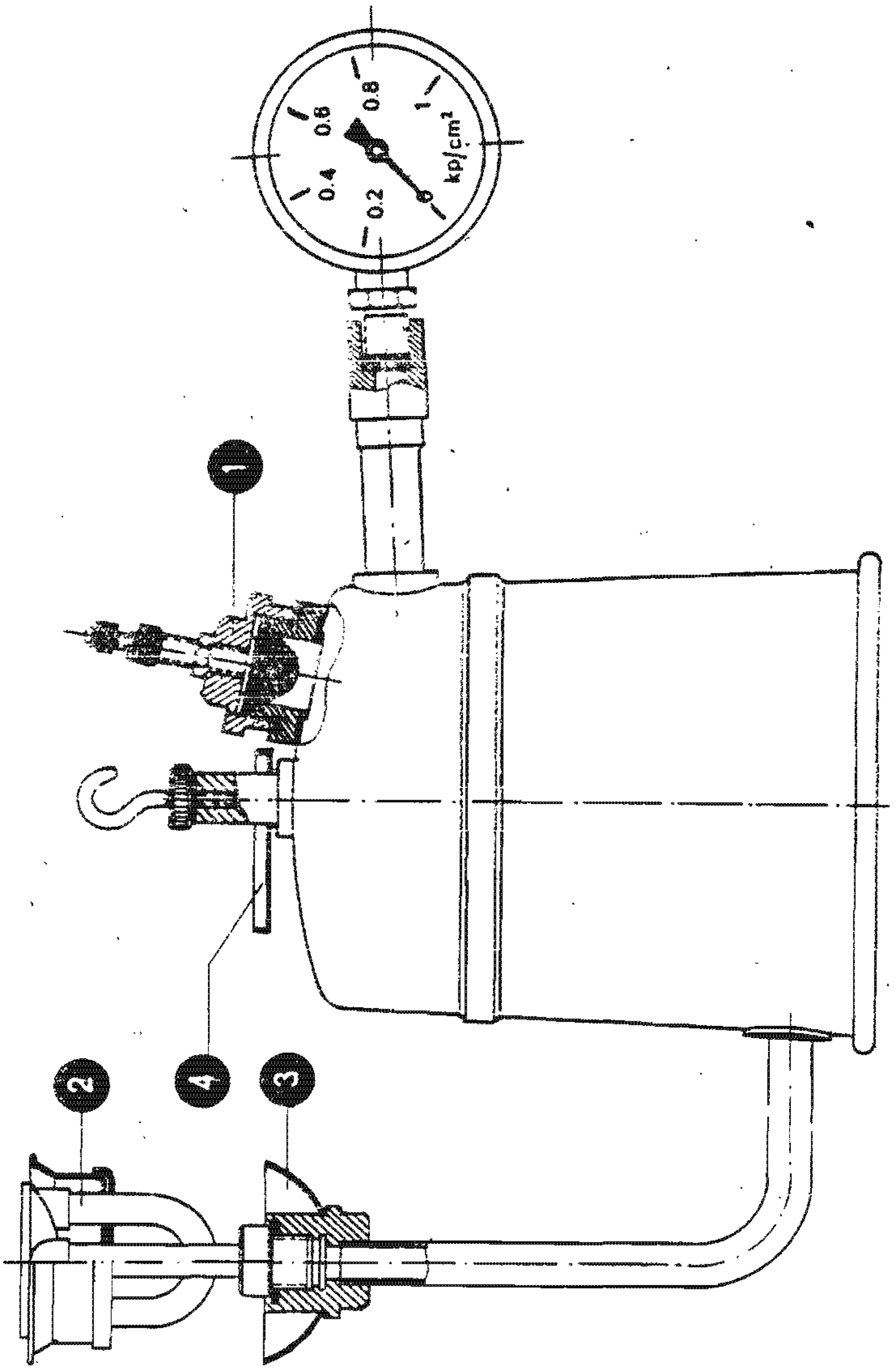
Burner for lean gases

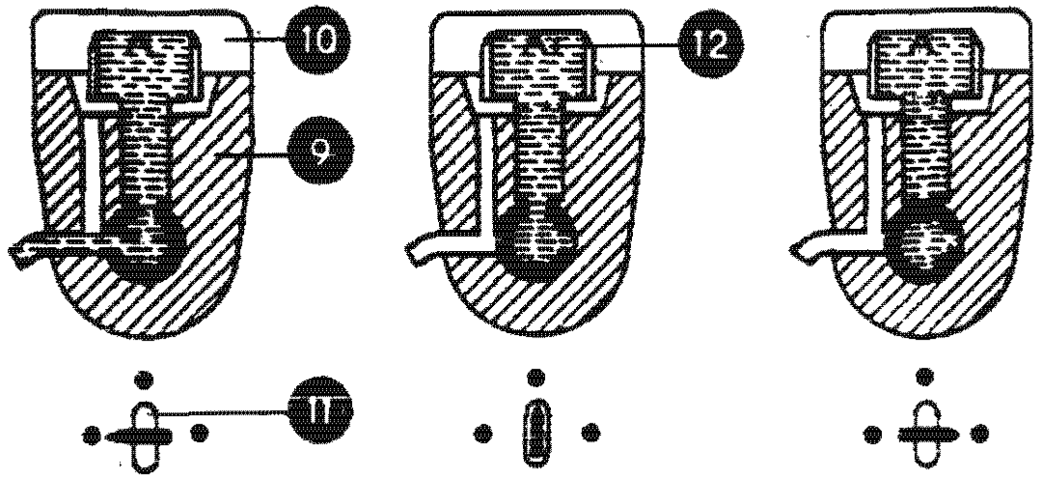


Precision gas meter **Bild 5**



Burning unit

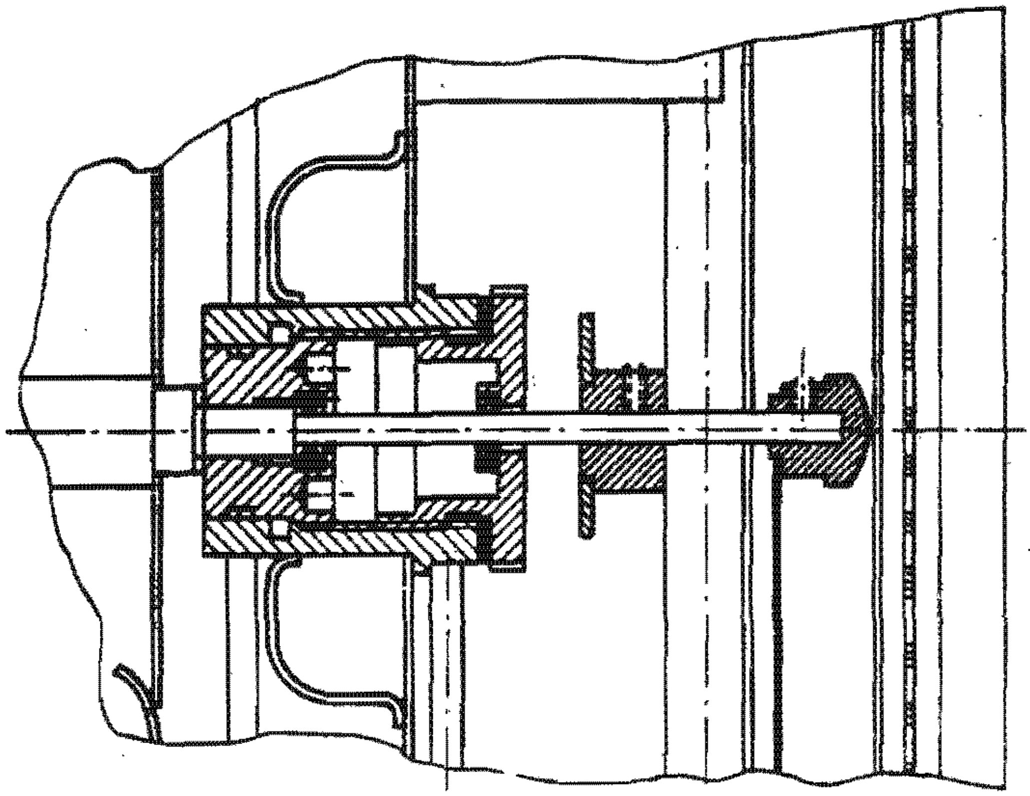
Bild 9



Let out Testing Operation

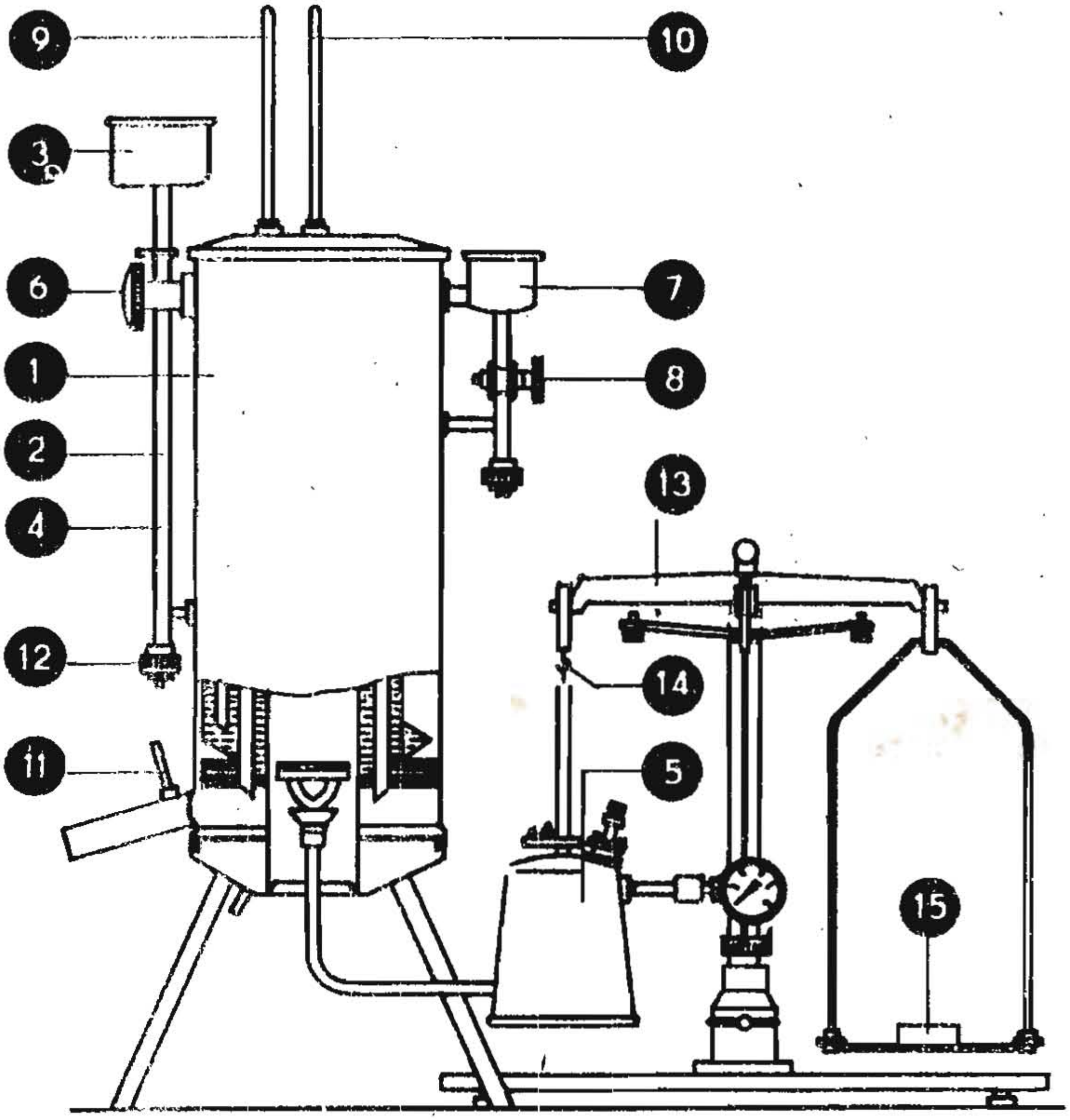
Rated value overflow of precision gas meter

Bild 6



Shaft stuffin box

Bild 7



Laboraty Precision Calorimeter for Liquid Fuels

Bild 8