

Estimation of the uncertainty for an isothermal precision gas calorimeters

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Abstract

The paper presents a brief description of a new method of measurement of the heat of combustion of gases and the design of a new gas calorimeter. The results of measurements of the thermal power and the heat of combustion of methane are analyzed with the aim to determine their uncertainties. It is proposed to divide the sources of uncertainty in three groups depending on the method of their processing. The investigations carried out prove the correctness of the measurement of both the thermal power and the heat of combustion of methane. The following values of the uncertainties of the heat of combustion of methane were determined: the relative standard uncertainty Type A $\leq 0.02\%$ and the combined relative standard uncertainty $\leq 0.04\%$. The obtained results permit recommending the proposed gas calorimeter as a prototype of a standard gas calorimeter. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Gas calorimetry; Heat of combustion of methane; Uncertainty; Accuracy of measurement results

1. Introduction

The paper considers the results of measurements obtained by using a new gas calorimeter based on a new method of measurement. Therefore, it is expedient to present briefly at first the method of measurement and the design of the calorimeter and then to pass to the analysis of the results of the measurements.

The measurement method consists in that the heat of combustion of gas is transferred into the latent heat of the phase transition liquid to gas. This latent heat is then removed from the system by using the Peltier effect. The compensation is performed by automatically keeping the temperature of this phase transition constant. Because of a nonuniform distribution of the

temperature in the combustion chamber it is impossible to perform the direct compensation of the heat of combustion of gas. The optimum solution of the problem has been found in using a heat pipe. According to the principle of action of a heat pipe, there are three zones: heating zone, transition zone and cooling zone. Besides, the thermal conductance of heat pipes is extremely high, it is even greater than such well-known excellent thermal conductors as copper and silver have. Therefore, by using a device of this type it is possible to transfer very quickly and without loss the heat of combustion of gas into the heat of evaporation of the working liquid, to move the working liquid's vapor from the heating zone via the transition zone to the cooling zone in order to compensate the heat of condensation with the help of the Peltier effect. The liquid that is formed by condensation of the vapor is then returned again to the heating zone of the heat

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pipe. Thus, the following succession of heat processes Q takes place:

$$Q_{\text{Combustion}} \Rightarrow Q_{\text{Evaporation}} \Leftrightarrow Q_{\text{Condensation}} \Leftarrow Q_{\text{Peltier}} \quad (1)$$

The following design of the gas calorimeter (Fig. 1) has been chosen after a set of preliminary experiments. The main part of the calorimeter is a thermosyphon which is one of the types of a heat pipe. A heat exchanger with a gas burner and a compensation heater are placed in the lower part of the thermosy-

phon which is the heating zone of the heat pipe. The heat exchanger is dipped up to one half into the working liquid, which is freon-11 in this case. The cooling (condensation) zone is in the upper part of the thermosyphon. The cooling and condensation of the freon vapor is performed with the help of a pile of Peltier elements. A platinum resistance thermometer is used as a control thermometer and is placed in the upper part of the cooling zone of the thermosyphon. The condensation is performed at a given temperature which is automatically kept constant.

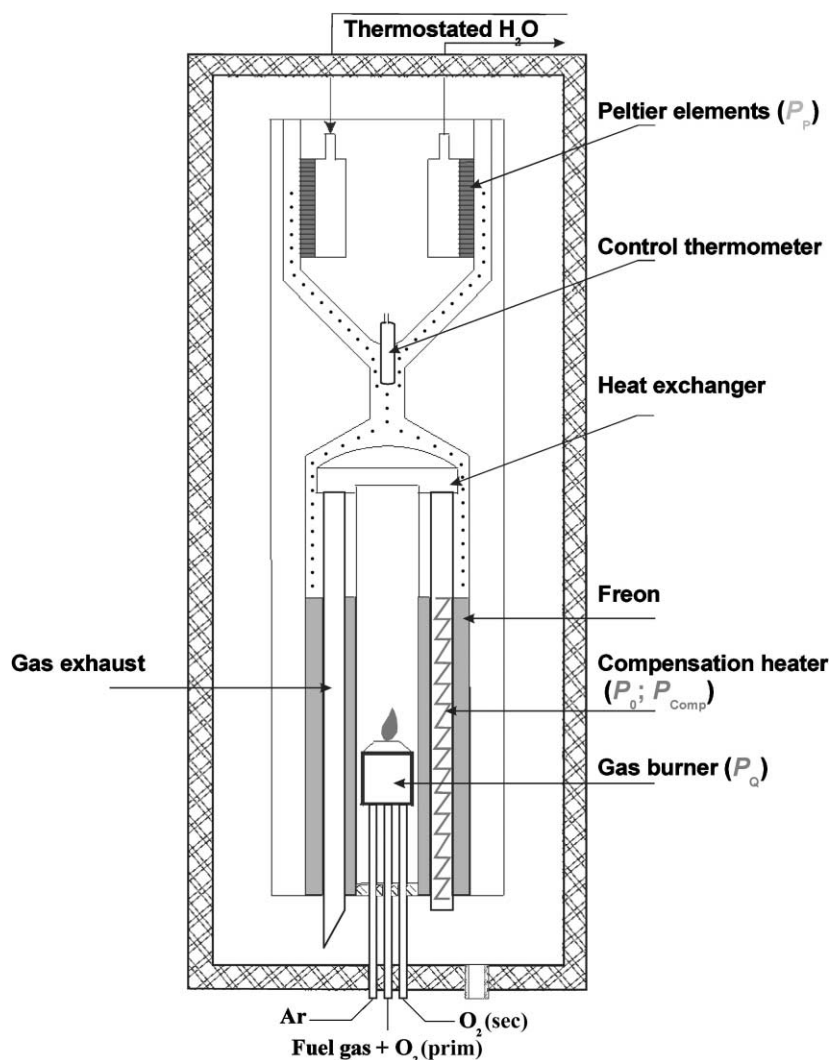


Fig. 1. Design of the isothermal gas calorimeter.

The thermosyphon is made of titanium and is thermally well insulated. The gas burner is designed for operating with the gas flow rate ranging from 1.5 to 3 l/h. The thermal power ranging from 8 to 42 W is given off from the gas burner depending on the heat of combustion of the fuel gas and its flow rate. Therefore, the maximum power of the compensation heater was set equal to 70 W.

When applying a constant current to the Peltier elements and keeping the temperature of condensation constant, a constant thermal power must be given off in the calorimeter. During the zero run (when the gas burner is switched off) this constant thermal power will be given off only by the compensation heater. Further, we shall denote this constant power as the base value (P_0). During combustion of gas, this constant thermal power consists of two parts: the thermal power given off by the gas burner (P_Q) and the thermal power (P_{Comp}) which is given off by the compensation heater as a compensation power in order to keep the temperature of the phase transition constant.

The thermal power which is given off by the burner (P_Q) is determined in this case as the difference between the base value (P_0), i.e. the thermal power from the compensation heater in the zero run, and the compensation thermal power from the compensation heater during gas combustion (P_{Comp}).

$$P_Q = P_0 - P_{\text{Comp}} \quad (2)$$

Let us proceed now, after this brief consideration of the calorimeter design and the method for the determination of the heat of combustion of gas, to the consideration of the experimental results and the estimation of their uncertainties.

2. Identification and analysis of the sources of uncertainty

The estimation of the uncertainty is performed in accordance with GUM-93 [1] in the succession shown in Fig. 2. The first step is the exact definition of the measurand. Then it is necessary to state as many sources of uncertainty as possible. Here a problem arises as to what should be further done with all these uncertainties. Proceeding from the recommendations expressed in GUM-93 ([1], 4.3.10) that “it is important not to “double-count” uncertainty components”,

the components of the uncertainties Type B given in Fig. 2 are divided into three groups. In our opinion, only the uncertainty components which are boldly printed should be directly considered as uncertainties Type B. The uncertainty components of the second group (normally printed) should be taken into account by the estimation of the uncertainty Type A. Finally, the uncertainties of the third group (italic type) can be decreased by using special calorimetric techniques up to such a value that it will be possible to neglect them. The uncertainties of the third group will be considered later.

Now let us consider the measurements of the thermal power with the help of the heat pipe and the uncertainties of these measurements.

3. Analysis of the uncertainty of the measurement of the thermal power

The first step is to determine the uncertainty during operation of the calorimeter in the zero run. The study consisted in evaluating the long-time stability of the base value (P_0). The results of the measurements are presented in Fig. 3. It should be noted that the effect of the fluctuations of the ambient temperature on the base value has not been established because the correlation factor of these two quantities was equal only to 0.33. This proves the sufficiently good thermal insulation of the calorimeter.

When calculating the uncertainty Type A (standard deviation) we tried to follow the recommendation ([2], p. 93) to use the real value of the uncertainty, rather than the pessimistic or optimistic ones. The difference between these three estimations is clearly seen from processing the results given in Fig. 3.

1. The pessimistic value of the uncertainty Type A was obtained by calculating the results of the measurements for the time interval equal to 1 min (6 measurements): $\bar{P}_0 = (55.923 \pm 0.053)$ W.
2. The optimistic value of the uncertainty Type A was obtained by calculating the results of the measurements for the total duration of the experiment (the time interval equals to 30 h, or 10.800 measurements): $\bar{P}_0 = (55.923 \pm 0.001)$ W.
3. The real value of the uncertainty Type A was obtained by calculating the results of the measurements

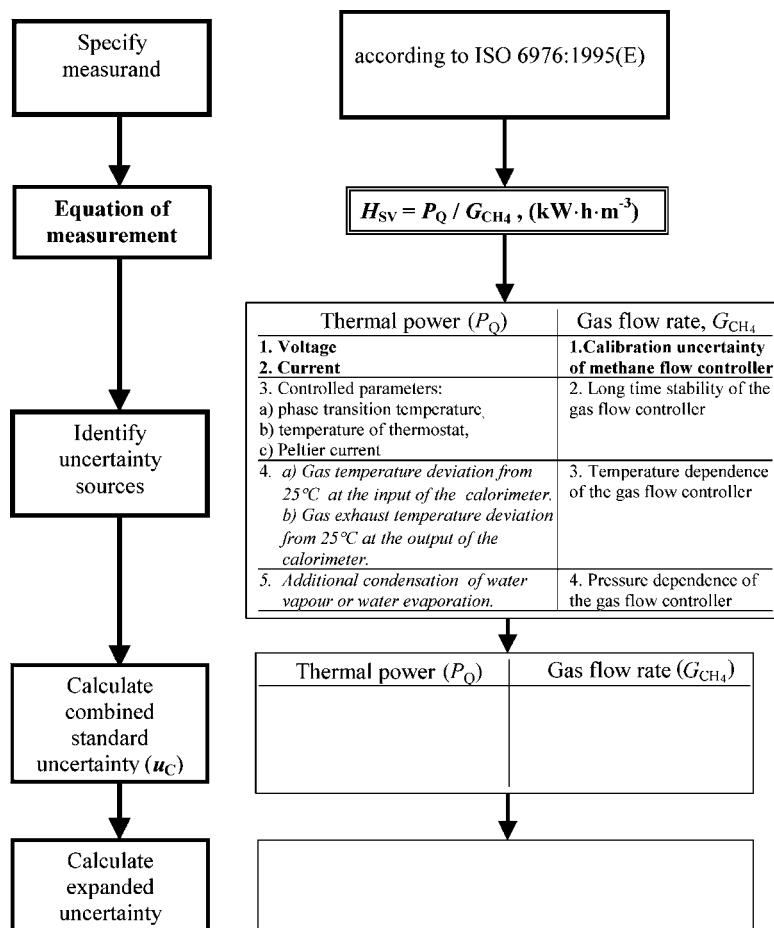


Fig. 2. The uncertainty estimation process.

for the time interval equal to 1 h (360 measurements): $\bar{P}_0 = (55.923 \pm 0.003)$ W.

The calculation of the combined relative standard uncertainty of the measurement of the thermal power during the operation of the calorimeter in the zero run is presented in Table 1. It follows from the table that the combined relative standard uncertainty is equal to 0.008% when measuring the thermal power over the whole working range of the calorimeter under the conditions of the zero run.

At this point a question arises: is the obtained estimation of the uncertainty sufficient to allow one to make a conclusion about the accuracy and quality of the measurement of the thermal power by using this

new measurement method and this new type of calorimeter?

4. Study of the accuracy of measurements of the thermal power

There is a statement ([2], p. 87) that the smaller the uncertainty is, the smaller the inaccuracy is. At the same time, it is noted in ([1], 3.3.2) that “an unrecognized systematic effect cannot be taken into account in the evaluation of the uncertainty of the result of a measurement but contributes to its error”. Thus, there are two quantities, uncertainty and error, which effect the accuracy of a measurement. However, the question

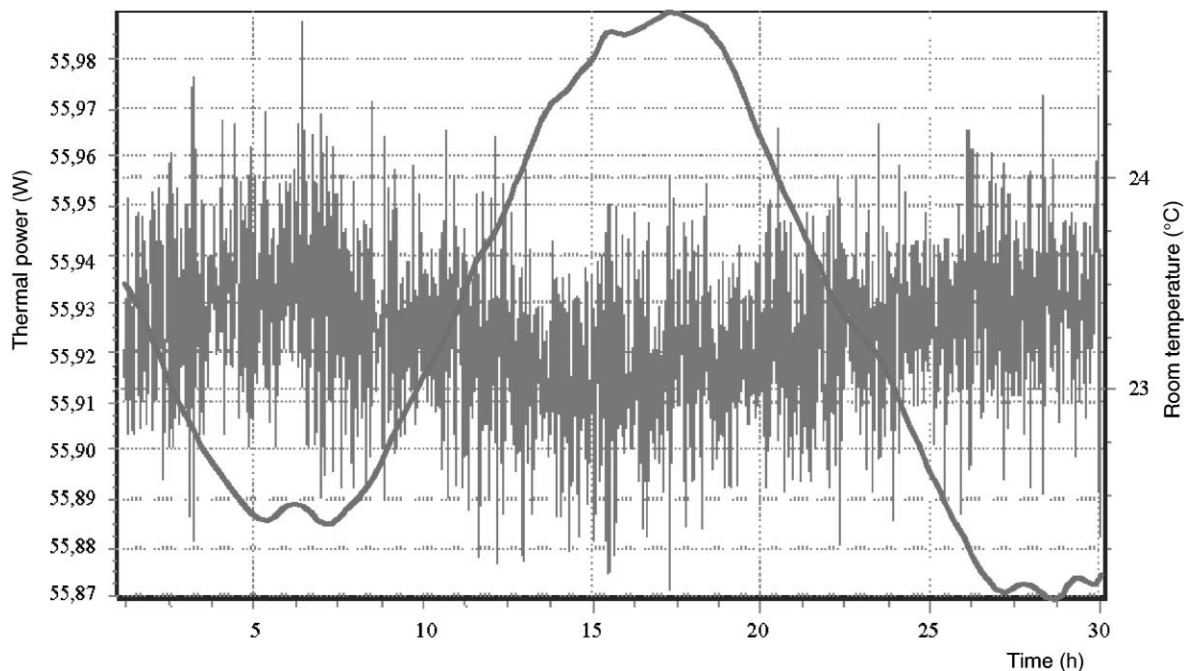


Fig. 3. Long-time stability of the base value.

Table 1

Uncertainty of the measurement of the thermal power during the zero run of the calorimeter

Name	Symbol/equation	Thermal power	
		$P_{\min} = 10 \text{ W}$	$P_{\max} = 75 \text{ W}$
1 Voltage at the heater (V)	U_H	11.25000	30.8000
2 Current (A)	I_H	0.89000	2.43000
3 Standard resistor (Ω)	R_3	0.10000	0.10000
4 Accuracy class of the resistor (%)	a	0.01	0.01
5 Uncertainty of the measurement of the voltage (V)	δ_U	$\pm 1.9 \times 10^{-4}$	$\pm 9.7 \times 10^{-4}$
6 Uncertainty Type B of the measurement of the voltage (V)	$U_B^U = \delta_U / \sqrt{3}$	$\pm 2.3 \times 10^{-6}$	$\pm 7.9 \times 10^{-6}$
7 Relative uncertainty of U_B^U (%)	$U_B^U = U_B^U / U_H$	1.1×10^{-4}	5.6×10^{-4}
8 Uncertainty Type B of the measurement of the resistance when determining the current (Ω)	$U_{B,R}^I = aR_3 / \sqrt{3}$	1.0×10^{-3}	1.8×10^{-3}
9 Uncertainty Type B of the measurement of the voltage when determining the current (V)	$U_{B,U}^I = \delta_I / \sqrt{3}$	5.8×10^{-6}	5.8×10^{-6}
10 Uncertainty Type B of the measurement of the current (A)	$U_B^I = \sqrt{(1/R)^2 (U_{B,U}^I)^2 + (U/R^2) (U_{B,R}^I)^2}$	1.3×10^{-6}	4.5×10^{-6}
11 Relative uncertainty of U_B^I (%)	$U_B^I = U_B^I / I_H$	5.16×10^{-5}	1.48×10^{-4}
12 Uncertainty Type B of the measurement of the thermal power (W)	$U_B^P = P \cdot \sqrt{(1/U)^2 (U_B^I)^2 + (1/T)^2 (U_B^I)^2}$	5.8×10^{-3}	6.1×10^{-3}
13 Relative uncertainty Type B of the measurement of the thermal power (%)	$U_B^P = U_B^P / P$	5.9×10^{-4}	4.8×10^{-3}
14 Relative standard uncertainty Type A (relative experimental standard deviation of the mean) (%)	$U_A^P = s(\bar{P})$	6×10^{-3}	5×10^{-3}
15 Combined relative standard uncertainty (%)	$u_C(P) = \sqrt{(U_A^P)^2 + (U_B^P)^2}$	8×10^{-3}	

whether these quantities are interrelated remains open.

In order to answer this question let us consider the condition given in ([2], p. 247) that is used for the determination of the existence of a significant difference between two results of the measurement of one and the same quantity:

$$|x_1 - x_2| \leq \beta u_0 \quad (3)$$

where β is a numeric factor in the range from 1 to 3, u_0 is the uncertainty which refers to the difference $|x_1 - x_2|$ that is found from the equation:

$$u_0^2 = u^2(x_1) + u^2(x_2) \quad (4)$$

If Eq. (3) is applied to the calibration of a device, then with taking into account that $u_N \ll u_G$, it takes the following form:

$$|x_N - x_G| \leq \beta u_G \quad (5)$$

where x_N is the conventional true value of the standard used and x_G is the value obtained for this standard from the calibration.

The difference presented in Eq. (5) is nothing but the error in full agreement with the definition of this quantity ([1], B.2.19).Eq. (5) is of great importance due to the following reasons:

1. In this equation, the concept of the notion ‘error’ as the difference between a conventional true value of the measurand and result of a measurement is expressed unambiguously.
2. The difference between uncertainty and error can be clearly seen.

3. At the same time here, it is presented the relation between these quantities.

Depending on the relation between these two quantities, the following cases may take place:

1. Eq. (5) is fulfilled;
2. The difference in the left part of Eq. (5) is greater than the right part.

In the first case, the accuracy of the result can be represented by the uncertainty of the measurement while in the second case, this uncertainty does not characterize the accuracy of the result of the measurement.

So, the estimation of the uncertainty of measurement is necessary, but not sufficient. The estimation of the uncertainty is insufficient in case measurements are carried out with a new technique or new devices. Due to the fact that in our case we have both of these factors, it was necessary to prove the accuracy of the measurements of the thermal power.

With this aim, the gas burner was changed by a second electric heater designated further as imitator. A set of measurements was carried out in which different (four) values of electric power were supplied to the imitator. The measurements of the thermal power liberated by the imitator have been performed by two ways: (1) the well-known method of measuring the voltage and current and (2) with the help of the heat pipe by using Eq. (2). The results of the measurements obtained by the first method were considered as conventional true values. Therefore, it was possible from

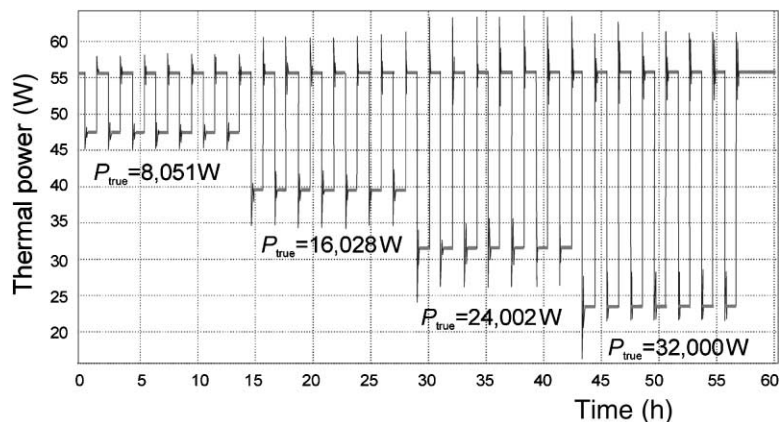


Fig. 4. Measurement of the thermal power of the compensation heater during the operation of the heat pipe with the imitator.

basing on Eq. (5) to find the error and hence, the accuracy of the measurement of the thermal power.

The measurements were carried out in the following way. At first, during the period of 1 h the base value of the calorimeter was measured (the zero run). Then, also during 1 h, a constant electric power was supplied to the imitator and again, during the period of 1 h, the base value was measured. This procedure was repeated seven times with each of the four values of the given electric power. Fig. 4 presents the corresponding measurements and Table 2 gives the results of these measurements. With the aim to obtain the actual

estimation of the uncertainty of the thermal power measurements, we have used the measurements carried out during the last 30 min for each period of time, i.e. 180 measurement points. Besides, the average value and its standard deviation have been calculated from the obtained seven results in each set of measurements.

Together with the results of the measurements and their average values, the table presents the mean of the base value for each set of the measurements of the imitator power (\overline{P}_0^n). The standard deviation of the average base value for all sets of measurements was found to be equal to 0.021%, which characterizes the

Table 2
Results of studying the accuracy of the measurement of the thermal power

	P_Q (W)	P_{true} (W)
P_1	8.044 ± 0.006	8.048 ± 0.000
P_2	8.045 ± 0.005	8.050 ± 0.000
P_3	8.048 ± 0.005	8.050 ± 0.000
P_4	8.051 ± 0.006	8.051 ± 0.000
P_5	8.048 ± 0.006	8.052 ± 0.000
P_6	8.052 ± 0.006	8.052 ± 0.000
P_7	8.049 ± 0.006	8.052 ± 0.000
\overline{P}	8.048 ± 0.003	8.051 ± 0.001
$\Delta P = \overline{P_{\text{true}}} - \overline{P_Q} = 0.003$ W (0.04%), $\overline{P}_0^1 = (55.949 \pm 0.003)$ W		
P_1	16.017 ± 0.009	16.030 ± 0.000
P_2	16.012 ± 0.009	16.027 ± 0.000
P_3	16.016 ± 0.005	16.026 ± 0.000
P_4	16.020 ± 0.006	16.026 ± 0.000
P_5	16.017 ± 0.005	16.027 ± 0.000
P_6	16.020 ± 0.005	16.029 ± 0.000
P_7	16.025 ± 0.005	16.031 ± 0.000
\overline{P}	16.018 ± 0.004	16.028 ± 0.002
$\Delta P = \overline{P_{\text{true}}} - \overline{P_Q} = 0.010$ W (0.06%), $\overline{P}_0^2 = (55.947 \pm 0.006)$ W		
P_1	23.983 ± 0.006	23.998 ± 0.000
P_2	23.986 ± 0.005	24.002 ± 0.000
P_3	23.989 ± 0.006	24.005 ± 0.000
P_4	23.989 ± 0.005	24.006 ± 0.000
P_5	23.991 ± 0.005	24.007 ± 0.000
P_6	23.983 ± 0.005	24.000 ± 0.000
P_7	23.982 ± 0.006	23.998 ± 0.000
\overline{P}	23.986 ± 0.004	24.002 ± 0.004
$\Delta P = \overline{P_{\text{true}}} - \overline{P_Q} = 0.016$ W (0.067%), $\overline{P}_0^3 = (55.965 \pm 0.009)$ W		
P_1	31.971 ± 0.006	31.996 ± 0.000
P_2	31.967 ± 0.006	31.995 ± 0.000
P_3	31.972 ± 0.005	31.996 ± 0.000
P_4	31.974 ± 0.005	31.999 ± 0.000
P_5	31.980 ± 0.005	32.000 ± 0.000
P_6	31.980 ± 0.006	32.006 ± 0.000
P_7	31.986 ± 0.005	32.008 ± 0.000
\overline{P}	31.976 ± 0.006	32.000 ± 0.005
$\Delta P = \overline{P_{\text{true}}} - \overline{P_Q} = 0.024$ W (0.075%), $\overline{P}_0^4 = (55.964 \pm 0.013)$ W		

long-time stability of the base value during the total time of the measurements.

The maximum relative standard deviation from the average value does not exceed 0.0373% and this value will be used for calculating the combined standard uncertainty. The estimation of the uncertainty in this case was similar to the above-considered procedure. The combined relative standard uncertainty of the measurement of the power in this set of measurements was found to be equal to 0.0375%. Thus, practically the whole uncertainty is determined by the standard deviation.

As it follows from Table 2, a systematic deviation from the conventional true value can be noted. This deviation depends on the value of the power supplied to the imitator, which permits one to conclude that the reason for this deviation is the heat loss through the wires feeding the imitator. Since the deviation does not exceed 0.1%, this value can be considered as the maximum absolute error when measuring a thermal power with the given technique.

5. Estimation of the uncertainty when measuring the heat of combustion of methane

At first, let us consider again the uncertainties of the third group. In order to decrease the thermal effect caused by the above-mentioned reasons the following measures were taken:

1. The temperature difference between the gases entering or leaving the calorimeter and the calorimeter was kept within 0.1 °C. Under these conditions, the resulting thermal effect was equal to or less than 0.01% of the total heat.
2. Unwanted condensation or evaporation of water was eliminated by adjusting the quantity of argon. For example, during the combustion of methane the quantity of argon was equal to that of methane.

Table 3 shows the working parameters with which the measurement of the heat of combustion of methane has been performed. As it can be seen from the presented data, the conditions given in Tables 1 and 2 have been completely fulfilled.

The results of the measurements of the heat of combustion of methane are presented in Table 4. After combining the results of these two runs of measurements the heat of combustion of methane was found to be $(11.062 \pm 0.002) \text{ kW h m}^{-3}$ or $(55.503 \pm 0.009) \text{ MJ kg}^{-1}$; the relative experimental standard deviation of the mean was 0.016%.

The estimation of the uncertainty of the above-presented results was performed with taking into account the uncertainty of the measurement of the thermal power (U_{B,P_0}). The results of this estimation are shown in Table 5. As it follows from the data presented in the table, the relative uncertainty of the methane flow controller introduces the main contribution into the combined relative standard uncertainty.

In this case, it was also possible to check the accuracy of the measurement of the heat of combustion of methane by using Eq. (3). The following values recommended by ISO 6976-96 were taken as (x_1, u_1) : $x_1 = 55.516 \text{ MJ kg}^{-1}$ and $u_1 = 0.028 \text{ MJ kg}^{-1}$ (which corresponds to 0.05%). The value of the heat of combustion of methane and its uncertainty that have been obtained in this study are $x_2 = 55.503 \text{ MJ kg}^{-1}$ and $u_2 = 0.022 \text{ MJ kg}^{-1}$. The difference $|x_1 - x_2|$ is equal to 0.013 MJ kg^{-1} with the acceptable value (βu_0) equal to 0.036 MJ kg^{-1} ($\beta = 1$).

The absence of significant discrepancies proves our suggestions stated above that the noted systematic deviation when measuring the thermal power was caused by a heat loss at the imitator.

The value of the heat of combustion of methane recommended by ISO 6976-96 is the result of precision measurements carried out by using one and the same technique of Rossini. The absence of a significant

Table 3
Working parameters for the combustion of methane

Thermal insulation of the heat pipe		Gas supplied to the burner (l h^{-1})			I_p (mA)	t_{control} (°C)	t_{input} (°C)	t_{output} (°C)	
Vacuum jacket	Water jacket	O ₂ ^a (prim)	O ₂ ^a (sec)	Ar	CH ₄	1508.03 ± 0.03	24.800 ± 0.001	24.960 ± 0.002	24.972 ± 0.002
+	+	1.4	6.3	2.5	2.5				

^a Saturated with water at 25 °C.

Table 4

Measurement of the heat of combustion of methane at a methane flow rate of $4.9844 \times 10^{-4} \text{ g s}^{-1}$ ($= 2.5009 \times 10^{-3} \text{ m}^3 \text{ h}^{-1}$)

	P_0 (W)	P_{comp} (W)	P_Q (W)	Heat of combustion H_S	
				(kW h m ⁻³)	(MJ kg ⁻¹)
1st Run	55.441 ± 0.005	27.864 ± 0.019	27.577 ± 0.020	*	*
	55.434 ± 0.005	27.793 ± 0.009	27.641 ± 0.010	11.052	55.455
	55.448 ± 0.005	27.781 ± 0.009	27.667 ± 0.010	11.063	55.507
	55.454 ± 0.005	27.775 ± 0.010	27.679 ± 0.011	11.068	55.531
	55.475 ± 0.005	27.811 ± 0.009	27.664 ± 0.010	11.062	55.501
	\overline{H}_S^1			11.061 ± 0.003	55.498 ± 0.016
2nd Run	56.214 ± 0.005	28.548 ± 0.017	27.666 ± 0.018	11.062	55.505
	56.144 ± 0.005	28.552 ± 0.011	27.592 ± 0.012	*	*
	56.069 ± 0.007	28.392 ± 0.010	27.677 ± 0.012	11.067	55.527
	56.045 ± 0.007	28.372 ± 0.014	27.673 ± 0.016	11.065	55.519
	56.054 ± 0.005	28.401 ± 0.012	27.653 ± 0.013	11.057	55.479
		\overline{H}_S^2			11.063 ± 0.002

* This result could not be used for the calculation of the average value in accordance with the testing by the Fischer criterion.

Table 5

Uncertainty of the measurement of the specific heat of combustion of methane

No.	Name	Symbol/equation	Value
1	Basic value (W)	P_0	55.45
2	Compensation thermal power (W)	P_{comp}	27.80
3	Thermal power of the burner (W)	$P_Q = P_0 - P_{\text{comp}}$	27.65
4	Uncertainty Type B of the measurement of P_Q (W)	$U_{B,P_Q} = U_B^P$	
5	Relative uncertainty Type B of the measurement of the thermal power (%)	$U_{B,P_Q} = U_B^P$	6×10^{-3}
6	Flow rate of methane ($\text{g s}^{-1} \times 10^{-4}$)	G_m	4.9844
7	Calibration uncertainty of the methane flow controller (%)	$\delta(U_{B,G})$	± 0.06
8	Relative uncertainty of the methane flow controller (%)	$U_{B,G} = \delta U_{B,G} / \sqrt{3}$	0.035
9	Heat of combustion of methane (MJ kg ⁻¹)	$H_{S,m} = P_Q / G_m$	55.503
10	Relative uncertainty Type B (%)	$U_{B,H_{S,v}} = \sqrt{U_{B,P_Q}^2 + U_{B,G}^2}$	0.036
11	Relative standard uncertainty Type A (%)	$U_A = s(H_{S,v})$	0.016
12	Combined relative standard uncertainty (%)	$u_C = \sqrt{U_{B,H_{S,v}}^2 + U_A^2}$	0.039

deviation that has been shown above proves the correctness and validity of both the presented new method and new gas calorimeter.

6. Conclusions

The above-considered investigations allow us to make the following conclusions.

1. The proposed new calorimetric method permits performing direct measurements of heats of combustion of gases in accordance with the definition of this quantity.
2. A design of a gas calorimeter has been developed that allows performing absolute measurements, i.e. measurements without electrical calibration or without corresponding pure gases or gas mixtures for this purpose.
3. The established uncertainty of the measurement when using this calorimeter is as follows:
 - the relative standard uncertainty Type A of the base value in the zero run of the calorimeter is equal to or less than 0.02%;
 - the relative standard uncertainty Type A of the heat of combustion of gas is equal to or less than 0.02%;

- the combined relative standard uncertainty of the heat of combustion of gas is equal to approximately 0.04% for the specific heat of combustion and 0.1% for the volumetric heat of combustion.
4. The further increase of the accuracy of the measurement of the heat of combustion depends first of all on the increase of the accuracy of the measurement of the quantity of gas being burnt rather than an increase of the accuracy of the measurement of thermal effects.
 5. The proposed gas calorimeter can be used not only as a prototype of a standard gas calorimeter but it can be also recommended for the application in the gas industry for measuring the heats of combustion of different types of fuel gases.

And finally, the conclusion can be made that the estimation of the uncertainty is most probably an art rather than an established routine procedure.

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