THE REFERENCE CALORIMETER SYSTEM FOR METROLOGICAL ASSURANCE OF COMBUSTION ENERGY MEASUREMENTS

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Recently the Calorimetric Laboratory of D.I. Mendeleyev Institute for Metrology (VNIIM) has been focusing on improving the national system of metrological assurance of combustion energy measurements of gaseous fuels (gas calorimetry), solid and liquid fuels (bomb calorimetry).

The State Primary Standard of the units of combustion energy, specific combustion energy and volumetric combustion energy «GET 16-2010» makes the basis for ensuring the uniformity of measurements and providing traceability in the most important industrial fields of the country – fuel and energy complex, petrochemical, coal, metal and chemical industries.

D.I. Mendeleyev Institute for Metrology has been improving the State Primary Standard in the area of combustion calorimetry «GET 16-2010» since 2015 (last time it was improved in 2010). This work is performed in accordance with the State Government Assignment and is directed to expand the measurement range from 50 to up to 90 MJ/m^3 and decrease the lower range from 10 to 3 MJ/m^3.

Finally it will allow to develop the metrological assurance for precision measurements of calorific value of associated petroleum gas (APG), natural gas (NG), low-calorific gases (LCG): coke gas, blast-furnace gas, biogas and its mixes using modern calorimeters and chromatographs.

The reference calorimeter system (fig. 1) has been assembled, launched and researched by specialists of the Calorimetric Laboratory of VNIIM. The calorimeter system includes 2 reference calorimeters, which implement a direct calorimetric method of measurements of inferior heat of combustion: in the range of 25 to 90 MJ/m^3 – typical for the different types of NG and APG (the calorimeter for APG – CAPG), in the range of 3 to 25 MJ/m^3 – typical for the different types of LCG (the calorimeter for LCG – CLCG).

Figure 1 – The Reference Calorimeter System (CAPG / CLCG)

The calorimeter system is intended for long-term continuous measurement. Scheme and work principles (fig. 2) are based on a comparison of the gas calorific value with the velocity of its feed rate. The calorimeter contains a gas burner and an electrical heater inside the thermal unit. Heat balance between these parts is constantly maintained during combustion process (compensation method).

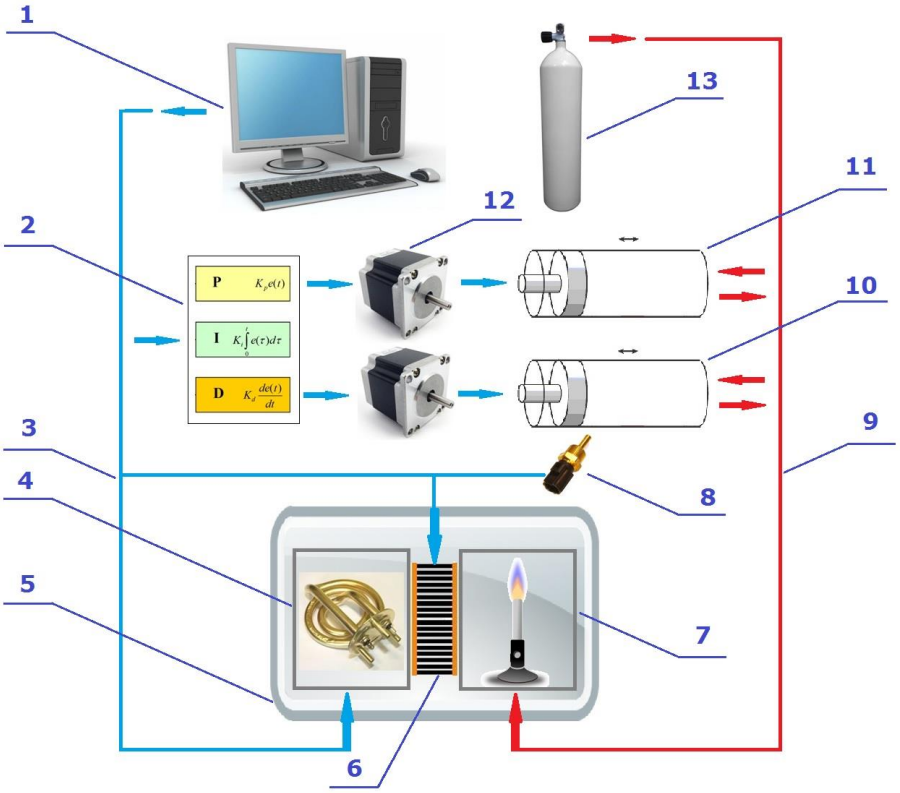

Figure 2 – Scheme of CAPG / CLCG:
1 – control and regulation unit (embedded computer); 2 – PID-regulation unit; 3 – electronic communication line; 4 – comparative cell (with electric heater inside); 5 – thermal unit with air thermostat; 6 – heat flow sensor; 7 – measuring cell (with gas burner inside); 8 – pressure sensor system (absolute and gauge); 9 – gas pipeline; 10, 11 – cylinders, forcers; 12 – step engines; 13 – gas bottle

The CLCG contains additional gas mixing system for combustion of gases with low calorific value: gases in the range of (3 – 10) MJ/m^3) are diluted by pure methane in a volume ratio of 3/1 (60% of LCG and 30% of methane) for continuous and steady burning.

Calibration of the calorimeters is performed using high-purity gases – methane, ethane and propane, hydrogen, hydrogen-helium mix.

Interlaboratory comparisons have been performed using a NG imitator and 2 other types of reference gas calorimeters: the «KATET» – heat pipe based calorimeter, the «B-06АК» – calorimeter-comparator. Obtained measurement results confirm the declared accuracy (see table 1).

Accuracy of the CAPG has also been researched by means of testing various imitators of APG (see table 2), and estimated by a limit of 0,3%. Accuracy of the CLPG is currently being researched and approximately estimated at 0,5%.

Table 1 – Interlaboratory comparison results

CAPG / CLCG	«KATET» heat pipe based calorimeter	«B-06AK» calorimeter-comparator
Developed / approved as a standard at:		
by the end of 2017	2010	2010
Measurement range, MJ/m^3		
3 ÷ 90	10 ÷ 50	25 ÷ 50
Expanded relative uncertainty, %		
0,30 ÷ 0,50	0,14	0,20
Measuring results of NG imitator (H^{inf}_{ref} = 32,05 MJ/m^3, according to ISO 6976:1995)		
32,06 (CAPG)	32,06	32,08

Table 2 – Measurement results in APG range (calibrated within ethane & propane)

	H^{inf}_{ref}, MJ/m^3	$H^{inf}_{measured}$, MJ/m^3	($H^{inf}_{measured}$-H^{inf}_{ref})/ H^{inf}_{ref}, %
APG imitator № 1 (methane – 46,37, ethane – 40,35, propane – 10,13, n–butane – 2,55, n–pentane – 0,6 mol. %)	51,97	51,91	-0,12
APG imitator № 2 (methane – 25,55, ethane – 54,57, propane – 10,13, n–butane – 3,96 mol. %)	59,28	59,34	0,10
APG imitator № 3 (methane – 1,57, ethane – 75,00, propane – 18,72, butane – 4,69 mol. %)	66,82	66,84	0,02
APG imitator № 4 (methane – 11,43, ethane – 55,0, propane – 20,0, butane – 8,0, pentane – 4,05, hexane – 1,5, nitrogen – 0,01, carbon dioxide – 0,01 mol. %)	71,03	70,98	-0,08
Note: calculation results are obtained according to ISO 6976:1995. All of the gases are prepared by a gravimetric method. Its component composition is exactly known			

The technical task to implement the possibility of burning various gases in a range of (3 – 90) MJ/m^3 with a relative accuracy of less than 0,5% has been successfully completed.

Now the priority is to create the certified standards of gas mixtures with different calorific values. That will be used for calibration and verification of industrial gas calorimeters.

The production of the reference calorimeter system is being carried out by the domestic scientific enterprise – JSC «Teplofizicheskie pribory (Thermophysical equipment)» (Russia, Saint-Petersburg).