

STATE STANDARDS

STATE PRIMARY STANDARD OF TEMPERATURE UNIT IN THE RANGE 0–3200°C GET 34-2020: PRACTICAL IMPLEMENTATION OF THE NEW DEFINITION OF KELVIN

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We consider the necessity and means of modernizing the State primary standard GET 34-2007 of the unit of temperature in the range of 0–3000°C. The problem of transition to a new definition of kelvin is highlighted. It is shown that the new definition of kelvin does not directly affect the status of the current international temperature scales ITS-90 and PLTS-2000, however, it provides significant advantages for measuring thermodynamic temperatures below 20 K and above about 1300 K. In this regard, the main direction of modernization of GET 34- 2007 in the range of 273.15–1235 K is associated with the improvement of methods and means of implementing the ITS-90. As part of the work on the modernization of the standard in the range above 1235 K, a complex of equipment was created that allows reproducing the kelvin in accordance with its new definition by two methods: absolute primary radiometric thermometry and relative primary radiometric thermometry. The methods are recommended by the Thermometry Advisory Committee of the International Bureau of Weights and Measures. The basic principles of the implementation of these methods, as well as the composition and metrological characteristics of the GET 34-2020 approved in 2020, are outlined. The results of key comparisons of GET 34-2020 in the range of 273.16–692.477 K are presented, as well as the results of temperature measurements of a number of high-temperature reference points and their comparison with the published results of similar measurements by leading national metrological institutes.

Keywords: international temperature scale, thermodynamic temperature, comparator, energy brightness, spectral band, integrating sphere, trap detector, monochromator.

Introduction. At the 26th General Conference on Weights and Measures, held on November 13–16, 2018 in Paris, an updated International System of Units was adopted. It provides for new definitions of the four basic units (including the kelvin) of physical quantities based on fundamental physical constants. The new definition of kelvin, introduced into practice by the decision of the General Conference on Weights and Measures in May 2019, has the following formulation [1]: “Kelvin, symbol K, is a unit of thermodynamic temperature. It is determined by adopting a fixed numerical value of the Boltzmann constant k , which should be $1.380649 \cdot 10^{-23}$ and which is expressed in units of $\text{J} \cdot \text{K}^{-1}$, which is equivalent to $\text{kg} \cdot \text{m}^2 \cdot \text{s}^{-2} \cdot \text{K}^{-1}$, where kilogram, meter and second are defined in terms of h , c and $\Delta\nu_{\text{Cs}}$.”

Here h is Planck’s constant; c is the speed of light in vacuum; $\Delta\nu_{\text{Cs}}$ is the cesium frequency corresponding to the transition between two hyperfine levels of the unperturbed ground state of the ^{133}Cs atom. This definition incorporates the exact value of the Boltzmann constant to be $k = 1.380649 \cdot 10^{-23} \text{ J} \cdot \text{K}^{-1}$.

The following arguments were put forward in favor of the new definition of the unit of temperature [2]:

– the new definition of the kelvin makes it independent of any material substance, technical implementation, as well as temperature or temperature range. In particular, the new definition will improve temperature measurements in a range far from the triple point of water;

- establishing the exact value of the Boltzmann constant k is important not only for metrology, but also for science in general. In this case, the error in measuring the thermodynamic temperature will not be significant;
- the new definition of kelvin through the Boltzmann constant does not require replacing the ITS-90 with a more perfect temperature scale, but it also does not prevent such a replacement;
- in the long term, it is possible to gradually improve the temperature scale to reduce uncertainty and expand temperature ranges without the expensive costs and inconvenience that arose when changing previous scales.

The consequence of the redefinition of the kelvin is the need to modernize the domestic state standards of the unit of temperature in order to ensure the reproduction of the kelvin in accordance with its new definition.

According to the estimates of the TG-SI Working Group of the BIPM Advisory Committee on Thermometry, the new definition of kelvin does not directly affect the status of the current international temperature scales ITS-90 and PLTS-2000 and allows measurement of thermodynamic temperatures less than 20 K and more than about 1300 K – in areas where primary thermometers can provide uncertainty less than the uncertainty of ITS-90 [2]. Taking into account this assessment of the TG-SI working group, during the modernization of the State primary standard for the unit of temperature in the range of 0–3000°C GET 34-2007, the main attention is given to measuring temperatures over 1235 K using primary thermometry methods. In the range of 273.15–1235 K, no fundamental changes in the composition of GET 34-2007 are required, since in the foreseeable future the current ITS-90 will retain its status [2].

At present, the requirements of domestic science and industry for the accuracy and expansion of the range of temperature measurements are increasing. Analysis of the metrological characteristics of the most accurate working measuring instruments (MI), produced in the Russian Federation and abroad, showed that over the past 15–20 years, the accuracy of the MI temperature registered in the State Register of MI increased by 1.5–2 times. The development of critical technologies, in particular, technologies for creating materials for hypersonic aircraft, implies the provision of the possibility of measuring temperatures well above 3000°C.

Thus, during the modernization of GET 34-2007, special attention was paid to ensuring the possibility of reproducing kelvin in accordance with its new definition in the range of 1235–3473 K, and the problem of improving the accuracy of the standard in the range of 273.15–1235 K was also addressed.

Composition and description of the GET 34-2020 equipment complex, which realizes the reproduction of the temperature unit in the range of 273.15–1235 K. The composition of the standard in the range 273.15–961.78 K is determined by the regulation on the ITS-90 [3]. To reproduce ITS-90 in the range of 273.15–1235 K, GET 34-2020 includes the following main devices:

- three reference platinum resistance thermometers RRT-25, for the temperature range 273.15–933.473 K;
- three high-temperature reference platinum resistance thermometers (HRT) for the temperature range 933.473–1234.93 K;
- a set of equipment for reproducing reference points of the temperature scale MTS-90 in the range of 933.473–1234.93 K, containing furnaces PRT 50-700, PRT 700-1100 and ampoules with pure substances – water, gallium, indium, tin, zinc, aluminum, and silver;
- a set of equipment for measuring the resistance of reference thermometers: an F900 resistance comparator and a set of thermostated reference resistance measures MC3050T.

GET 34-2020 also includes the following auxiliary devices:

- PRT 50-700, an oven for annealing thermometers in the range of 50–700°C;
- PRT 700-1100, an oven for annealing thermometers in the range of 700–1100°C;
- a calibrator for bridges used to measure resistance.

In Fig. 1 we show a block diagram of a modernized complex of equipment from the GET 34-2020, intended for reproducing the ITS-90 in the range of 273.15–1235 K.

In the process of improving the apparatus configuration of the standard GET 34-2007, which reproduces the unit of temperature in the range of 0–961.78°C, the following tasks were resolved:

- the random component of the error in measuring the resistance of reference thermometers was reduced by using the cumulative average operator, which made it possible to significantly increase the ratio of the useful signal to noise;

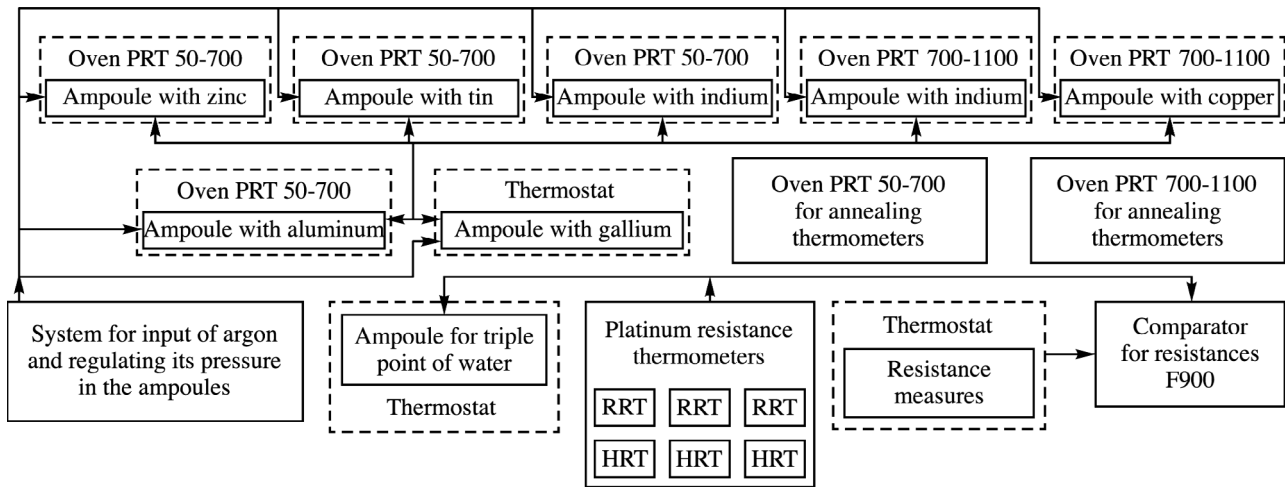


Fig. 1. Structure and block diagram of GET 34-2020 that realizes the International temperature scale in the range 273.16–961.78 K; *RRT*, *HRT* – reference platinum resistance thermometer and high-temperature reference platinum resistance thermometer, respectively.

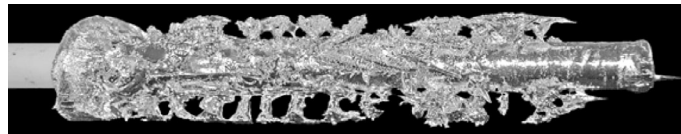


Fig. 2. Structure of the interface between the solid and liquid phases for Indium after its initiation.

– a method for assessing the effect of impurities on the temperature of phase transitions of metals used to implement the reference points of the temperature scale of the ITS-90 [4, 5] was investigated. The method for assessing the effect of impurities on the temperature of phase transitions of metals (indium and zinc) assumes uniform formation of a layer of solidifying metal and movement of the interface between the solid and liquid phases of the metal from the outer surface of the ingot to its center. The results of studying the processes of solidification of indium and zinc [6] showed that the actual crystallization process may differ from the model used to calculate the uncertainties caused by impurities in the metal. The crystallization process depends on the speed and method of initiation of the onset of crystallization, as well as on a number of other factors. As a result, the phase interface can be discontinuous and move uniformly from the outer surface along the vertical of the ingot to the center (Fig. 2). According to the research results, it was found that the method stated in the document [4] can be successfully used if, during the crystallization of the metal, the formation of the phase separation is uniform along the ingot and the phase boundary moves uniformly from the outer surface of the ingot to its center;

– the inhomogeneity of the temperature field in the working volume has been significantly reduced as a result of the use of the developed furnace designs. The inhomogeneity of the temperature field in the working volume of the furnace is one of the main factors that determine the error in the implementation of the reference point of the temperature scale and the result of thermometer calibration at the reference points. Field inhomogeneity depends on the design of the furnace, and auxiliary devices, which affect the configuration of the temperature fields around the ampoule with the metal and along the immersed part of the reference thermometer.

Based on the calculation of thermophysical models and experimental studies of NPP Etalon (Omsk) together with the Mendeleev VNIIM the furnaces PRT 50-700 were, providing the possibility of forming in their working space the temperature fields, necessary to reduce the uncertainty of the reproduction of the ITS-90 reference points and the uncertainty of the calibration of reference thermometers.

In Fig. 3 we show the possibilities for changing the configuration of the temperature field in the working oven volume using the temperature regulators. The configuration of the temperature field in the ampoule was estimated by measuring the solidification temperature of zinc at different distances from the sensitive element of the thermometer to the bottom of the

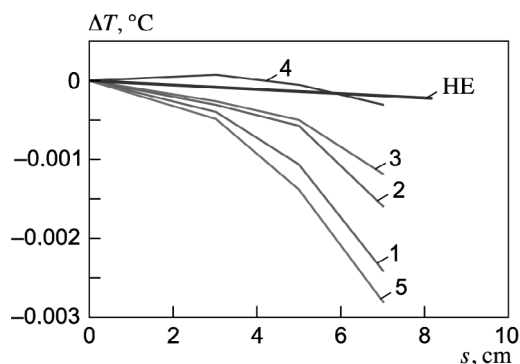


Fig. 3. Investigation of the possibilities of regulating the temperature field in the working volume of the furnace with five realizations of the phase transition of zinc: the temperature field along the height of the ampoule channel with the temperature drop settings on the lower and upper guard heaters -2.0 and $+4.0^{\circ}\text{C}$ (1); -2.2 and $+4.4^{\circ}\text{C}$ (2); -2.4 and $+5.2^{\circ}\text{C}$ (3); -2.6 and $+5.5^{\circ}\text{C}$ (4); -2.6 and $+6.2^{\circ}\text{C}$ (5); HE – hydrostatic effect; s is the distance from the sensitive element of the thermometer to the bottom of the ampoule.

ampoule. Five realizations of phase transitions (solidification) were carried out in a PRT 50-700 furnace, which has three controlled heaters, with five different settings of the upper and lower guard heaters. The temperature field was measured along the height of the ampoule channel with the temperature drop settings on the lower and upper heaters -2.0 and $+4.0^{\circ}\text{C}$; -2.2 and $+4.4^{\circ}\text{C}$; -2.4 and $+5.2^{\circ}\text{C}$; -2.6 and $+5.5^{\circ}\text{C}$; -2.6 and $+6.2^{\circ}\text{C}$, respectively. To illustrate the capabilities of the furnace, Fig. 3 shows the theoretical dependence of the solidification temperature of zinc on the hydrostatic pressure of the metal column – the hydrostatic effect.

Reproduction of the unit of temperature in the range 1235–3473 K. In the range above the solidification temperature of silver (1235 K), the Advisory Committee on Thermometry recommended two methods of measuring thermodynamic temperature that can be used to reproduce the unit of temperature in accordance with its new definition [1]: absolute primary radiometric thermometry based on measuring the power of thermal radiation; relative primary radiometric thermometry, based on high-temperature datum points.

Realization of a direct method for measuring thermodynamic temperature. The direct method is based on Planck's law, which establishes a relationship between the thermodynamic temperature T and the spectral density of the radiance of thermal radiation $L(\lambda, T)$ at a wavelength λ :

$$L(\lambda, T) = \frac{c_1}{\pi\lambda^5 n^2} \left[\exp\left(\frac{c_2}{\lambda T n}\right) - 1 \right]^{-1},$$

where $c_1 = 2hc^2 = 1.91042972 \cdot 10^{-16} \text{ V}\cdot\text{m}^2$; $c_2 = hc/k = 0.014387768 \text{ m}\cdot\text{K}$ are the first and second radiation constants; n is the refractive index of air.

To measure the energy brightness, a trap detector (TD) is used, whose transfer function has the form

$$I_{\text{TD}} = K \int_0^{\infty} S(\lambda) L(\lambda, T) d\lambda,$$

where K is a coefficient that takes into account all optical, geometric, electrical and other factors affecting the measured photocurrent; $S(\lambda)$ is the spectral sensitivity of the TD to the heat flow power.

Thus, the radiance can be determined based on the results of measurements of the TD current and its spectral sensitivity to brightness. Figure 4 shows a block diagram for measuring the spectral sensitivity of the TD, which is determined from the measured values of the current initiated laser radiation. The power of the laser radiation is measured with an absolute cryogenic radiometer (ACR) Cryorad III (L-1 Standards and Technology, Inc., USA) at laser position I (cf. Fig. 4).

As a source of laser radiation, a highly stable continuous laser (HSCL) Supercontinuum SuperK EVO-04 (NKT Photonics Inc, USA) is used together with an acousto-optical spectral filter (AOSF) SuperK VARIA (NKT Photonics Inc, USA) with adjustable power, as well as with tunable wavelengths and the width of the spectral emission band. The laser

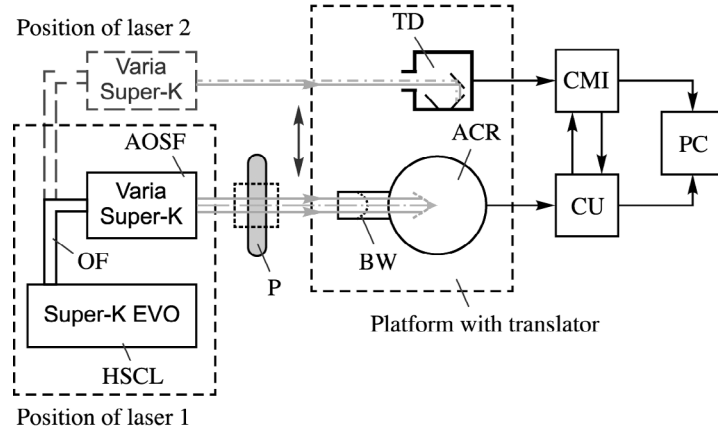


Fig. 4. Scheme for the spectral sensitivity measuring of trap-detector for laser positions 1 and 2: 1 – measurement of the radiation power of the Super-K EVO laser using an acousto-optical filter; 2 – measuring the spectral sensitivity of trap-detector to the power of the incident radiation; ACR – absolute cryogenic radiometer; CMI – cluster of measuring instruments; CU – control unit; HSCL – high stability continuous laser; BW – Brewster window; OF – optical fiber; AOSF – acousto-optical spectral filter; P – polarizer; PC – personal computer; TD – trap-detector.

radiation passes through the radiation polarizer P and the Brewster window BW to the sensitive area of ACR, after which the value of the power of the incident laser radiation is measured. Next, the platform with the translator is moved to position 2, after which the HSCL laser is sighted to the center of the TD input diaphragm. Then the TD is calibrated according to the spectral sensitivity to the radiation flux, expressed in units of measurement [A/W].

The measurement of the thermodynamic temperature of an absolutely blackbody (BB) by a direct method is realized by comparing the spectral energy brightness of the investigated BB and a special radiation source. The special source is an integrating sphere, whose inlet is connected to the outlet of the acousto-optic tunnel filter. The integrating sphere forms a flow at the outlet close to Lambert radiation.

To determine the energy brightness of the sphere radiation, the current of the TD, illuminated by the radiation of the integrating sphere in a certain solid angle, is measured. To form a known solid angle and further determine the geometric factor, precision diaphragms with diameters of 4 and 8 mm are used, which are installed on the input aperture of the trap detector and the front plane of the housing of the integrating sphere, respectively. To minimize stray illumination, a special hood is installed between the integrating sphere and the TD.

After adjusting the “integrating sphere – TD” system, the geometric factor g is calculated using the formula

$$g = 2\pi r_1 r_2 \left[r_1^2 + r_2^2 + d^2 + \sqrt{(r_1^2 + r_2^2 + d^2)^2 - 4r_1^2 r_2^2} \right]^{-1},$$

where r_1 and r_2 are the radii of the diaphragms installed, respectively, on the integrating sphere and TD; d is the distance between the diaphragms.

The circuit for measuring the TD photocurrent is shown in Fig. 5. The photo current is caused by the luminous flux in the solid angle determined by two precision diaphragms d_1 , d_2 at the outlet of the integrating sphere and at the input of the TD, respectively. In turn, an optical interference filter OIF with known spectral characteristics ($\lambda_0 = 650$ nm, full width of the half maximum is 20 nm) is installed on the TD. To eliminate the influence of the dark current, the photocurrent I_{TD} of the trap detector illuminated by the radiation of the integrating sphere is measured, and then the photocurrent is measured with the laser off $I_{n,1}$. The difference between these measured values is taken as a useful signal $I = I_{TD} - I_{n,1}$.

Using the iterative Newton–Raphson algorithm, the thermodynamic temperature corresponding to the brightness is calculated

$$T_{i+1} = T_i + \left\{ \left[I - K \int_0^\infty S(\lambda) L(\lambda, T) d\lambda \right] \left[\frac{c_2}{T_i^2} K \right]^{-1} \int_0^\infty S(\lambda) L(\lambda, T) \left\{ n\lambda \left[1 - \exp\left(-\frac{c_2}{n\lambda T_i}\right) \right] \right\}^{-1} d\lambda \right\}.$$

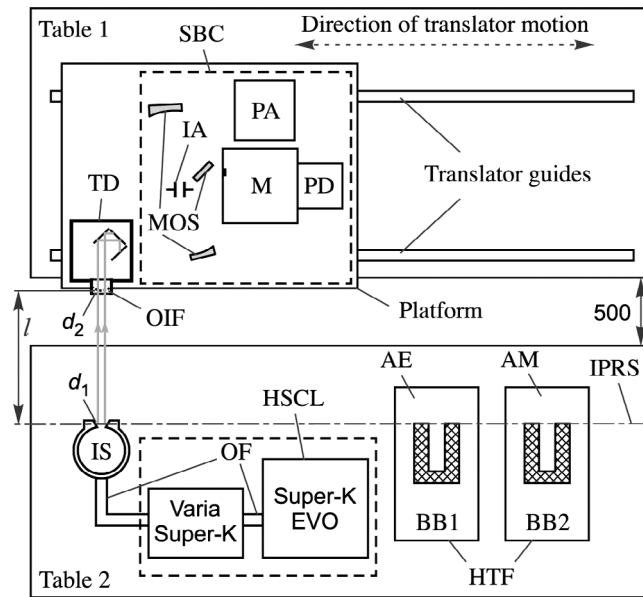


Fig. 5. Measurement of the trap-detector photocurrent that is caused by the radiation flux limited by solid angle determined by diaphragms at exit of an integrating sphere and at entrance of trap-detector: AM – ampoule with metal; AE – ampoule with eutectic; BB1, BB2 – blackbody’s; HTF – high temperature furnace; MOS – mirror optical system; IS – integrating sphere; SBC – spectral brightness comparator; M – monochromator; OIF – optical interference filter; PA – pico-ammeter; IA – intermediate aperture; IPRS – installation plane of radiation source; PD – photodiode; d_1 , d_2 – aperture; the other symbols are the same as in Fig. 4.

According to the calculated value of the thermodynamic temperature corresponding to the spectral energy brightness of the integrating sphere, the thermodynamic temperature of the blackbody emitter is measured, the measurement scheme is shown in Fig. 6. Measurements are performed by comparing the spectral radiance of the blackbody and the integrating sphere [9]. For this, the mirror optical system MOS of the spectral brightness comparator SBC, which is part of the standard, is sighted at the input diaphragm of the IS integrating sphere, and in platform position 1; the picoammeter PA measures the photocurrent initiated by the IS radiation. Next, the platform is translated into position 2, MOS is sighted at the inlet diaphragm of the ampoule with the eutectic metal alloy AE or ampoules with pure metal AM, located in the high-temperature furnace HTF models BB1, BB2. The radiation of the measured ampoule is focused by the MOS on the entrance slit of the monochromator M, then falls on the sensitive area of the photodiode PD, and the photocurrent is measured with the PA.

Taking into account the results of measurements of the comparator photodiode currents, the thermodynamic temperature of the investigated blackbody radiator is calculated:

$$T_{BB} = c_2 \left\{ \lambda \ln \left(K_i^{\text{rel}} \frac{i_{\text{sph}}}{i_{\text{BB}}} \right) \left[\exp \left(\frac{c_2}{\lambda T_{\text{sph}}} \right) - 1 \right] + 1 \right\}^{-1},$$

where $\lambda = (\lambda_1 + \lambda_2)/2$ and λ_1, λ_2 are, respectively, the upper and lower transmission limits of the monochromator of the spectral brightness comparator; K_i^{rel} – coefficient taking into account the difference between the spectral distributions of the radiation power of the integrating sphere and the blackbody at a given value of the thermodynamic temperature; $i_{\text{sph}}, i_{\text{BB}}$ – photodiode currents initiated by the radiation of the integrating sphere and the blackbody model, respectively; T_{sph} is the temperature corresponding to the brightness of the radiation of the integrating sphere.

Implementation of the method of relative primary thermometry. The method of relative primary thermometry is based on the use of high-temperature reference points, which are used as the temperatures of phase transitions of eutectic alloys metal–carbon (Me–C) [10, 11]. The values of the thermodynamic temperature of phase transitions, i.e., reference points, and their uncertainties should be determined by a direct method. Research carried out in leading national metrological institutes, including Mendeleev VNIIM, showed that reproducibility of high temperature fiducials can be high, almost comparable to the reproducibility of fiducials based on pure metals.

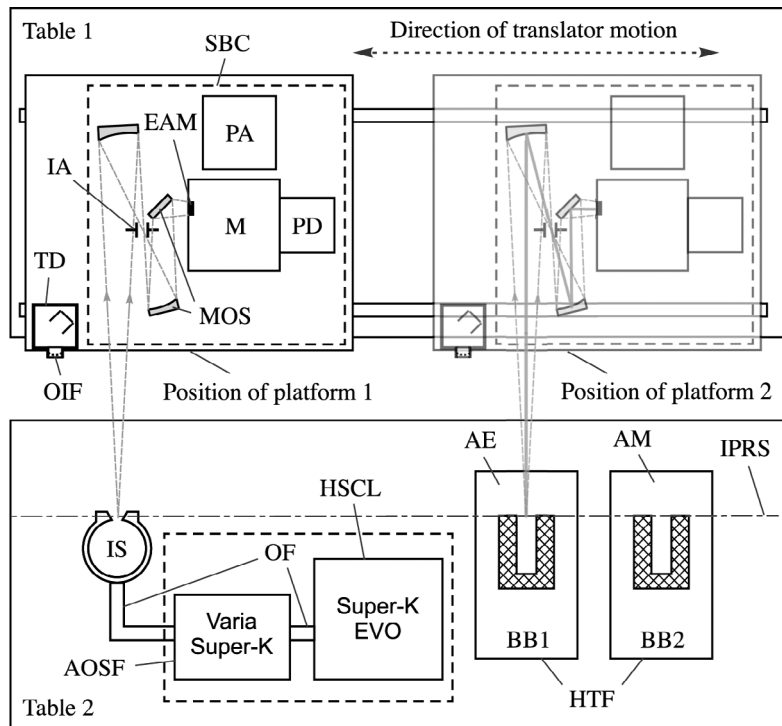


Fig. 6. Circuit for measuring the comparator photodiode current initiated by the integrating sphere radiation and by the blackbody radiation: EAM – entrance aperture of the monochromator; the other symbols are the same as in Figs. 4, 5.

The standard GET 34-2020 includes a set of equipment for implementing the method of relative primary thermometry, consisting of the following devices.

1. Two high-temperature emitters, whose cavity include cells in the form of blackbody models. The cells are filled with Me–C eutectics; a radiating cavity with a diaphragm is located in the center of the cell. The standard includes cells with pure metals (silver and copper), as well as cells with high-temperature eutectic alloys cobalt–carbon (Co–C), rhenium–carbon (Re–C), and tungsten carbide–carbon (WC–C).

2. A comparator of spectral brightness of thermal emitters, containing a double monochromator, a mirror focusing optical system, a silicon photodetector and a positioning system of emitters.

3. Temperature lamps used to transmit a unit of temperature and highly stable current sources to power lamps.

The thermodynamic temperature T_{BB} of the emitter of the blackbody BB in the method of conditional primary thermometry is calculated by the formula

$$T_{BB} = c_2 \left\{ \lambda \ln \left(\frac{I_{rp}}{I_{meas}} \right) \left[\exp \left(\frac{c_2}{\lambda T_{rp}} \right) - 1 \right] + 1 \right\}^{-1},$$

where I_{rp} is the value of the photocurrent of the comparator, initiated by the radiation of the cell of the high-temperature reference point; I_{meas} is the value of the photocurrent initiated by the radiation of the investigated blackbody emitter, the whose temperature is being measured; T_{rp} is the temperature of the phase transition of the reference point.

Metrological characteristics of GET 34-2020. The range of temperature unit reproduction is 0–3200°C. The standard provides reproduction and transmission of a unit with a standard deviation (RMS) of the measurement result for five independent measurements. Table 1 shows the ranges of the following metrological characteristics of GET 34-2020: RMS; non-excluded systematic error $\delta_{n,s}$; total standard uncertainty u_Σ ; expanded uncertainty U_p at a coverage factor $k = 2$.

Results of comparisons of GET 34-2020 with national standards of other countries. An objective assessment of the equivalence of GET 34-2020 to the national standards of the leading metrological institutes of other countries can be obtained on the basis of the results of their comparisons. In the period 2017–2018, key comparisons of the Advisory Committee for Thermometry K-9.1 were carried out, whose purpose was to assess the equivalence of the temperature unit

TABLE 1. Ranges of Metrological Characteristics of GET 34-2020

Range, °C	RMS, °C	$\delta_{n.s.}$, °C	u_{Σ} , °C	U_p , °C
0–961.78	$(0.03–1.20) \cdot 10^{-3}$	$(0.04–1.70) \cdot 10^{-3}$	$(0.04–1.70) \cdot 10^{-3}$	$(0.08–3.40) \cdot 10^{-3}$
961.78–3200	0.08–0.80	0.12–1.04	0.094–1.000	0.19–2.00

TABLE 2. Results of Comparisons of Standards in the Range of 0.010–419.527°C

Reference point	$U_{\Sigma}(\text{FP})$, mK		ΔT , mK, K9-1
	PTB	VNIIM	
Ga	0.27	0.134	–0.02
In	0.86	0.52	0.55
Sn	0.90	0.60	0.55
Zn	1.30	0.92	0.44

TABLE 3. Comparison of Thermodynamic Temperature of Phase Transitions of Copper and Eutectics

Metal	T_{VNIIM} , K	T , K, citation	ΔT , K
Cu	1357.79	1357.77 [3]	0.020
Co–C	1597.23	1597.36 [13]	0.126
Re–C	2746.82	2747.00 [13]	0.184
WC–C	3020.93	3021.00 [14]	0.070

standard GET 34-2020 from the Mendeleev VNIIM. The comparisons confirmed the closeness of the metrological characteristics of the national standards of Germany (PTB) and Russia. The results of comparisons of standards in the range of 0.010–419.527°C are given in Table. 2, where $U_{\Sigma}(\text{FP})$ is extended uncertainty of reference points; ΔT is the difference in temperatures determined by the PTB and VNIIM standards [12].

At temperatures above 961.78°C, it was not possible to compare the modernized standard with other national standards that reproduce the unit of temperature in accordance with its new definition. Therefore, the results of temperature measurements of high-temperature reference points measured by the modernized standard GET 34-2020 were compared with previously published results obtained by other national metrological institutes.

Table 3 shows the thermodynamic temperature T_{VNIIM} of phase transitions of copper and eutectics, measured by the direct method at VNIIM, and the published results of measurements of T performed at VNIIOFI (Russia), LNE-CNAM (France), and NPL (Great Britain).

Conclusion. As a result of the improvement of GET 34-2007, GET 34-2020 was created and approved by Order of Rosstandart No. 2198 dated December 23, 2020. GET 34-2020 is the new State primary standard of temperature unit in the range of 0–3200°C; it contains equipment complexes for the implementation of ITS-90 in the range of 273.15–1235 K and reproduction of Kelvin above the solidification point of silver in accordance with the new definition of Kelvin.

The achieved values of the investigated metrological characteristics of GET 34-2020, as well as the results of international comparisons allow us to assert that GET 34-2020, created as an improvement, meets the needs of science and industry, and is at a level equivalent to the best national standards of other countries.

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