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*Masharipov Shodlik Masharipovich,
PhD in technical sciences,
associate professor, Tashkent State
Technical University, Tashkent, Uzbekistan
E-mail: shodlik29081986@mail.ru*

*Miralieva Aziza Kayumovna,
Senior lecturer,
Tashkent State Technical University,
Tashkent, Uzbekistan*

*Fattoev Firuz Farkhod ugli,
teaching assistant,
Tashkent State Technical University,
Tashkent, Uzbekistan,*

*Rakhmatullaev Sarvar Anvarovich,
teaching assistant,
Tashkent State Technical University,
Tashkent, Uzbekistan*

DEVELOPMENT OF THE METHOD OF CALCULATION OF UNCERTAINTY OF MEASUREMENT RESULTS AND EVALUATION OF ACCURATE CHARACTERISTICS IN THE FIELD OF ANALYTICAL MEASUREMENTS

Abstract. The issues of assessing the uncertainty of the results of analytical measurements in modern laboratory practice are considered taking into account the requirements of international standards in the field of metrology. The results of the development of a methodology for calculating the uncertainty of measurement results and evaluating the accuracy characteristics, the main content, procedure and calculation sequence according to the requirements of the international document GUM (Guide to the expression of measurement results) are presented.

Keywords: uncertainty of the measurement result, sources of uncertainty, calculation algorithm, standard uncertainty, total standard uncertainty, expanded uncertainty, quality of measurements, quantity, coverage factor, measured quantity.

The developed methodology consists of the following sections: purpose of the methodology; statement of the measuring problem; measurement model; measurement results; analysis of input quantities; correlation; total uncertainty; expanded uncertainty. This technique is designed to calculate the measurement uncertainty when

determining the mass concentration of aldehydes in vodka.

The methodology is developed in accordance with the requirements of GUM (Guidelines for the Expression of Uncertainty of Measurement Results), EURACHIM / SITAC CG 4 Guidelines (Quantitative Description of Uncertainty in Analytical

Measurements), ILAC G17: 2002 Presentation of the concept of measurement uncertainty in tests in conjunction with the application of ISO / IEC 17025 and other existing regulatory and methodological documents on the concepts of uncertainty.

The method is based on the chromatographic separation of microimpurities in a sample of vodka and their subsequent detection by a flame ionization detector (PID). The analysis takes 15–25 minutes. The operating principle of the chromatograph used is based on the application of gas adsorption and gas-liquid chromatography methods in the isothermal mode and the linear programming mode of the temperature and (or) flow rate of the carrier gas of chromatographic columns. Chromatographs are an analytical unit in the form of a monoblock with a medium or large column thermostat, on which an analytical module with detectors, injectors, dosing devices, chromatographic columns is installed. The chromatograph also includes a personal computer, NetChrom V2.1 chromatographic information processing software, and chromatographic analysis techniques. The increased volume of the column thermostat allows you to place in it, in addition to several columns, both packed and capillary longer, input devices and switch columns. Chromatograph devices have a high inertness to the analyzed compounds.

Chromatographs are available in two versions: version 1 with a column thermostat volume of 6 l, version 2 with a column thermostat volume of 14 L. Chromatographs are equipped with a wide range of detectors, both single and multi-detector (up to three detectors, both universal and selective): flame ionization detector (PID), thermal conductivity detector (DTP), electron-capture detector (ECD), flame pho-

tometric detector (PFD), thermionic detector (TID), photoionization detector (PID), thermochemical detector (TCD), gas ionization detector (HID). The Kristallux-4000M gas chromatograph is fully automated, from the input of the sample to the processing of the chromatographic information, including the functions of automatic control of the temperature of thermostats, flow rates and pressure of the carrier gas (EUPG system), auxiliary gases, automatic ignition of detectors, control of flame burning during operation, measuring detector signals using a 24-bit ADC.

A gas chromatograph includes more than 30 basic models, each of which can be adapted to a specific consumer task. The chromatograph consists of an analytical unit, a control station, control and processing of chromatographic information, which is used as a personal computer, and the program "NetChrom", running in Windows. In addition, additional programs are supplied: calculation of the calorific value of natural gas, diagnostics of transformer oil, calculation of Shekhar control charts, identification of multicomponent mixtures (for example, vegetable oil, cognac, hydrocarbon fuel, etc.), data output to an external monitor.

One computer can work in real time with several analytical units (up to eight), and in addition, control the work and process signals from the Crystal 2000 and Crystallux 4000 gas chromatographs, and process signals from other brands of chromatographs through ADCs. Information is exchanged between a computer, analytical units and a chromatograph via standard interfaces such as RS-232C, USB. It is possible to control the chromatograph from a distance of up to 3000 m. To set modes and process information without using a computer, there is a remote control panel for the chromatograph based on a microcomputer with OC Windows.

Table 1. – Values Contributing to Uncertainty

№	Influencing value	Designation	Unit of measurement
1	2	3	4
1.	Hamilton CO., Reno Nevada, Microliter™ # 701 Made in USA 10 ml syringe, 0.2 ml scale (mm ³)	U_{ui}	ml
2.	2 ml micro vial, Agilent scale [0.5–1.5], scale division 0.5 ml	U_m	ml

1	2	3	4
3.	Chromatograph Crystallux-4000 M	U_x	%
4.	Mechanical stopwatch, type SOS pr 2b, error 1.1%	U_s	S
5.	Convergence of measurement results and arithmetic mean	σ_c	%

Since the measurement is the product and the ratio of the uncorrelated input quantities, the total uncertainty is presented as the relative total uncertainty by the formula

$$u_c(X) = X \sqrt{\left(\frac{u(m_{\text{нир.}})}{m_{\text{нир.}}}\right)^2 + \left(\frac{u(V_{\text{р-ранир}})}{V_{\text{р-ранир}}}\right)^2 + \left(\frac{u(V_{\text{кислоты}})}{V_{\text{кислоты}}}\right)^2 + \left(\frac{u(V_{\text{водки}})}{V_{\text{водки}}}\right)^2 + \left(\frac{u(V_{\text{нир}})}{V_{\text{нир}}}\right)^2 + \left(\frac{u(D)}{D}\right)^2 + \left(\frac{u(\sigma_c)}{1}\right)^2}$$

Where $u(m_{\text{нир.}})$, $u(V_{\text{р-ранир}})$, $u(V_{\text{кислоты}})$, $u(V_{\text{водки}})$, $u(V_{\text{нир}})$ calculated by the formula:

$$u(m_{\text{нир.}}) = \sqrt{u(m_{\text{нир.лин.}})^2 + u(m_{\text{нир.в.}})^2 + u(m_{\text{нир.сч}})^2} = 4.330 \cdot 10^{-3}$$

$$u(V_{\text{р-ранир}}) = \sqrt{u(V)^2 + u(V_t)^2} = 4.127 \cdot 10^{-2}$$

$$u(V_{\text{кислоты}}) = \sqrt{u(V_1)^2 + u(V_{t1})^2} = 8.254 \cdot 10^{-3}$$

$$u(V_{\text{водки}}) = \sqrt{u(V_2)^2 + u(V_{t2})^2} = 2.063 \cdot 10^{-2}$$

$$u(V_{\text{нир}}) = \sqrt{u(V_3)^2 + u(V_{t3})^2} = 8.254 \cdot 10^{-3}$$

The expanded uncertainty of the presented methodology is calculated by substituting the calculated values in the formula.

$$u_c(X) = 1.351 \cdot \sqrt{\left(\frac{4.330 \cdot 10^{-3}}{0.100}\right)^2 + \left(\frac{4.127 \cdot 10^{-2}}{100}\right)^2 + \left(\frac{8.254 \cdot 10^{-3}}{2.00}\right)^2 + \left(\frac{2.063 \cdot 10^{-2}}{5.00}\right)^2 + \left(\frac{8.254 \cdot 10^{-3}}{1.50}\right)^2 + \left(\frac{2.887 \cdot 10^{-3}}{0.125}\right)^2 + \left(\frac{0.025}{1}\right)^2} = 0.0646$$

Calculation of extended uncertainty. The expanded uncertainty U is obtained by multiplying the standard uncertainty of the output quantity $u_c(y)$ by the coverage factor k .

$$U = k \cdot u_c(y)$$

When choosing a coverage factor value, consider:

- the required level of confidence;
- any information on the intended distribution;
- information on the number of observations used to estimate random effects.

The coverage factor when evaluating the expanded uncertainty is chosen in accordance with the following recommendations.

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