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Methods of Estimation of Uncertainty of Results of Direct and Indirect Measurements of Analytical Values

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ABSTRACT: Methods of estimating uncertainty of results of direct and indirect measurements of analytical values have been analysed. Analytical expressions for numerical calculation of main sources of physical and chemical values are given. Significant sources of measurement uncertainty have been simulated. The apparatus effects may include, for example, error limits of the analytical weights; The presence of a temperature regulator that can maintain an average temperature that differs (within specified limits) from the temperature recorded; Automatic analyzer, which can be subject to overload effects. Taking into account these specifics of analytical measurements, calculations of standardized standard uncertainties of measurement results according to GOST 1770 have been performed

KEYWORDS: Coverage factor, coverage probability, coverage interval, type A uncertainty, type B uncertainty, extended uncertainty, measurements, measured value, analytical values, direct measurements, indirect measurements, correlated value, uncorrelated value

I. INTRODUCTION

Many important decisions are based on chemical quantitative analysis; Results are used, for example, to estimate yields, to test materials for compliance with specifications or statutory restrictions, or to estimate monetary value. Whenever decisions are based on analytical results, it is important to have some indication of the quality of the results, that is, the extent to which they can be used to achieve the goal. Users of chemical analysis, particularly in those areas related to international trade, face increasing pressure to avoid duplication of effort often required to obtain them. Confidence in data obtained outside the user 's own organization is a prerequisite for achieving the above goal.

II.LITERATURE SURVEY

In some analytical chemistry sectors, this is now a formal (often statutory) laboratory requirement to introduce quality assurance measures to guarantee the ability and provide the data of the required quality. Such measures include the use of proven methods of analysis; The use of certain internal quality control procedures; Participation in qualification programmes; Accreditation based on ISO/IEC 17025:2017 and establishing traceability of measurement results. Depending on the type of information available on the value and the possible variability of the value (statistical or non-statistical), the uncertainties of the input values are known to be estimated by type A or type B.

III. RESULTS AND DISCUSSION

If the value information is statistical, i.e. obtained experimentally by multiple measurements or tests, its standard uncertainty due to random effects is estimated by type A (1)

$$u_A(\bar{x}) = s(\bar{x}) = \sqrt{\frac{1}{n(n-1)} \cdot \sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2} \qquad (1.)$$

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where $\bar{x}$ – assessment (an average of arithmetic value) of entrance X size; x_i – is the result of the i-th observation of the input value; n – is the number of observations.

Experimental dispersion of observations is estimated by (2):

$$s^2(x) = \frac{1}{n-1} \cdot \sum_{i=1}^{n} (x_i - \bar{x})^2 \qquad (2)$$

Before measurement, first of all, we draw up a list of influencing factors on extended uncertainty of measurements.When measuring the density of ethanol (C_2H_5OH) by means of a measuring bulb (according to GOST 1770), the density is according to the equation.

$$\rho(C_2H_5OH) = \frac{m_2 - m_1}{V} \qquad (3)$$

Where m_1 - is the mass of the flask, m_2 - is the mass of the flask with ethanol.

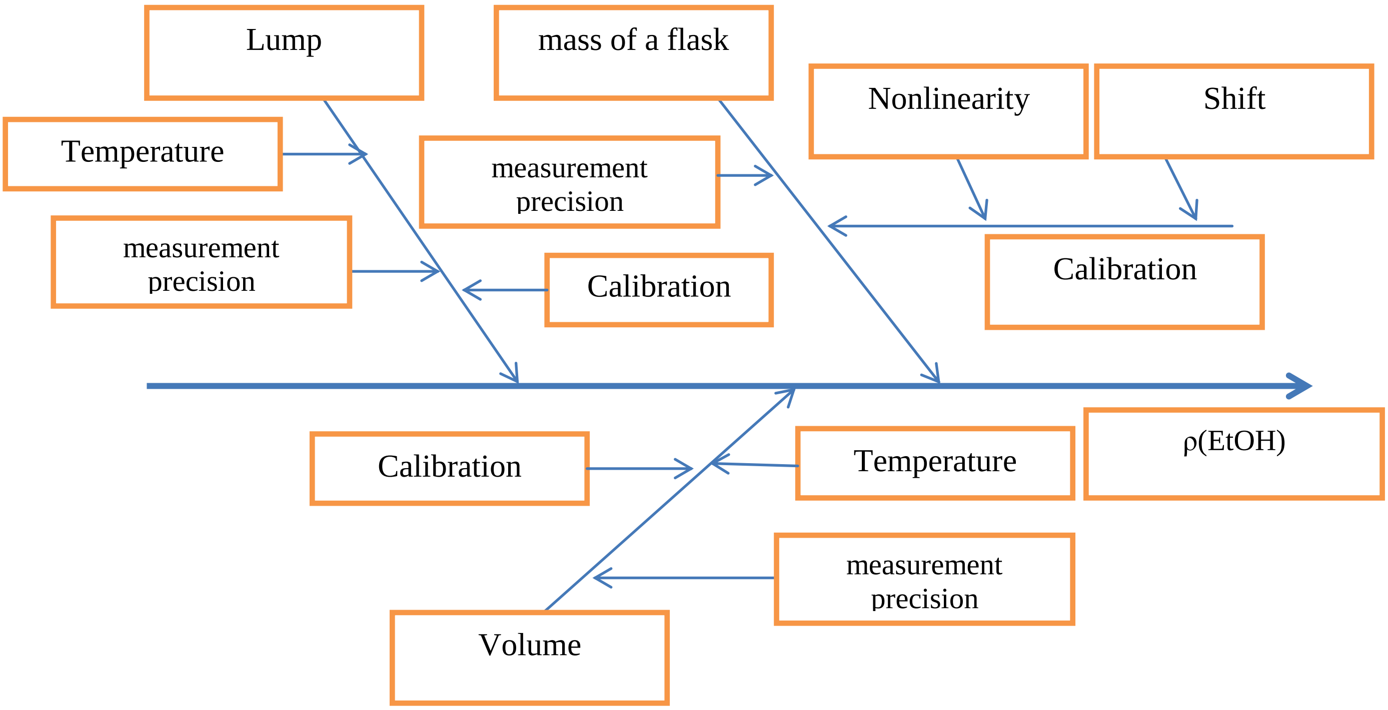

Fig. 1. Main groups and subgroups of sources of uncertainty in ethanol density measurement

This mathematical model reflects the main sources of uncertainty (Fig. 1). From fig. 1. It can be seen that the main sources of measurement uncertainty are directly related to the total mass of the bulb and volume. Besides, normalized limit of bulb error is regulated at t = 20 °C.

The total standard uncertainty is calculated by formula (4):

$$u_c(y) = \sqrt{\sum_{i=1}^{n} \left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i)} \qquad (4)$$

If the input values are not correlated. Otherwise, for correlated input values, it is calculated by formula (5)

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$$u_c(y)=\sqrt{\sum_{i=1}^{n}\sum_{j=1}^{n}\frac{\partial f}{\partial x_i}\frac{\partial f}{\partial x_j}u(x_i,x_j)}=\sqrt{\sum_{i=1}^{n}\left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i)+2\sum_{i=1}^{n-1}\sum_{j=i+1}^{n}\frac{\partial f}{\partial x_i}\frac{\partial f}{\partial x_j}u(x_i,x_j)} \quad (5)$$

where private derivatives $\partial f/\partial x_i$ –sensitivity coefficients; $u(x_i,x_j)$ –covariance of entrance sizes. Sensitivity indexes

$$c_i=\frac{\partial f}{\partial x_i} \quad (6)$$

Show how the output estimate y changes with the values of the input estimates $x_1, x_2,...,x_n$.

With (6) in mind, the formulae (4) and (5) are converted to the following expressions:

$u_c(y)=\sqrt{\sum_{i=1}^{n}\left(\frac{\partial y}{\partial x_i}\right)^2\cdot u^2(x_i)}=\sqrt{\sum_{i=1}^{n}u_i^2(y)}$	(7)
$u_c(y)=\sqrt{\sum_{i=1}^{n}\left(\frac{\partial y}{\partial x_i}\right)^2\cdot u^2(x_i)+2\sum_{i=1}^{n-1}\sum_{j=i+1}^{n}\left(\frac{\partial y}{\partial x_i}\right)\cdot\left(\frac{\partial y}{\partial x_j}\right)\cdot u(x_i)\cdot u(x_j)\cdot r(x_i,x_j)}$	(8)
$u_i(y)=\frac{\partial y}{\partial x_i}\cdot u(x_i)$	(9)

For the special case where all input estimates are correlated with correlation coefficients $r(x_i, x_j) = +1$, equation (8) is reduced to

$$u_c(y)=\sum_{i=1}^{n}u_i(y) \quad (10)$$

For the sum or difference of the two correlated values (Y = X1 ± X), the total standard uncertainty (according to (8)) will be:

$$u^2(y)=u^2(x_1)+u^2(x_2)\pm 2\cdot u(x_1)\cdot u(x_2)\cdot r(x_1,x_2) \quad (11)$$

If the two inputs X_i and X_j are correlated to a certain extent, that is, they are dependent on each other in one way or another, then when estimating the total standard uncertainty among the contributions of the uncertainties of the inputs, their covariance must be taken into account, which is estimated by the following formula:

The degree of correlation is determined by the correlation coefficient. The estimated correlation coefficient is obtained from equation (11).

$$r(\bar{x}_i,\bar{x}_j)=u(\bar{x}_i,\bar{x}_j)/u(\bar{x}_i)\cdot u(\bar{x}_j)\cdot,\ \ i\neq j,\ \ \left|r(\bar{x}_i,\bar{x}_j)\right|\le 1 \quad (12)$$

Figures 2 and 3 show the permissible errors from the nominal capacity of glass measuring utensils and the limits of the permissible error on the volume of pipettes with one mark. If in the certificate or other technical documentation the limits of permissible error are given without indication of confidence probability or the estimate is given in the form of maximum range (± a), and the form of distribution is unknown, a uniform law of distribution should be used (Fig. 4.)

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Table 1.
Permissible errors from nominal capacity of glass measuring utensils, cm^3

Nominal capacity	Admissible error				
	Cylinders		Graduated cylinders	Flasks	
	The 1st class	The 2nd class		The 1st class	The 2nd class
5	0,10	0,10	-	0,025	0,05
10	0,10	0,20	-	0,025	0,05
25	0,25	0,50	-	0,04	0,08
50	0,25	1,00	2,50	0,06	0,12
100	0,50	1,00	5,00	0,10	0,20
200	-	-	-	0,15	0,30
250	1,25	2,00	5,00	0,15	0,30
300	-	-	-	0,20	0,40
500	2,50	5,00	12,50	0,25	0,50
1000	5,00	10,00	25,00	0,40	0,80
2000	10,00	20,00	-	0,60	1,20

Table 2.
Limits of permissible error by volume of pipettes with one elevation, cm^3

Nominal capacity	Limit blundered	
	The 1st class	The 2nd class
0,5	±0,005	±0,01
1	±0,008	±0,015
2	±0,01	±0,02

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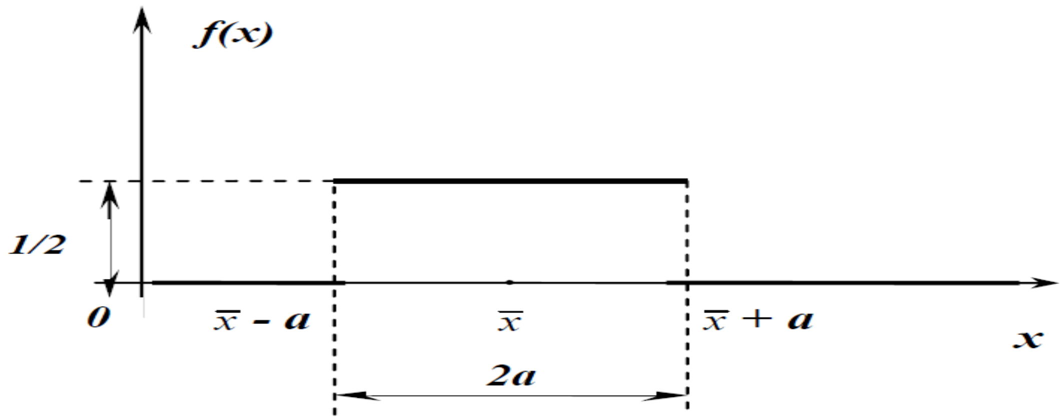
5	±0,015	±0,03
10	±0,02	±0,04
10,77	±0,02	±0,04
20	±0,03	±0,06
25	±0,03	±0,06
50	±0,05	±0,1
100	±0,08	±0,15
200	±0,1	±0,2



Rice 2. Uniform distribution probability density function
(A- interval half-width, x - random measurement result)

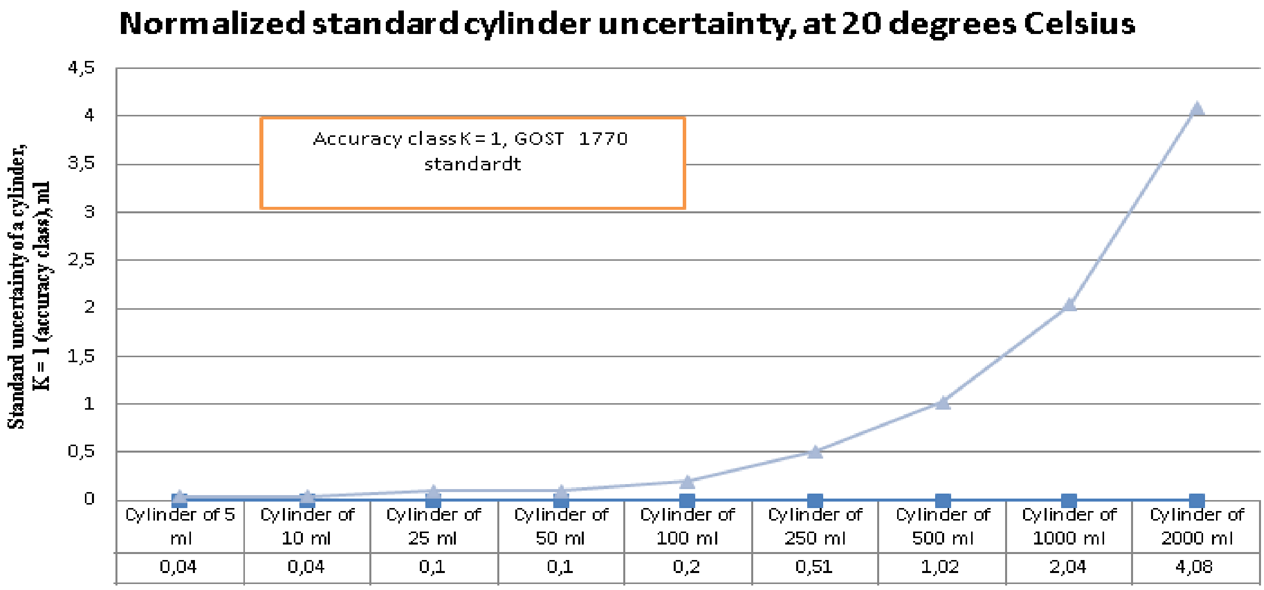


Fig. 3. Normalized standard cylinder uncertainty (u_{CT}), K = 1 (accuracy class)

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On tab. 1. 2 shows the permissible errors from the nominal capacity of glass measuring utensils and the limits of the permissible error by volume of pipettes with one mark. If in the certificate or other technical documentation the limits of permissible error are given without indication of confidence probability or the estimate is given in the form of maximum range (± a), and the form of distribution is unknown, then a uniform law of distribution should be used (Fig.2.). Figure 3 shows the normalized standard cylinder uncertainty, K = 1 (accuracy class). It can be seen from the figure that the standard uncertainty as the nominal cylinder volume increases accordingly decreases. In addition, the value of uct depends on the accuracy class K (Fig.4).

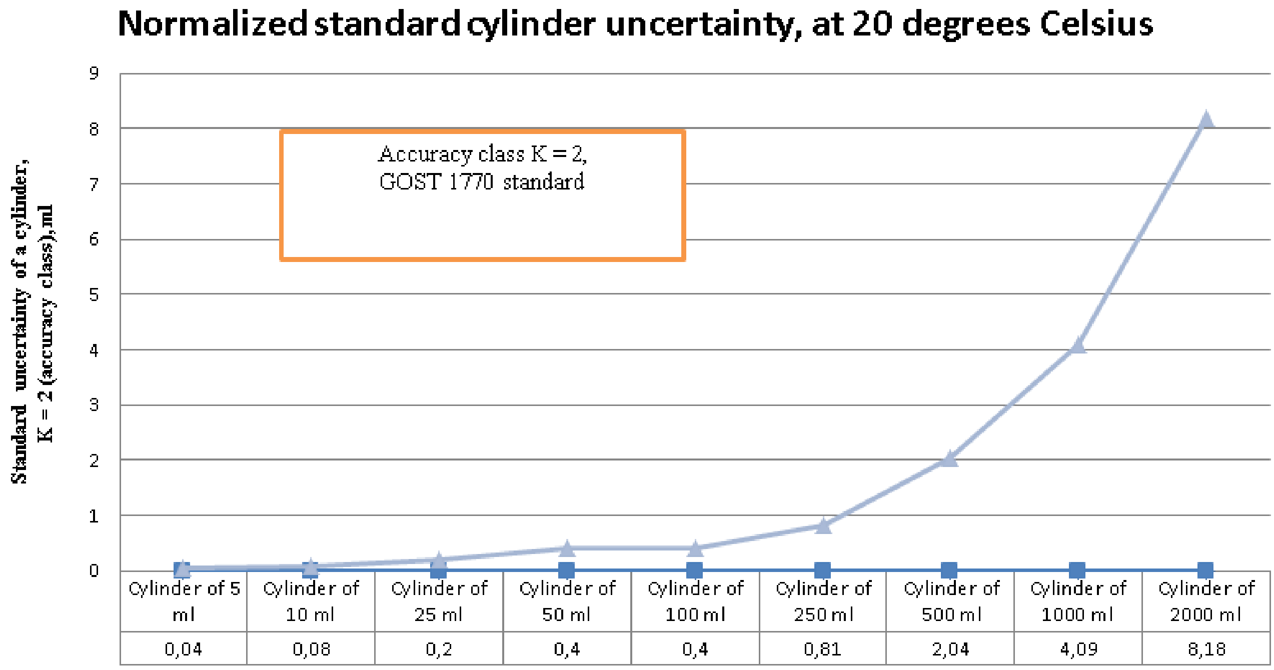

Fig. 4. Normalized standard cylinder uncertainty (u_{cm}), K = 2 (accuracy class)

V.CONCLUSION

The document of the International organization for cooperation in accreditation of laboratories (ILAC/ILAK) in which also questions of introduction of the concept of measurement uncertainty at tests taking into account application of the ISO/IEC 17025 standard are devoted is devoted to the major factors (not in all cases they significant) affecting total measurement uncertainty. According to ISO 10012 and JCGM 104, some uncertainties may not be significant compared to other components, so their detailed definition may be unreasonable for economic or technical reasons. In such a case, decisions and justifications must be registered. In all situations, efforts to assess and record measurement uncertainties should be commensurate with the importance of measurement results to product quality.

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