Ural Scientific Research Institute for Metrology,
ROSSTANDART, RUSSIA

Report of the CCQM-K130

Nitrogen mass fraction measurements in glycine

FINAL REPORT: July 2016

Coordinating laboratory:

Ural Scientific Research Institute for Metrology, ROSSTANDART,

Ekaterinburg, **UNIIM**

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1 ABSTRACT

Mass fraction of nitrogen is very important pointer because the results of these measurements are often used for determination of protein mass fraction that is an important indicator of the quality of the vast majority of food products and raw materials, in particular dry milk powder. Proteins-enzymes catalyze chemical reactions, protein along with fats and carbohydrates is one of the indicators characterizing the energy value of food, so its definition is mandatory for all food products.

The aim of this key comparison CCQM-K130 and pilot study P166 is to support National Measurement Institutes (NMIs) and Designated Institutes (DIs) to demonstrate the validity of the procedures the employed for determination of nitrogen mass fraction in glycine.

The study material for this key comparison and pilot study has been selected to be representative as one of the aminoacid – the simplest part of the protein. Glycine is an amino acid, single acid that does not have any isomers (melting point –290 °C; specific heat of evaporation − 528,6 J/kg; specific melting heat − 981,1 J/kg; pKa − 2,34, molar mass - 75,07 g/mol, density - 1,607 g/cm^3).

Ural Scientific Research Institute for Metrology (UNIIM) acted as the coordinating laboratory of this comparison and pilot study.

Eight NMIs participated in this key comparison and two NMIs participated in Pilot study.

The results of Pilot study are excluded from the Report B.

2 INTRODUTION

Nitrogen mass fraction is a relevant indicator for food products and food raw materials. Kjeldahl Titrimetric method is often used for the measurements of this pointer. Despite the occurrence of a number of the other methods for the measurements of nitrogen content, such as Dumas method, infrared spectroscopy, chromatography etc., Kjeldahl method remains the most accurate and reliable method of the measurement of nitrogen (protein) mass fraction. Kjeldahl method is admitted as a reference method by various organizations, the most known of them are listed [1]:

- AOAC International
- American Oil Chemists' Society
- American Public Health Association (APHA)
- American Society for Testing and Materials (ASTM)
- Association of American Cereal Chemists
- European Commission
- International Dairy Federation (IDF)
- International Organization for Standardization (ISO)
- U.S. Department of Agriculture

  U. S. Environmental Protection Agency (EPA)

But according to technical report participants are allowed to use any suitable methods of analysis.

There are no CMCs in measurement of nitrogen mass fraction in glycine in the database of BIPM. But China NIM (National Institute of Metrology) has calibration and measurement capabilities in determination of nitrogen mass fraction in non fat milk powder. Mechanism for measurement service delivery of this CMC is kept by CRM GBW08509. This CMC was approved on 13 June 2013.

But key comparison has never been carried out in the field of measurement both glycine and milk powder.

3 LIST OF PARTICIPANTS

Eight NMIs or DIs participated in the key comparison CCQM-K130. Table 1 contains the full names of all participating NMIs and DIs and contact persons.

Table 1 List of participants

Institute	Abbreviation	Country	Contact persons	Kind of comparison
National Institute of Metrology, Quality and Technology	INMETRO	Brazil	Eliane C. P. do Rego, Wagner Wollinger, Tânia M. Monteiro, Lucas J. de Carvalho	Key
Instituto Nacional de Calidad	INACAL	Perú	Steve Ali Acco Garcia	Key
SP Technical Research Institute of Sweden	SP	Sweden	Conny Haraldsson	Key
National Institute of Industrial Technology	INTI-1	Argentina	M. Alejandra Rodriguez, Gabriela Rodriguez	Key
Laboratorio Tecnológico del Uruguay	LATU	Uruguay	Karino Salvo	Key
State Enterprise All-Ukrainian State Research and production Center of Standardization Metrology, Certification and Consumers' Rights Protection	UkrCSM	Ukraine	Vladimir Gavrilkin, Sergij Kulyk	Key
Measurement Standards Laboratory of New Zealand	MSL	New Zealand	Laly Samuel	Key
Ural Scientific Research Institute for Metrology	UNIIM	Russia	Maria Medvedevskikh, Maria Krasheninina	Key

4 SAMPLE

The comparison material for the CCQM-K130 was analytical grade glycine from a commercial supplier. The material was supplied as a white solid and was not subject to further purification. The analysis certificate provided with the material describes its purity as 99,7 %. This material of glycine was subdivided into vials from dark glass. But before packing material of glycine was dried until dry substances under the temperature of 105 degrees above zero during two hours. Then vials were packaged in double waterproof bags. Each vial contains 5 g.

After preparation of the samples, homogeneity test has been carried out. Homogeneity test for glycine is presented in table 2.

Table 2 Results of homogeneity testing between bottles (5 replicates for each bottle)

Vial	**Nitrogen mass fraction, %**					**Mean value in vial, %**
	1	**2**	**3**	**4**	**5**	
1	18,57	18,48	18,56	18,53	18,47	18,52
2	18,57	18,53	18,51	18,56	18,45	18,52
3	18,47	18,42	18,50	18,44	18,53	18,47
4	18,52	18,50	18,51	18,47	18,62	18,52
5	18,72	18,50	18,52	18,55	18,66	18,59
6	18,66	18,50	18,67	18,54	18,46	18,57

In order to estimate the inhomogeneity contribution u_h, a 1-way Analysis of Variances (ANOVA) has been carried out with the experimental homogeneity data (table 1). The standard uncertainty due to (in)homogeneity, u_h, value for glycine (see Table 3, 4) were calculated according to ISO Guide 35 using the Equations (1) and (2).

$$u_h = \sqrt{\frac{MS_{among} - MS_{within}}{n}} \quad (1)$$

$$u_h = \sqrt{\frac{MS_{within}}{n}} \sqrt[4]{\frac{2}{N(\mathrm{n}-1)}}, \quad (2)$$

where N=6, n=5.

Table 3 ANOVA analysis

Vial	***number***	***sum***	***average***	***dispersion***
1	5	92,619	18,524	0,0022
2	5	92,611	18,522	0,0024
3	5	92,363	18,473	0,0022
4	5	92,619	18,524	0,0031
5	5	92,944	18,589	0,0089
6	5	92,833	18,567	0,0093

Table 4 ANOVA analysis

source	***SS***	***df***	***MS***	***F***
Among	0,040918	5	0,008184	1,7524
Within	0,112078	24	0,004670	
Sum	0,15299	29		
standard uncertainties due to inhomogeneity, u_h		**0,027**	%	Equation (1)
standard uncertainties due to inhomogeneity, $\boldsymbol{u_h}$		-	-	Equation (2)
relative standard uncertainties due to inhomogeneity, $\boldsymbol{u_{ho}}$		**0,14**	**%**	

Stability test for glycine is presented in table 5 and figure 1. Long-term stability study has been conducted with the help of isochronous experiment. Four samples were being kept in the drying oven under the temperature (90±5) °C. Time of sample keeping in such conditions is accounted according to equation:

$$\tau = \frac{T}{2^{\frac{t_1 - t_0}{10}}}, \quad (3)$$

где τ –time of conducting the experiment, days;

T – estimated shelf life, days;

t_1 - temperature of testing of samples (90±5), °C;

t_0 - temperature of keeping of samples, °C.

Table 5 Results of measurement of nitrogen mass fraction in glycine

№	Date	Nitrogen mass fraction, %
1	21.08.2015	18,52
2	24.09.2015	18,56
3	26.11.2015	18,46
4	21.12.2015	18,70
mean of stability test, Xs		18,56
standard deviation of the data of key comparison participants, S		0,10
Xs+S		18,66
Xs-S		18,46
slope,*b*		0,0073
standard uncertainty of slope, u_{slope}		0,0071
confidence interval $t_{0,05;(n-2)} \cdot u_{slope}$		0,03
standard uncertainty due to long-term (in)stability, u_s		0,67
relative standard uncertainty due to long-term (in)stability, u_{so}, %		0,32
time measurements in key comparison, t, days (according to isochronous experiment)		360

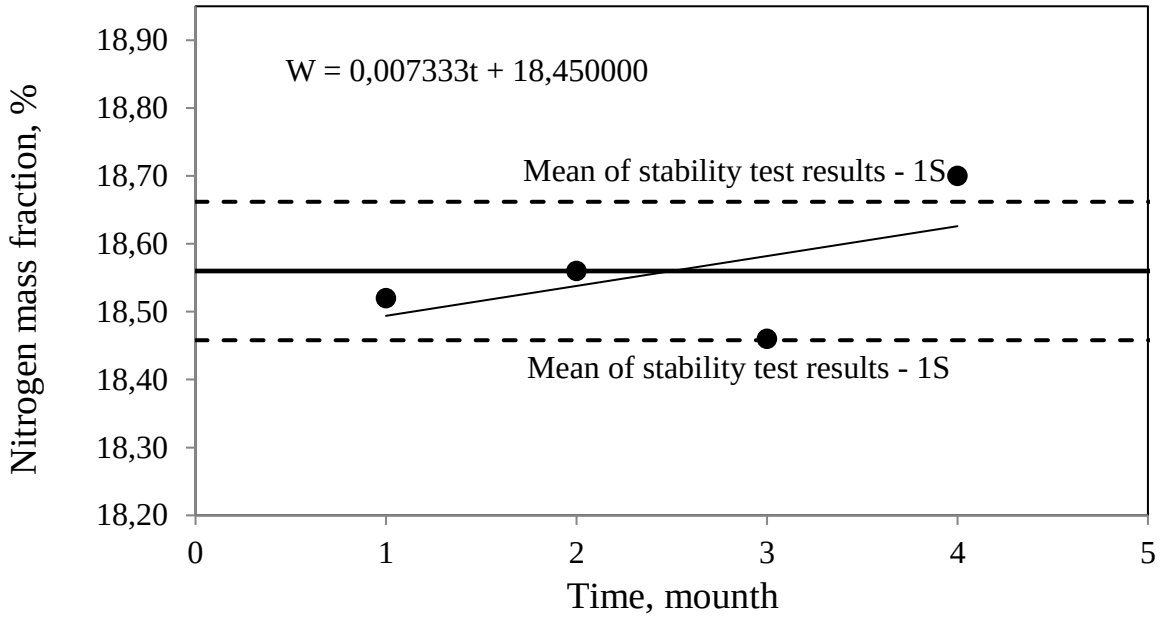

Figure.1 - Stability test for glycine

Data in table 5 was accounted using linear regression method. Standard uncertainty due to instability was calculated using formula:

$$u_s = \sqrt{u_{slope} \cdot t}\,, \tag{4}$$

Additionally, standard uncertainty due to short-term instability has been estimated. The statistical evaluation of the homogeneity, long-term and short-term stability has indicated that standard uncertainties due to inhomogeneity is 0,027 %, and long-term instability is 0,042 % and short-term instability is 0,012 %.

The samples has been sent to the participants by DHL on 19th August 2015. Each sample has been accompanied by veterinary certificate of international view. All samples arrived to their destination without damage but for different countries, it has taken different time: from several days to two month. The dispatch dates and receipt dates are given in Table 6.

The deadline for reporting results was set by end of February 2016 in order to prepare a presentation for discussion at the CCQM IAWG meeting in April 2016. All participants reported their results in time (except LATU and INTI).

Table 6 Sample sent dates, receipt dates and report dates

Institute	Sample dispatch date	Sample receipt date	Date report sent
INMETRO	19 August 2015	28 August 2015	29.02.2016
INACAL	19 August 2015	23 November 2015	29.02.2016
SP	19 August 2015	2 September 2015	29.02.2016
INTI-1	19 August 2015	16 September 2015	29.02.2016
LATU	19 August 2015	26 August 2015	10.03.2016
UkrCSM	19 August 2015	2 September 2015	29.02.2016
MSL	19 August 2015	2 September 2015	29.02.2016

5 INSTRUCTIONS FOR PARTICIPANTS

Technical protocol has been sent to the participants by e-mail.

The technical protocol (appendix A) contained background information, timing of the comparison, and information on the participating institutes. Information on sample preparation and recommendation of condition for measurements was given.

Each participant is allowed to use any suitable method of analysis.

Participants were requested the results of nitrogen mass fraction in glycine. The results should be reported accompanied by a full uncertainty statement (including a combined standard uncertainty and an expanded uncertainty with a coverage factor applied). In addition, the report should include technical details on the measurement procedure, traceability links (as calibrations) and uncertainty contributions.

6 METHODS OF MEASUREMENT

Seven participants used Kjeldahl method for the measurements and one participant used elemental method of analysis. Some details on measurements as derived from the reports are given in Table 7 and Table 8.

Table 7 Details of sample mass and titrant

Institute	Method of analysis	Approx. sample mass, g	Titrant, its molar concentration
INMETRO	Kjeldahl	0,7	Sulphuric acid 0,25 M
INACAL	Kjeldahl	0,500±0,005	Hydrochloric acid, 0,1 M
SP	Elemental method	0,035	-
INTI-1	Kjeldahl	0,130±0,007	Hydrochloric acid, 0,1 M
LATU	Kjeldahl	0,155±0,005	Hydrochloric acid, 0,1 M
UkrCSM	Kjeldahl	0,15±0,1	Sulphuric acid 0,05 M
MSL	Kjeldahl	0,15	Hydrochloric acid, 0,1 M
UNIIM	Kjeldahl	0,16±0,1	Sulphuric acid 0,05 M

Table 8 Traceability details

Institute	Traceability
INMETRO	Traceability is provided by using calibration material: buffer materials (pH=4,01±0,02 (25 ºC); pH=7,00±0,02 (25 ºC)). These buffer materials were acquired for automatic titrator Metrohn and were verified by Electrochemical Laboratory from Inmetro, whose is a CRM producer for pH solutions, using the pH primary measuring system.
INACAL	Traceability is provided by using potassium hydrogen phthalate (KHP) that is certified by coulometric titration.
SP	Traceability is provided by using calibration material: TRIS reference from Slovak Institute of Metrology LOT A0704414.
INTI-1	Traceability is provided by using: - Hydrochloric acid, 0,1 M (f=1) TitriPUR, Batch HC393273. The concentration of this volumetric solution was determined with volumetric standard TRIS (Merck).The determined titer at 20°C was 1,000 with an expanded measurement uncertainty of ±0,003 (k=2 coverage factor for 95% coverage probability). The certified value is traceable to primary standard NIST SRM 723e by means of volumetric standard TRIS, measured in the accreditated calibration laboratory of Merck KGaA in accordance to DIN EN ISO/IEC 17025. - L-Tryptophan, Merck, assay (perchloric acid titration, calculated on dry substance) > 99,0%. - Ammonium sulfate, Merck, assay (alkalimetric) > 99,5%. - Glycine, Merck, minimum assay (perchloric acid titration) 99,7% (mass fraction).
LATU	Traceability is provided by using: - Tris (hydroxymethil) aminomethane, reference material for acidimetry, traceable to NIST Standard Reference Material (SRM), lot 122408J, shelf life 2017/03/31. - L-Tryptophan certified reference material TraceCERT, EXP Jun/16 FLUKA lot BCBH4262V.
UkrCSM	Traceability is provided by using: Certified reference material of Sodium carbonate NIOCHIM (DSZU 023.36-06); mass fraction of Sodium carbonate 99.668 % in dried at 270 – 300 °C material that is certified by titration.
MSL	Traceability is provided by using: -NMIJ CRM3201-a05- 0.1mol/kg HCl traceable to SI and certified by coulometric titration.
UNIIM	Traceability is provided by using: - UNIIM GSO 10450-2014 (high purity sodium carbonate that is used for determination molar concentration of sulphuric acid) that is certified by coulometric titration.

7 RESULTS AND DISCUSSION

7.1 Uncertainty

Participants have used different approaches for estimations of measurement uncertainty of nitrogen mass fraction by Kjeldahl method and Elemental method of analysis and have accounted different sources of uncertainty in budget of uncertainty. Some details about sources of uncertainty are given in Table 9.

Table 9 Details about results and sources of uncertainty

Institute	Accounted sources of uncertainty
INMETRO	**Type A** - repeatability of measurement results **Type B** - standard uncertainty due to volume of titrant in blank - standard uncertainty due to volume of titrant in sample - standard uncertainty due to factor of titrant - standard uncertainty due to sample mass
INACAL	**Type A** - repeatability of measurement results **Type B** - standard uncertainty due to molecular weight of nitrogen and potassium hydrogen phthalate
SP	**Type A** - mean instrument signal for test portion 1 - mean instrument signal for Reference for test portion 1 - mean instrument signal for test portion 2 - mean instrument signal for Reference for test portion 2 - mean instrument signal for test portion 3 - mean instrument signal for Reference for test portion 3 **Type B** - amount content of base expressed as TRIS - atomic weight of nitrogen
INTI-1	**Type A** - repeatability of measurement results. **Type B** - standard uncertainty due to sample weight - standard uncertainty due to titrant volume of hydrochloric acid standard volumetric solution (blank and test sample) - standard uncertainty due to concentration of hydrochloric acid standard volumetric solution
INTI-2	**Type A** - standard uncertainty type A was not presented. **Type B** -standard uncertainty due to titrant volume of hydrochloric acid standard volumetric solution (blank and test sample) -standard uncertainty due to sample weight -standard uncertainty due to concentration of hydrochloric acid standard volumetric solution -standard uncertainty due to repeatability
LATU	**Type A** - repeatability of measurement results **Type B** - standard uncertainty due to titration of hydrochloric acid standard volumetric solution (blank and test sample)

	- standard uncertainty due to sample mass - standard uncertainty due to concentration of the hydrochloric acid - standard uncertainty due to reproducibility of the laboratory on different days
UkrCSM	**Type A** - repeatability of measurement results **Type B** - sample weighting - EP determination - titrant volume determination - titrant concentration determination - nitrogen atomic mass uncertainty
MSL	**Type A** - repeatability of measurement results. **Type B** - standard uncertainty due to volumetric including pipette, burette and volumetric flask-calibration, repeatability, readability and end point bias - standard uncertainty due to standardization of NaOH - standard uncertainty due to CRM - standard uncertainty due to moisture measurement - standard uncertainty due to balance calibration, repeatability, buoyancy - standard uncertainty due to sample weight - standard uncertainty due to method recovery - standard uncertainty due to homogeneity
UNIIM	**Type A** - repeatability of measurement results **Type B** - standard uncertainty due to sample weight - standard uncertainty due to titrant volume of sulphuric acid standard volumetric solution (blank and test sample) - standard uncertainty due to concentration of sulphuric acid standard volumetric solution - standard uncertainty due to certified value of GSO 10450-2014 that was used for determination of molar concentration of sulphuric acid - standard uncertainty due to detection of end point of titration

7.2 Formulas

Preliminary inspection of value x_i and associated uncertainties $u(\overline{x}_i)$ has been carried out in accordance with CCQM guidance note [2] using the following equation

$$\frac{x_i - med(x)}{u(x_i)}, \tag{5}$$

The results of preliminary inspection have shown that in general there are consistent results with a small number of outlying results. It means that it's case – C according to the CCQM guidance.

Check of consistency have performed according to the CCQM guidance note [3] using algorithm is shown bellow (only results of participants key comparison used for calculation).

$$\overline{x}_u = \sum_{i=1}^{m} \frac{x_i / u^2(x_i)}{\sum_{i=1}^{m} 1/u^2(x_i)}, \tag{6}$$

$$\chi^2_{obs} = \sum_{i=1}^{m} \left(\frac{x_i - \overline{x}_u}{u(x_i)} \right)^2, \tag{7}$$

where x_i - result of value of *i* NMI, $u(\overline{x})$ - standard uncertainty of $\overline{x}$.

After calculations using formulas (6), (7) was compared χ^2_{obs} with m-1 and with $\chi^2_{0.05,m-1}$, the 95 percentile of χ^2 with m-1 of freedom.

If $\chi^2_{obs} < m-1$, it is normally safe to proceed with the assumption that the results are mutually consistent and that the uncertainties account fully for the observed dispersion of values.

If $m-1 < \chi^2_{obs} < \chi^2_{0.05,m-1}$ the data provide no strong evidence that the reported uncertainties are inappropriate, but the remains a risk that additional factors are contributing to the dispersion. Refer to the prior working group decision on presumptive consistency and proceed accordingly.

If $\chi^2_{obs} > \chi^2_{0.05,m-1}$ the data should be considered mutually inconsistent.

Candidates of the key comparison reference values (KCRV) were estimated following the CCQM guidance note [2] using different approaches. The result from participant in the parallel pilot study has not been taken into account to determine the KCRV. Results and uncertainties have been taken from the reports as they were. Formulas for calculation are shown bellow.

$$\overline{x} = \frac{1}{m} \sum_{i=1}^{m} x_i, \tag{8}$$

$$u(\overline{x}) = \frac{\sum_{i=1}^{m} (x_i - \overline{x})^2}{m(m-1)}, \tag{9}$$

where x_i - result of value of *i* NMI, $u(\overline{x})$ - standard uncertainty of $\overline{x}$.

Uncertainty-weighted mean

$$\bar{x}_u = \sum_{i=1}^{m} w_i x_i \,, \tag{10}$$

$$w_i = \frac{1/u^2(x_i)}{\sum_{i=1}^{m} 1/u^2(x_i)} \,, \tag{11}$$

$$\frac{1}{u^2(\bar{x}_u)} = \sum_{i=1}^{m} 1/u^2(x_i) \,, \tag{12}$$

where $u(x_i)$ - standard uncertainty of x_i.

Median

$$med(x) = \begin{Bmatrix} \frac{1}{2}(x'_{m/2} + x'_{m/2+1}), & even \;\; m \;\; even \\ x'_{(m+1)/2}, & m \;\; odd \end{Bmatrix}, \tag{13}$$

$$u^2(med(x)) = \frac{\pi}{2m}\hat{\sigma}^2 \,, \tag{14}$$

$$\hat{\sigma} = 1.483 med(|d_i|) \,, \tag{15}$$

where $d_i = x_i - med(x)$.

7.3 Nitrogen mass fraction in glycine

The reported values of nitrogen mass fraction and uncertainties of all results have been summarized in Table 10. Estimations of candidates KCRV have been obtained by different approaches (arithmetic mean, weighted mean, median) are presented in Table 10 (only results of participants key comparison used for calculation KCRV). The same results are displayed graphically in Figures 2, 3.

It is proposed to use the median of the KCRV, because:

- $\chi^2_{obs} > \chi^2_{0.05,m-1}$ in this case the data is mutually inconsistent,
- The reported uncertainties are not very different,
- There two extreme values according to $(x_i - med(x))/u(x_i)$,
- According to figure 2 transformed distribution for reported results of NMIs and DIs for nitrogen mass fraction is asymmetric.

Table 10 – Reported values of nitrogen mass fraction and uncertainties

№	Kind of com-pari-son	NMI/ DIS	Nitrogen mass fraction, %	Combined standard uncertainty, u_c, %	Expanded uncertainty, U(k=2), %	di, %	U(di), %	Verdict
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*
1	Key	INACAL	18,508	0,04	0,07	-0,05	0,09	+
2	Key	LATU	18,513	0,07	0,13	-0,05	0,14	+
3	Key	MSL	18,524	0,09	0,17	-0,04	0,18	+
4	Key	UNIIM	18,535	0,05	0,11	-0,03	0,12	+
5	Key	UkrCSM	18,585	0,06	0,13	0,03	0,14	+
6	Key	INMETRO	18,589	0,05	0,09	0,03	0,11	+
7	Key	INTI-1	18,606	0,05	0,10	0,05	0,12	+
8	Key	SP	18,655	0,03	0,05	0,10	0,08	-
median			18,560	0,03	0,05	**KCRV**		
mean			18,564	0,02	0,04			
weighted mean			18,588	0,01	0,03			
Consistency test						**Conclusion**		
χ^2_{obs}			$\chi^2_{0.05,m-1}$		*m*	$\chi^2_{obs} > \chi^2_{0.05,m-1}$		
14.02			2.2		8	**inconsistent**		

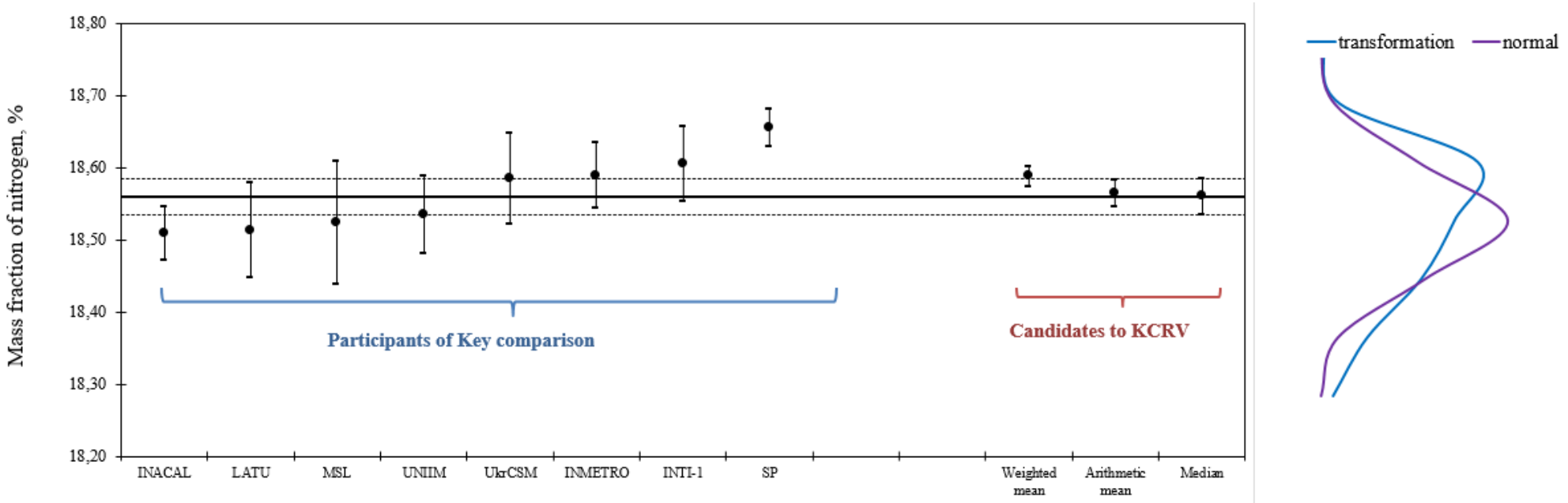

Figure 2 Error bars show standard uncertainty. The solid and dashed horizontal lines are the **median** and upper and low limits of the corresponding standard uncertainty, respectively.

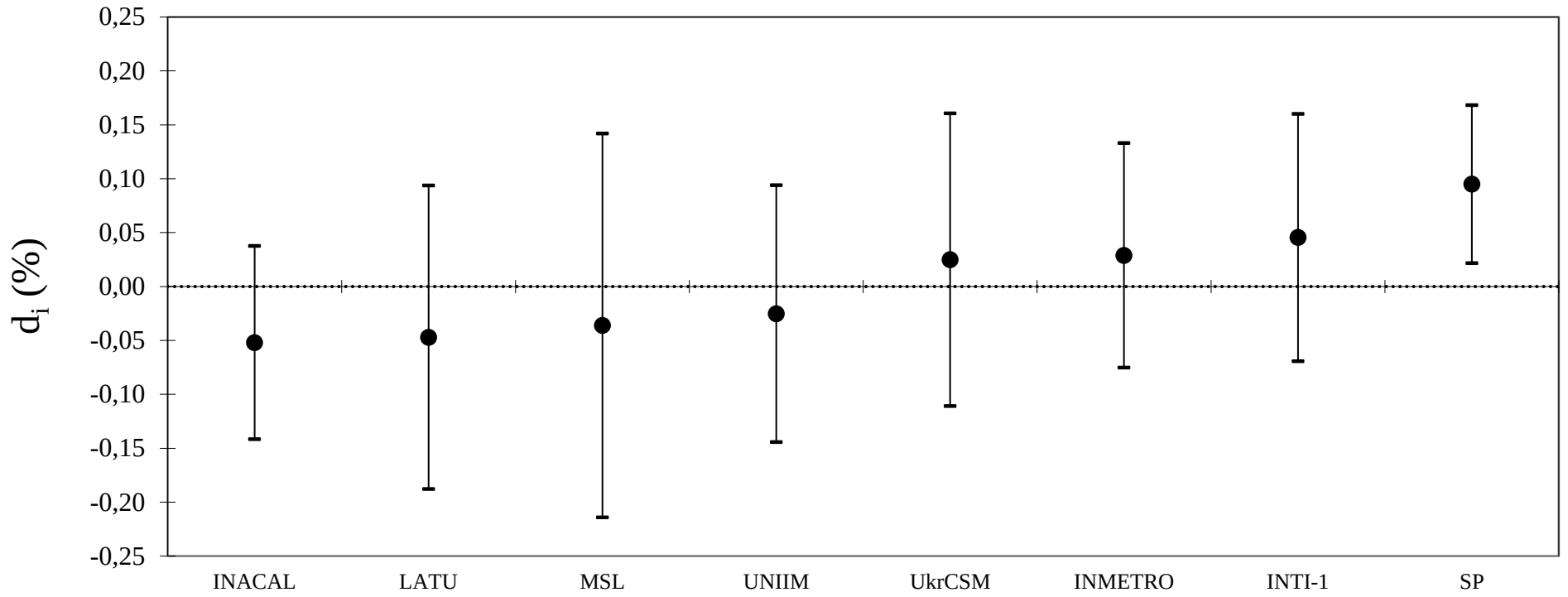

Figure 3 Degrees of equivalence d_i and expanded uncertainty $U（d_i）$ (k=2)

7.4 Discussion

Taking into account the final results it's possible to say that measurement results of almost all participants are consistent between each other.

8 EQUIVALENCE STATEMENTS

The equivalence statements have been calculated according to the BIPM guideline. The degree of equivalence (and its uncertainty) between a NMI result and the KCRV is calculated according to the following equations:

$$d_i = x_i - x_{ref}\,, \tag{16}$$

$$U\left(d_i\right) = 2\sqrt{\left(u^2\left(x_i\right) + u^2\left(x_{ref}\right)\right)}\,, \tag{17}$$

where d_i is the degree of equivalence between the NMI result x_i and the KCRV x_{ref}, and U (d_i) is the expanded uncertainty *(k = 2)* of the d_i calculated by combining the standard uncertainty $u(d_i)$ of the NMI result *xi* and the standard uncertainty $u\ x_{ref}$ of the KCRV x_{ref} (it is supposed that $\mathrm{cov}\left(x_i, x_{ref}\right)$ is ineligible). The equivalence statements for CCQM-K130 are given in Table 10 and Figures 2, 3.

9 CONCLUSIONS

The Median is proposed for the KCRV. The use of median are agreed by all participants. This Key comparison can be used in order to support calibration and measurement capabilities in determination of nitrogen mass fraction in glycine and other aminoacids with nitrogen in amino group (during decomposition by the Kjeldahl method). This Key comparison can't be used in order to support calibration and measurement capabilities in determination of nitrogen mass fraction in compounds with nitrogen in other forms, where additional proof of applicability is necessary.

10 ACKNOWLEDGEMENTS

UNIM gratefully acknowledges the help and collaboration from LATU, MSL, UNIIM, UkrCSM, INMETRO, INTI-1, SP, INACAL.

11 REFERENCES

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2. CCQM Guidance note: Estimation of a consensus KCRV and associated Degrees of Equivalence. Version: 10.

Appendix A – Technical Protocol

CCQM-K130/ CCQM-P166

Nitrogen mass fraction measurements in glycine

1. Introduction

Mass fraction of nitrogen is very important pointer because the results of these measurements are often used for determination of protein mass fraction that is an important indicator of the quality of the vast majority of food products and raw materials.

After discussing results of Pilot comparisons in the field of nitrogen mass fraction in dry milk powder on the session TC 1.8 "Physical Chemistry" COOMET it was decided to offer to carry out and Key comparisons "Nitrogen mass fraction measurements in glycine" - amino acetic acid, as the representative of high-purity substances.

The comparison is being carried out for the purpose of the confirmation of follow measurement capacity:

NMI Service Identifier	Measurement Service Category	Matrix	Measurand		Range of certified values in reference materials			Range of expanded uncertainty for certified values				Mechanism for measurement service delivery	Comments It is being recommended
			Analyte of component	Quality	From	To	Unit	From	To	Unit	Is the expanded uncertainty a relative one?		
	High purity chemicals	Glycine	nitrogen	Mass fraction	18,47	18,85	%	-	-				

2. Measurand and reporting

Mandatory measurand (for CCQM-K130) – value of mass fraction of nitrogen.

The aim of CCQM-K130 / CCQM-P166 is to measure mass fraction of nitrogen in glycine.

Each participant shall report the results for the value of of mass fraction of nitrogen. The results should be reported in mass fractions, accompanied by a full uncertainty statement (including a combined standard uncertainty and an expanded uncertainty with a coverage factor applied). In addition the report should include technical details on the measurement procedure, traceability links and uncertainty contributions.

3. Guidance values and target uncertainty

Analyte / matrix: the objects of comparisons are nitrogen mass fraction in glycine.

Sample of glycine in the range nitrogen mass fraction from 18,47 % to 18,85 % and in the range of moisture less than 0,05 % is delivered by UNIIM.

Target uncertainty is expected on the level of 0,1 %.

4. KCRVs

Processing of obtained measurement results of nitrogen mass fraction will be carried out according to the following articles:

- Cox M.G. “The evaluation of key comparison data”
- Jorg W.Muller. “Possible Advantages of a Robust Evaluation of Comparisons”

It’s offered to try different approaches: the arithmetic mean, weighted mean, median for the evaluation of reference value.

The reference is invited to try out different ways: the arithmetic mean, weighted mean, median, etc.

5. Methods of measurement

Each participant may use any suitable method(s) for the measurement of the mass fraction of nitrogen.

6. Planned time schedule

call for participants:	by end of April 2015
latest registration of participant:	by end of July 2015 (updated)
latest arrival of samples at participants:	by end of September 2015
latest report of results:	by end of February 2016
report A:	by end of May 2016
report B:	by end of July 2016

7. Samples

Sample of glycine in the range nitrogen mass fraction from 18,47 % to 18,85 % and in the range of moisture less than 0,05 % is delivered by UNIIM.

Packaging and labeling:

The material of the sample is a reagent of aminoacetic acid with a mass fraction of the basic substance of at least 99,5 %, which is a white powder, packed in dark glass vial, fitted with a sealed screw caps. Jars further sealed in waterproof bag made from polyethylene. Mass of glycine in one vial is 5 g. The package has the label with the sample name.

Storage conditions:

- Ambient temperature, °C 20±5
- Protection from the straight sun light

Storage life is 2 years.

Note: After opening the package the samples are selected for the measurement of mass fractions of nitrogen, the remaining portion of the sample material must not be stored.

Before carrying out the measurements, the package integrity is checking by means of visual observation. The package is opened and samples are selected.

8. Pilot laboratory

Laboratory of metrology of moisture measurement and certified reference material (241)

NMI's name and abbreviation

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