Development of a Metrological Support Complex for the Food Industry

Anna S. Sergeeva, Natalia L. Vostrikova, and Maria Yu. Medvedevskikh

Abstract The contribution of experts from the Laboratory for Metrological Support of Moisture Measurement and Reference Materials to improving the metrological support system for the food industry is considered. The article summarizes over 40 certified reference materials (CRMs) for the composition of food products, raw materials, and additives developed from 2008 to 2020. The development of each new CRM included the following steps: material preparation; stability and homogeneity study thereof; establishment of the certified value using state primary (GET 173) and secondary (GVET 176–1) measurement standards; primary reference measurement procedures; an interlaboratory experiment for checking the applicability of CRMs. The creation of CRMs for the composition of grain and powdered milk products accompanied GET 173 development in 2008. The approval of GVET 176–1 in 2010 allowed the certified value of nitrogen (protein) mass fraction to be added to CRMs. The successful participation of UNIIM in comparisons resulted in the publication of 6 CMC (calibration and measurement capabilities) lines for the measurement of nitrogen mass fraction in glycine, milk powder, grain, egg powder, porridge, and feed in the BIPM database. In 2016–2019, 35 CRMs for the composition of dairy and meat products, egg powder, baby food, starch products, oil crops, as well as products on their basis, were created together with the development of primary reference procedures for measuring the mass fraction of fat, crude fat, ash, and carbohydrates. In addition, CRMs for the composition of food additives (glycine, melamine, and cystine) and dairy products were developed to provide metrological support for IR analyzers, as well as a CRM for the composition of reconstituted milk to control measurement results via the enzyme-linked immunosorbent assay (ELISA). The developed CRMs are organized by the application fields of the Technical Regulations of the Customs Union and the sectors comprising the international food triangle

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model. These CRMs can be used to ensure uniform measurement of the identification and nutritional value indicators of food products and food raw materials.

Keywords Food products · Reference materials · Measurement standards · Primary reference measurement procedures · Nutritional value indicators · Identification indicator

Introduction

Data on the nutritional value of food products and raw materials are required for performing various tasks: production planning, in-process control, development of new food products, labeling, establishment of product compliance with legal requirements, development of dietary guidelines, menu planning, etc. The indicators of nutritional value include the content of proteins, fats, and carbohydrates. Several food products and raw materials are also subject to requirements for ash content. Water (moisture) content is used as an additional indicator of food quality [1]. When measuring the above indicators, it is necessary to use measurement procedures of specified accuracy while ensuring metrological traceability to a specific comparison basis [2]. Metrological traceability is achieved by using reference materials whose certified values are established using state primary standards and primary reference measurement procedures (PRMPs).

The nomenclature of food products is extensive, thus making it impossible to create a certified reference material (CRM) for each product. This factor prompted the AOAC INTERNATIONAL[1] to develop a food triangle model in the early 1990s [3, 4]. This model allows all food products to be categorized into nine sectors according to their protein, fat, and carbohydrate content; the vertices of the triangle correspond to 100% of each component. For most products allocated to one sector, it is sufficient to study one or two food matrices from that sector to validate an analytical method. This model was adopted by the National Institute of Standards and Technology (NIST,[2] USA) when developing CRMs for the composition of food products and raw materials [5–8]. In the Russian Federation, due to a lack of a unified concept for CRM development, the nomenclature of available CRMs was very limited both in terms of matrices and certified characteristics. In addition, most CRMs were not traceable to state primary standards or PRMPs.

In order to improve metrological support provided for the food industry, over 40 CRMs for the composition of food products, raw materials, and additives were developed by the specialists of the Laboratory for Metrological Support of Moisture Measurement and Reference Materials (UNIIM) from 2008 to 2020. The certified characteristics of these CRMs are traceable to state primary standards and PRMPs. The present article considers the results of CRM development at UNIIM from two

[1] AOAC INTERNATIONAL – Association of Analytical Communities, available at: https://www.aoac.org.

[2] NIST – The National Institute of Standards and Technology, available at: https://www.nist.gov/.

Table 1 Nomenclature of CRM materials

Technical regulation	CRM material
TR CU 015/2011 On safety of grain [11]	Wheat, rye, and combined feed
TR CU 021/2011 On food safety [12]	Egg powder, starch, and chocolate
TR CU 024/2011 On safety of fat-and-oil products [13]	Peanut butter, grist, and oil cake
TR CU 033/2013 On safety of milk and dairy products [14]	Milk powder (full-cream, skim), milk-based formula; freeze-dried cottage cheese and sour cream; dried cheese
TR CU 034/2013 On safety of meat and meat products [15]	Freeze-dried beef, pork, and poultry
TR CU 027/2012 On safety of certain types of specialized food products including the therapeutic and preventive dietary food [16]	Milk/milk-free instant rice, buckwheat, and cornmeal porridges for babies
TR EAEU 040/2016 On safety of fish and fish products [17]	Freeze-dried pollock fillet

angles: (1) taking into account the legislative requirements of the Russian Federation in the field of measurement uniformity assurance and technical regulation; (2) in the context of the international food triangle model.

Materials and Methods

The food product nomenclature is so extensive that it is impossible to develop a CRM for each food product. Thus, the first step was to create a nomenclature of CRM materials [9] in continuation of that presented in [10]. Firstly, several stable and homogeneous matrices representing the entire range of products covered by the Technical Regulations of the Customs Union[3] (TR CU) were selected for each effective TR CU (Table 1).

Secondly, the selected CRM materials were allocated to the sectors comprising the food triangle model [3, 4]. Finally, a list of certified characteristics (indicators of nutritional value and identification) was selected, taking into account the available standard and measurement base.

The development of each new CRM included the following stages:

– material preparation (mixing, conditioning, freeze-drying, fraction selection);
– study of long- and short-term stability, homogeneity of the CRM material taking [18] into account;

[3] Effective Technical Regulations of the Customs Union, Rosstandart, available at: https://www.rst.gov.ru/portal/gost/home/standarts/technicalregulationses.

– establishment of the certified value using state primary and secondary standards, as well as PRMPs; estimation of characterization-associated standard uncertainty of the certified value taking [19] into account;
– interlaboratory experiment conducted to verify the applicability of CRMs.

The following primary and secondary standards were used to establish certified values of reference materials at UNIIM:

– GET 173–2017 State Primary Measurement Standard for the units of mass fraction and mass (molar) concentration of water in solid and liquid substances and materials [20];
– GVET 176–1-2010 State Secondary Measurement Standard for the units of mass fraction and mass (molar) concentration of components in solid and liquid substances and materials on the basis of the volumetric titrimetric method [21], which is in turn traceable to GET 176–2019 State Primary Measurement Standard for the units of mass (molar, atomic) fraction and mass (molar) concentration of components in liquid and solid substances and materials on the basis of coulometry.[4]

In addition, the following PRMPs were developed for establishing the certified values of reference materials [22]:

– PRMP for the mass fraction of fat in food products and food raw materials M.241.01/RA.RU.311866/2018[5];
– PRMP for the mass fraction of ash in food products and food raw materials M.241.02/RA.RU.311866/2018[6] [23];
– PRMP for the mass fraction of carbohydrates in food products and food raw materials[7];
– PRMP for the mass fraction of crude fat (oil content) in oilseeds and products on their basis[8] [24].

[4] GET 176–2019 State Primary Measurement Standard for the units of mass (molar, atomic) fraction and mass (molar) concentration of components in liquid and solid substances and materials on the basis of coulometry. In: Federal Information Fund for Ensuring the Uniformity of Measurements. https://fgis.gost.ru/fundmetrology/registry/12/items/1382712.

[5] State Primary Reference Measurement Procedure for the mass fraction of fat in food products and food raw materials M.241.01/RA.RU.311866/2018 (FR.PR1.31.2019.00001). In: Federal Information Fund for Ensuring the Uniformity of Measurements. https://fgis.gost.ru/fundmetrology/registry/6/items/595556.

[6] State Primary Reference Measurement Procedure for the mass fraction of ash in food products and food raw materials M.241.02/RA.RU.311866/2018 (FR.PR1.31.2019.00002). In: Federal Information Fund for Ensuring the Uniformity of Measurements. https://fgis.gost.ru/fundmetrology/registry/6/items/595557.

[7] State Primary Reference Measurement Procedure for the mass fraction of carbohydrates in food products and food raw materials (FR.PR1.31.2019.00005). In: Federal Information Fund for Ensuring the Uniformity of Measurements. https://fgis.gost.ru/fundmetrology/registry/6/items/1057023.

[8] State Primary Reference Measurement Procedure for the mass fraction of crude fat (oil content) in oilseeds and products on their basis (FR.PR1.31.2019.00009). In: Federal Information Fund for

Results and Discussion

The first reference materials developed for the food industry were CRMs for the composition of grain, its products, and powdered milk products (Table 2). These CRMs were created as part of activities involving the transfer of water (moisture) content units from GET 173–2008 State Primary Standard for the units of mass fraction and mass concentration of moisture in solid substances and materials [25]. GET 173 developed by the laboratory in 2006–2008 was further improved in 2013 and 2017 [20]. During CRM development, the laboratory applied its accumulated experience in creating and using moisture measurement procedures, estimating measurement uncertainty, and preparing homogeneous and stable material for interlaboratory comparative studies and international comparisons [26, 27].

Further activities of the laboratory were aimed at improving GET 173, developing and certifying measurement procedures, assessing the CRMs for the mass fraction of moisture in terms of their applicability to food quality control [28], as well as providing metrological support for the measurement of nitrogen (protein) mass fraction. In 2010, GVET 176–1-2010 State Secondary Measurement Standards for the units of mass fraction and mass (molar) concentration of components in solid and liquid substances and materials on the basis of the volumetric titrimetric method was approved [21]. Furthermore, as part of GVET 176–1-2010 development, a CRM for the composition of grain and its products (GSO 9734–2010) and a CRM for the composition of milk powder (GSO 9563–2010) were created (Table 3).

GSO 9563–2010 was used in international comparisons within the COOMET project 508/RU/10 (Fig. 1). Following comparisons, GSO 9563–2010 was recognized as an interstate CRM (MSO 1781:2012) to be applied in the territory of the member-states of the Euro-Asian Council for Standardization, Metrology, and Certification.

The mass fraction of nitrogen (protein) in GSO 9563–2010 and GSO 9734–2010 was determined using the Kjeldahl method recognized as an umpire method by

Table 2 CRM having a certified characteristic of moisture mass fraction

CRM name	Range, %	Error, ± Δ, %
GSO 8989–2008 CRM of 1st-category for the mass fraction of moisture in grain	7.0–18.0	0.1
GSO 8990–2008 CRM of 2nd-category for the mass fraction of moisture in grain	7.0–25.0	0.2–0.3
GSO 9564–2010 CRM for the mass fraction of moisture in grain products	7.0–16.0	0.2
GSO 10148–2012 CRM for the mass fraction of moisture in powdered milk products	2.0–10.0	0.08–0.12

Ensuring the Uniformity of Measurements. https://fgis.gost.ru/fundmetrology/registry/6/items/1057069.

Table 3 CRMs having the mass fraction of moisture, nitrogen, and protein as their certified characteristics

CRM name	Certified characteristics	Range, %	Error, ± Δ, %
GSO 9563–2010 CRM for the composition of milk powder (ASM-1) [21]	moisture mass fraction	2.00 – 5.00	0.15
	nitrogen mass fraction*	1.00 – 7.00	0.03
	protein mass fraction*	6.0 – 45.0	0.2
GSO 9734–2010 CRM for the composition of grain and its products	moisture mass fraction	7.0 – 25.0	0.2 – 0.3
	nitrogen mass fraction*	1.00 – 8.00	0.04 – 0.06
	protein mass fraction*	5.00 – 50.00	0.25 – 0.35

* Values are given in terms of dry matter

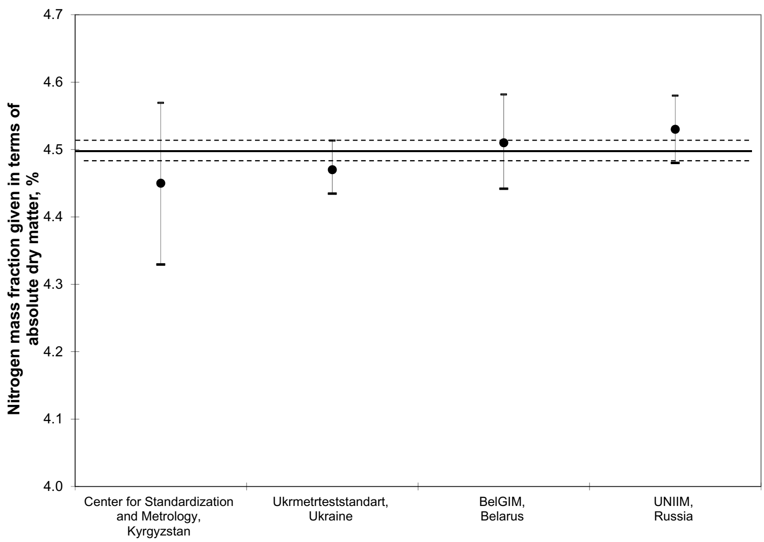

Fig. 1 Results of the COOMET 508/RU/10 comparisons

several international organizations [29]. However, since this method is very labor-intensive and time-consuming, testing laboratories increasingly use rapid measurement methods: Dumas combustion method and IR spectroscopy [30, 31]. These methods require the construction of calibration characteristics using pure substances. In order to provide metrological support for express analyzers, UNIIM developed CRMs for the composition of glycine [32], melamine [33], and cystine (Table 4).

In the following years, work was under way on recognizing the measurement capabilities of UNIIM in the field of nitrogen content measurement at the international level. The following comparisons were carried out: CCQM-K130&P166 key comparisons for determining the mass fraction of nitrogen in pure substance (glycine) [34], as well as pilot and key comparisons for determining the mass fraction of nitrogen in milk powder (CCQM-P167 and CCQM-K149) [35]. The results

Table 4 CRMs for the composition of glycine, melamine, and cystine

CRM name	Certified characteristics	Range, %	Error, ± Δ, %
GSO 10272–2013 CRM for the composition of glycine [32]	nitrogen mass fraction	18.47–18.66	1.0 rel%
	mass fraction of the main substance	99.0–100.0	1.0 rel%
GSO 10825–2016 CRM for the composition of melamine [33]	nitrogen mass fraction	63.30–66.64	1.5 rel%
	mass fraction of the main substance	95.0–100.0	1.5 rel%
GSO 11337–2019 CRM for the composition of cystine	carbon mass fraction	29.8–30.1	0.3 abs%
	hydrogen mass fraction	4.9–5.1	0.2 abs%
	nitrogen mass fraction	11.5–11.8	0.2 abs%
	sulfur mass fraction	26.5–26.8	0.3 abs%

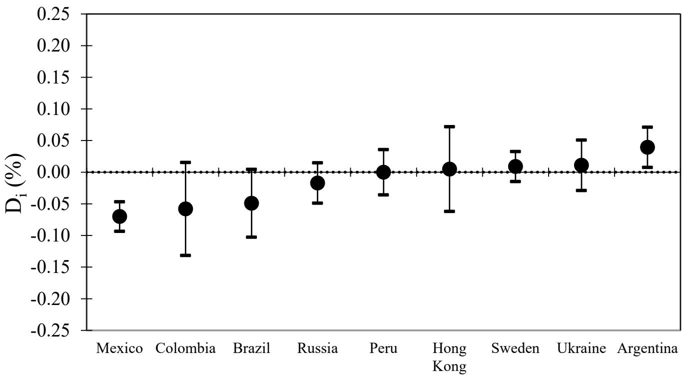

Fig. 2 Results of the CCQM-K149 comparisons determining the mass fraction of nitrogen in milk powder [35]

of the CCQM-K149 key comparisons using GSO 9563–2010 are shown in Fig. 2. The successful participation of UNIIM in comparisons led to the publication of 6 CMC[9] lines for the measurement of nitrogen mass fraction in glycine, milk powder, grain, egg powder, porridge, and feed in the BIPM[10] database.

In 2016–2019, the laboratory carried out work on the development, approval, and certification of PRMPs to establish the operationally determined indicators of nutritional value: mass fractions of fat, crude fat (oil content), carbohydrates, and ash. As a result, thirty-five CRMs for the composition of food products and food raw materials were developed using PRMPs: dairy and meat products, egg powder, baby food, oilseeds and products on their basis, as well as starch products [36]. Here, it should be noted that the CRM development was carried out in close cooperation

[9] CMC – calibration and measurement capabilities.

[10] BIPM - International Bureau of Weights and Measures. https://www.bipm.org.

with other organizations, specifically food industry specialists. For instance, V. M. Gorbatov Federal Research Center for Food Systems (RAS, Moscow) was the co-developer of GSO 11274–2019/GSO 11276–2019 for the composition of freeze-dried meat products [37]. As an example, Table 5 provides the metrological characteristics of GSO 11086–2018/GSO 11091–2018 CRMs for the composition of powdered milk products (ASM-2 CRM set UNIIM) and GSO 11399–2019 CRM for the composition of milk powder (ASM-3 CRM UNIIM).

The applicability of GSO 11086–2018/GSO 11091–2018 was confirmed by the results of interlaboratory comparisons (ILCs) 241-MP3 for determining the quality indicators of milk and dairy products. Table 6 summarizes the results of the ILC round 241-MP3-3, demonstrating the applicability of these CRMs to control the accuracy of measured moisture, protein, and fat mass fraction values.

Table 5 Metrological characteristics of GSO 11086–2018/GSO 11091–2018 and GSO 11399–2019

CRM name	Certified characteristics	Range, %	Error, ± Δ, %
GSO 11086–2018/GSO 11091–2018 CRMs for the composition of powdered milk products (ASM-2 CRM set UNIIM)	moisture mass fraction	2.00–10.00	0.08–0.12
	nitrogen mass fraction*	0.20–11.00	0.03
	protein mass fraction*	1.2–70.0	0.2
	fat mass fraction*	0.10–80.00	0.10–0.25
GSO 11399–2019 CRM for the composition of milk powder (ASM-3 CRM UNIIM)	lactose mass fraction*	30.0–55.0	5.0
	mass fraction of carbohydrates*	30.0–70.0	0.4

* Values are expressed in terms of dry matter

Table 6 Summarized results of the ILC round 241-MP3-3/2018 *determination of quality indicators of milk and dairy products*

CRM code	Parameter to be determined	Percentage of laboratories reporting satisfactory results	Percentage of laboratories reporting indeterminate results	Percentage of laboratories reporting unsatisfactory results
MP3(1)	moisture mass fraction	100	0	0
	protein mass fraction	84	11	5
	fat mass fraction	78	18	4
MP3(2)	moisture mass fraction	93	7	0
	protein mass fraction	90	0	10
	fat mass fraction	77	0	23

In 2020, CRMs for the composition of milk and light cream were developed to provide metrological support for rapid IR analyzers. The certified mass fraction values of dry matter, protein, fat, and lactose were established using GET 173–2017 and GVET 176–1-2010, as well as certified measurement procedures developed to expand the scope PRMPs (FR.PR1.31.2019.00001 and FR.PR1.31.2019.00005). Additional measurements were performed at the testing laboratory of the Ural State University of Economics (Yekaterinburg) employing standardized measurement procedures [38].

A new area of focus for the laboratory consists in providing metrological support for the enzyme-linked immunosorbent assay (ELISA). Thus, GSO 11168–2018 CRM for the composition of reconstituted milk (RM CRM UNIIM) was developed in collaboration with Chema LLC (Moscow) in 2018 [39]. The certified values of this CRM include the mass fraction of nitrogen and the mass concentration of milk powder.

To date, the Laboratory for Metrological Support of Moisture Measurement and Reference Materials produces over 40 CRMs for the composition of food products, food raw materials, and food additives; these CRMs can be used to provide metrological support for measuring the identification indicators of food products in order to ascertain their compliance with the TR CU requirements (Fig. 3).

The produced CRMs cover seven of the nine sectors comprising the international food triangle model [3, 4] (Fig. 4). Thus, they can be used to validate analytical methods for food products and food raw materials from these sectors.

In the near future, the laboratory plans to develop CRMs for the remaining sectors of the food triangle and product groups specified in the Technical Regulations: CRMs for the composition of freeze-dried fish, chocolate, peanuts, soy flour, and sugar. The

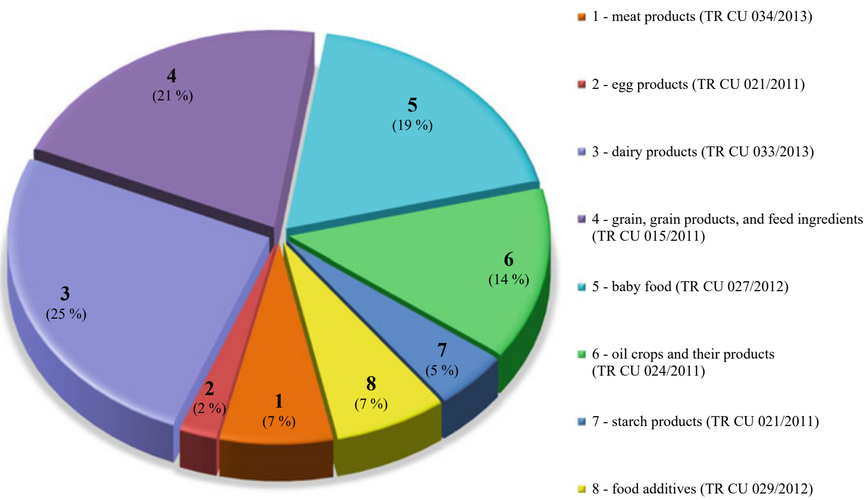

Fig. 3 Distribution of produced CRMs for the composition of food products depending on items governed by TR CU

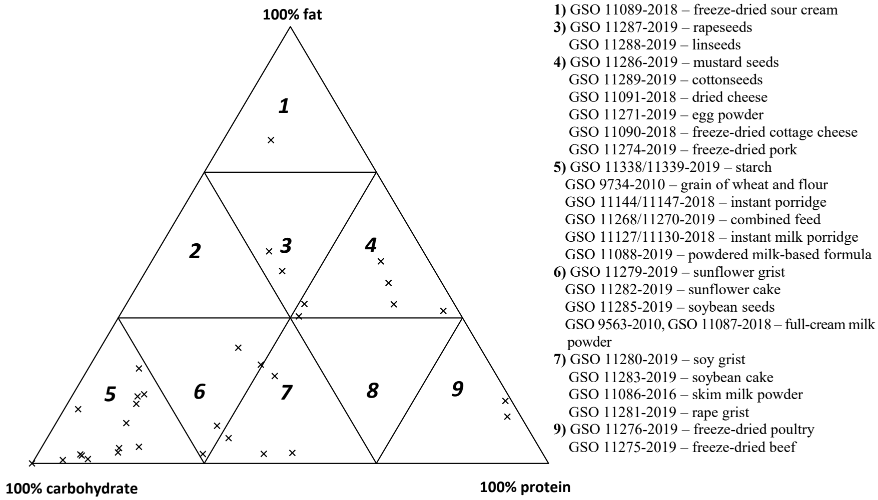

Fig. 4 Distribution of the produced CRMs among the sectors comprising the food triangle

next step would be to expand the certified characteristics of the developed CRMs by adding information on carbohydrate, fatty acid, and amino acid compositions.

Conclusion

In 2008–2020, the specialists of the Laboratory for Metrological Support of Moisture Measurement and Reference Materials (UNIIM) developed a complex designed to provide metrological support for measuring the nutritional value of food products, including state standards for measurement units (GET 173–2017 and GVET 176–1-2010), PRMPs, and over 40 CRMs for the composition of food products and food raw materials.

The developed CRMs ensure uniformity in measuring the identification and nutritional value indicators of food products and food raw materials to ascertain the compliance of products with technical regulation laws, as well as the consistency between measurement results obtained in the territory of the Russian Federation and internationally. Furthermore, the CRM nomenclature developed taking into account the international food triangle model helps to optimize the metrological support system of the food industry by using a limited number of matrices for a wide range of analyzed objects.

Author Contributions Anna S. Sergeeva: definition of the idea and methodology of the article; collection and analysis of literary data (including in foreign languages); preparation of the first draft of the article; writing, formatting, and revision of the text.

Natalia L. Vostrikova: supervision of the experimental studies; critical analysis of the article.

Maria Yu. Medvedevskikh: concept development; study initiation; definition of the idea and methodology of the article; supervision of the experimental studies; critical analysis of the article.

Conflict of Interest The article was prepared on the basis of a report presented at the IV International Scientific Conference "Reference Materials in Measurement and Technology" (St. Petersburg, December 1–3, 2020). The article was admitted for publication after the abstract was revised, the article was formalized and the review procedure was carried out.

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