Advances of the Research Department on Physicochemical and Electrical Measurements of FSUE "VNIIFTRI"

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Abstract. It is hard to imagine the development of the country's economy without constant development and improvement of its base of standards in order to ensure the uniformity of measurements both in a particular country and at the global level. For this purpose, there are four state metrological institutes with branches in Russia. The Research Department on Physicochemical and Electrical Measurements is a structural subdivision of The Federal State Unitary Enterprise «Russian Metrological Institute of Technical Physics and Radio Engineering». The activity of the Research Department is related to the measurement of pH, pX, inorganic components in liquids, with measurements of the parameters of particles in various matrices, gases dissolved in a liquid, such as oxygen, hydrogen, carbon dioxide, measurements of the parameters of air ions, etc. Scientists of the Research Department conducting research to develop the certified reference materials in order to establish the measurement traceability to the Russian Primary Standards, to the calibration and measurement capabilities of metrology institutes at the database of key comparisons of the International Bureau of Weights and Measures.

The Research Department is constantly working to expand its capabilities for metrological support of industry and scientific research in the Russian Federation on the topics assigned to the institute, is actively working to improve its reference base, participates in technical committees and working groups of regional and international organizations related to metrology. Takes an active part in international and Russian conferences and seminars, round tables and discussions.

This paper describes the achievements of the Research Department over the past two years.

As in all developed countries, Russia also faces the task of achieving the main priorities of the country's national development - improving the quality of life of the population and creating comfortable living conditions for them. This can be achieved only with a constant process of development and improvement of the scientific and applied sphere, health care, by achieving a safe environment, etc. According to scientists, in recent years, Russia has entered “in the group of leaders of the global scientific space - 5th
in the number of researchers (in full-time equivalent), 9th - in terms of internal expenditures on research and development (R&D)” [1]. According to them, “In the period from 2013 to 2019, the share of articles by domestic scientists indexed in the international database Web of Science increased from 2.3 to 3.0%.

In terms of the number of articles in areas determined by the priorities of scientific and technological development, in publications indexed in international databases, Russia has risen from 13th to 9th place in the world. Within the framework of the National Project “Science”, new instruments of state policy are being deployed, including the creation of scientific and scientific and educational centers of the world level, centers of competence of the National Technology Initiative, updating 50% of the instrument base of leading organizations performing research and development ”[1].

At the same time, it is difficult to imagine the development of the country’s economy without constant development and improvement of its reference base in order to ensure the uniformity of measurements both in a particular country and at the global level. For this purpose, there are four state metrological institutes with branches in Russia [2].

The Research Department of Physicochemical and Electrical Measurements (NIO-6) is a structural subdivision of The Federal State Unitary Enterprise « All-Russian Scientific Research Institute of Physicotechnical and Radio Engineering Measurements» (VNIIFTRI) [3]. The fields of its activity are related to measurements pH, pX, inorganic components in liquids, with measurements of the parameters of particles in various matrices, gases dissolved in a liquid, such as oxygen, hydrogen, carbon dioxide, measurements of the parameters of air ions, etc. Scientists of the NIO-6 conduct research on the development of state reference materials (GSO) in order to ensure traceability of measurements in the country directly to state primary standard of Russia, calibration and measurement capabilities (CMC) and the key comparison database (KCDB) of the International Bureau of Weights and Measures (BIPM).

The NIO-6 is constantly working to expand its capabilities for metrological support of industry and scientific research in Russia in the areas assigned to VNIIFTRI, is actively improving its reference base, participating in the work of technical committees and working groups of regional and international organizations related to metrology. Actively takes part in international and Russian conferences and seminars, round tables and discussions.

So, in December 2019, according to the successful results of the improvement, the state primary standard for the activity of hydrogen ions (pH) in aqueous solutions - GET 54 was approved, about what the corresponding entry was made in the Rosstandart database [4].

Metrological characteristics of the improved standard are:

- in the range of (1 - 12) pH of aqueous solutions in the temperature interval of (0-90) °C, absolute expanded uncertainty – \( U_{abs} = 0.004 \), with the coverage factor \( k=2 \);

- in the range of (0.01 - 1) pH of aqueous solutions in the temperature interval of (0-90) °C, absolute expanded uncertainty – \( U_{abs} = 0.117 \), with the coverage factor \( k=2 \).

One more calibration and measurement capability (CMC) was approved in the database of BIPM [5] for (10.01 - 10.02) pH with an absolute extended uncertainty - \( U_{abs} = 3.9E-3 \), with the coverage factor \( k=2 \), according to the results of the participation of the electrochemical laboratory of the NIO-6 in key comparison organized by the Working Group on Electrochemical Analysis (EAWG) of the BIPM Consultative Committee for Amount of Substance: Metrology in Chemistry and Biology (CCQM) CCQM- K18.2016 Key comparison on carbonate buffer [6].

In addition, we would like to note that the NIO-6 technical scientists became co-coordinators with NMJJ (Japan) in the such high level comparisons as the CCQM-K19.2018 and CCQM-K19.1.2018 Key Comparison on Borate Buffer, pH ~ 9.2 at the first time. Comparisons have not been completed yet.

Also, over the past two years, the laboratory staff have developed special electrochemical products (Figure 1).
Fig. 1. The new products of the VNIIFTRI for electrochemical laboratories:

a. electrode cleaning solution – Desolvator 630-1;
b. electrode storage solution - Strouge-630-2M and Strouge-630-3M;
c. electrolytes for filling glass electrodes – Glasslife 630-3M, Glasswork-630-3M and Glassnitro-630-1M.

And also, taking into account the expediency of combining electrochemical measurements in the same laboratory, the state primary standard of the unit of specific electrical conductivity of liquids in the range from 0.001 to 50 S / m - GET 132 has been handed from another NIO of the VNIIFTRI to the electrochemical laboratory of the NIO-6.

In 2020, based on the successful results of improvement, the state primary standard of units of dispersed parameters of aerosols, suspensions and powdery materials - GET 163 of another laboratory of the NIO-6 has been approved, about what a corresponding entry was made in the Rosstandart database [7]. The scientists of the laboratory studies particle measurements in various media.

According to the approved new metrological characteristics of the GET 163:

1. measurement capabilities for the last particle size measurement range from 0.03 μm to 2000 μm, relative expanded uncertainty – U_r = 2.4, with the coverage factor k=2, were expanded to 0.001 μm, at that in the range (0.001 - 0.03) μm, the relative expanded uncertainty - U, % = 2.7...3.8, with the coverage factor k=2;

2. measurement capabilities for two another particle parameters were added:
   - mass concentration of aerosol particles in the ranges (1 - 2000) mg / m³, relative expanded uncertainty - U_r = 1.3, and (2000 - 10000) mg / m³, relative expanded uncertainty - U, % = 1.3 ... 2.9, with the coverage factor k = 2;
   - zeta potential of particles in the range (-150 - +150) mV, relative expanded uncertainty - U, % = 4.4 ... 4.8.

At present, the staff of this laboratory intends to take part in a key comparison within the framework of the BIPM in the field of measuring the countable concentration of particles in liquids.

Over the past two years, the staff of the laboratory of mass spectrometric analysis is expanded their calibration and measurement cabality. Four new claimed CMC for the determination of elements in leather products in the range (10-100) mg/kg were confirmed and published in the KCDB BIPM [5]:

- As (arsenic), U_{abs} = 1.7;
- Pb (lead), U_{abs} = 0.4 – 3;
- Co (cobalt), U_{abs} = 0.3 – 2;
- Cd (cadmium), U_{abs} = 0.35 – 2.5,

with the coverage factor k = 2.
The successful participation in various international comparisons of scientists of the mass spectrometric analysis laboratory, GSOs are being developed in order to ensure the traceability of the measurement results of analytical laboratories to the CMCs of the BIPM database [5]. The developed GSOs will not only make up for the absence of the same type of imported certified reference materials (CRM) in the Russian market, mostly associated with the sanctions adopted against Russia, but will also be economically more advantageous for Russian laboratories.

So, over the past period, the mono element the reference materials of the mass fraction of Cd (cadmium), Co (cobalt), Li (lithium), Pb (lead) ions in an aqueous solution, developed in the laboratory, approved as GSOs, and some new reference materials are at the stage of approval as GSOs (Figure 2).

![Fig. 2 New state reference materials of the VNIIFTRI](image)

a. multi-element reference material (6 metal ions in aqueous solution); b. reference material of Ba (barium) ions in aqueous solution; c. reference material of Zn (zinc) ions in aqueous solution.

Four mono-element GSOs of the NIO-6 were awarded the quality mark according to the results of the competition at the annual exhibition Analytica-Expo-2021 in Moscow [8].

It should be noted that in March 2021 the Quality Managment System (QMS) of VNIIFTRI had the peer review by COOMET experts. The Confirmation of recognition - QSF-R78 of the QMS in accordance with the Standard ISO 17025:2017 [9] for 7 Metrological areas, including Physical Chemistry, have been given to VNIIFTRI.

Some facts about the international activities of the NIO-6 over the last two years:

- The NIO-6 scientists took part in 5 key and 1 pilot comparisons conducted within the framework of the CCQM;
- Revision of the recommendation of the International Organization of Legal Metrology OIML / R54 "pH scale for aqueous solutions" is being finalized;
- VNIIFTRI has become a member of the International Organization Cooperation on International Traceability in Analytical Chemistry (CITAC) represented by Deputy Head of NIO-6 Dr. Narine Oganyan since 2019. In 2021 she became the CITAC Web Editor and a member of the CITAC Executive Committee [10];
- At the beginning of 2021, Russia changed its observer status to a full member of the technical committee ISO/TK24/PC4 "Characterization of Particles"[11] of the International Organization for Standardization (ISO). VNIIFTRI was appointed as the authorized representative of Russia in the person of the Head of the NIO-6 Dr. Vladimir Dobrovolskiy.

Thus, over the past two years, thanks to metrological research at the NIO-6, the reference base has been improved and the measuring capabilities of VNIIFTRI have been expanded. The NIO-6 scientists took an active part in technical committees and working groups of regional and international organizations, in various metrological events.
References

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