PROTOCOL
of the meeting of COOMET Technical Committee 1.8 "Physics-Chemistry"

REPORT ON ACTIVITIES
from June 2016 to May 2017
WORK PROGRAMME
for June 2017 – May 2018

May 23-24, 2017

Meeting of COOMET Technical Committee 1.8 "Physics-Chemistry" was held in D.I. Mendeleyev Institute for Metrology (St. Petersburg, Russia) on May 23-24, 2017.

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The meeting was held in accordance with the Work Programme of Technical Committee 1.8 "Physics-Chemistry", approved by its participants.

I. In the course of work of Technical Committee 1.8 "Physics-Chemistry" the following reports were made

1. Opening speech of the Chairman of TC 1.8 "Physics-Chemistry" Leonid A. Konopelko (hereinafter referred to as the TC Chairman).

2. Report of the TC Chairman on results of TC work in the period from June 2016 to May 2017, including the ongoing and new CCQM and COOMET comparisons, and specifically the following **ongoing works** (with VNIIM as the Coordinator):

   622/RU/13 Supplementary comparison "C_3-C_5 components in mixtures of liquefied hydrocarbons"; participants: VNIIM (Russia), BelGIM (Belarus), "Ukrmetrteststandart" (Ukraine);

   649/RU/14 Pilot comparison "Study in the field of measuring of formed elements of blood: erythrocytes (RBC), leukocytes (WBC)"; participants: Laboratories of Kyrgyzstan;

   664/RU/15 Key comparison "Ambient air: Carbon monoxide (CO) in nitrogen 5 μmol/mol"; participants: KazInMetr (Kazakhstan), BelGIM (Belarus);

   678/RU/15 Key comparison "Propane in Nitrogen C_3H_8/N_2 1000 μmol/mol"; participants: KazInMetr (Kazakhstan), BelGIM (Belarus), "Ukrmetrteststandart" (Ukraine), VNIIM (Russia);

   611/RU/13 "Melamine in milk powder"; participants: UNIIM (Russia), "Ukrmetrteststandart" (Ukraine);

   654/RU/14 Pilot comparison "Cholesterol in human serum"; participants: Center for Standardization and Metrology of the Ministry of Economy of the Kyrgyz Republic, VNIIOFI (Russia);

   691/RU/16 Pilot comparison "Determination of polychlorinated dibenzo-p-dioxins and dibenzofurans in fat matrices (pork fat)"; participants: "Ukrmetrteststandart" (Ukraine), UNIIM (Russia);

   708/RU/16 Pilot comparison "Purity of anthracene (100 minus admixtures)"; participants: "Ukrmetrteststandart" (Ukraine), UNIIM (Russia).

3. Report of the Deputy Chairman Yuri A. Kustikov on his participation in the Plenary Session of Consultative Committee for Amount of Substance (CCQM), and report on COOMET TC 1.8 activities in BIPM Working Groups.

4. Report of Olga V. Efremova (VNIIM) on participation in the Meeting on Key Comparisons and Measuring Capabilities. The report contained information on claimed Calibration and Measurement

Capabilities (CMC) of the TC Member countries in the field of Physics-Chemistry. All COOMET CMCs were reviewed by CCQM experts and approved.

Additionally, a report was made on participation in the meeting of Gas Analysis Working Group (GAWG, April, Paris, 2017) and on the status of the ongoing and scheduled comparisons in 2016-2017 on the following subjects:

Ongoing comparisons:

- CCQM-K111 "Propane in nitrogen"; Coordinator: VSL (the Netherlands), participant from Russia: VNIIM;
- CCQM-K119 "Liquified Petroleum Gas"; Coordinator: NPL (Great Britain), participant from Russia: VNIIM;
- CCQM-K112 "Composition of Biogas"; Coordinator: VSL (the Netherlands), participant from Russia: VNIIM;
- CCQM-K90 "Formaldehyde in Nitrogen: 2 ppm"; Coordinator: BIPM, participant from Russia: VNIIM;
- CCQM-K116 "Water vapour in Nitrogen: 10 μmol/mol"; Coordinator: NPL (Great Britain), participant from Russia: VNIIM;
- CCQM-K117 "NH_3 in Nitrogen: 10-30 μmol/mol"; Coordinator: NPL (Great Britain) and NIST (USA), participant from Russia: VNIIM, distribution of materials: January 2017;
- CCQM-K118 "Natural gas"; Coordinator: BAM (Germany) and VSL (the Netherlands), participant from Russia: VNIIM, distribution of materials: January 2017;
- CCQM-K120 "Ambient CO_2"; Coordinator: BIPM, participant from Russia: VNIIM, distribution of materials: December 2016;
- CCQM-K137 "NO in Nitrogen: 30 μmol/mol to 70 μmol/mol (gravimetric comparisons)"; Coordinator: BIPM, participant from Russia: VNIIM, distribution of materials: March 2017;

Scheduled comparisons:

- CCQM-K150/P159 "Number concentration and charge of particles"; Coordinator: NPL (Great Britain), comparison starts in November, 2017;
- CCQM-K41.2017 "H_2S in Nitrogen: 10 μmol/mol"; repeat comparisons; Coordinator: KRISS (Korea), comparison starts in September, 2017;
- CCQM-K74.2018 "NO_2 in Nitrogen: 10 μmol/mol"; repeat comparisons; Coordinator: BIPM, comparison starts in 2018;
- CCQM-K10.2018 "BTEX 5 ppb"; Coordinator: NIST, comparison starts in 2018.

5. Report of Anatoliy I. Krylov (VNIIM) on participation of his coworkers M.V. Belyakov and A.Yu. Mikheeva in the meetings of Working Groups on Inorganic and Organic Analysis (IAWG and OAWG, April 2017, Paris), where the status of the ongoing and scheduled comparisons in 2016-2017 on the following subjects was discussed:

Ongoing comparisons in the field of inorganic analysis:

- CCQM-K123 "Trace elements and chrome compounds in biodiesel fuel"; Coordinator: NMIJ (Japan);
- CCQM-P160 "Measurement of isotope ratios and molar mass measurements of Si isotopes in isotopically enriched silicon"; Coordinator: PTB (Germany);

- CCQM-K139 “Comparison of the measurement of clinical markers in human serum”; Coordinator: HAS, participant from Russia: VNIIOFI;
- CCQM-K124/P158 “Trace elements and chrome compounds in drinking water”; Coordinator: NMIJ (Japan), participant from Russia: VNIIM;
- CCQM-K125/P159 “Iodine and other elements in infant formula”; Coordinator: GLHK (Hong Kong); participant from Russia: VNIIM;
- CCQM-K127 and CCQM-P162 “Toxic and trace elements in soils”; Coordinators: CENAM (Mexico) and JSI (Slovenia); participant from Russia: VNIIM;
- CCQM-K128 “Heavy metals and organo-tin (tributyltin) in leather powder: Pb, Cd, Hg, As, Sb, Co, Cu, Ni in the concentrations range from 30 mg/kg to 200 mg/kg and tributyltin from 100 mg/kg to 300 mg/kg; Coordinator: NIM; participants from Russia: VNIIFTRI, UNIIM (heavy metals) and VNIIM (tributyltin);
- CCQM-K140 “Carbon stable isotope (^{13}C) ratio delta values in honey”; Coordinator: LGC (Great Britain); participant from Russia: VNIIM;

Ongoing comparisons in the field of inorganic analysis:

- CCQM-K95.1 “PAHs in tea”; Coordinator: NIST (USA);
- CCQM-K104 “Purity assessment of high purity organic materials: Avermectin”; Coordinator: NIM (China);
- CCQM-K126/P161 “Carbamazepine in water”; Coordinator: GLHK (Hong Kong);
- CCQM-K115/P55.2 “Human C peptide. Peptide purity determination”; Coordinator: NIST (USA);
- CCQM-K132/P169 “Vitamin D in serum”; Coordinator: NIST (USA);
- CCQM-K142/P179 “Urea and Uric Acid in Human Serum or Plasma”; Coordinators: HSA (Singapore) and NIST (USA);
- CCQM-K55d/P117d “Mass fraction assignment of Folic acid in a high purity material”; Coordinator: BIPM; participant from Russia: VNIIM;
- CCQM-K102 “Polybrominated diphenyl ethers (PBDEs) in sediment”; Coordinator: IRMM (Belgium); participant from Russia: VNIIM;
- CCQM-K109/P148 “Urea and uric acid in human serum – high-polarity analytes in biological matrix”; Coordinator: HSA (Singapore); participant from Russia: VNIIM;
- CCQM-K131 “Low-Polarity Analytes in a Multicomponent Organic Solution: Polycyclic Aromatic Hydrocarbons (PAHs) in Acetonitrile”; Coordinator: NIST (USA); participant from Russia: VNIIM;
- CCQM-K138/P174 “Aflatoxins in fried fig”; Coordinator: UME (Turkey); participant from Russia: VNIIM;
- CCQM-P150.1 “QNMR purity study: piributicarb”; Coordinator: NMIJ (Japan); participant from Russia: VNIIM;
- CCQM-K78.a/P122.a “Mass fraction assignment of organic analytes in a multi-component aqueous calibration solution”; 2016-2017, Coordinator: BIPM; participant from Russia: VNIIM;
- CCQM-K141/P178 “Antibiotics in bovine tissue”; 2016-2017, Coordinator: NRC (Canada); participant from Russia: VNIIM;

Scheduled comparisons in the field of inorganic analysis:

- CCQM-K148/P187.a “Purity of materials: Bisphenol-A”; Coordinator: BIPM; participant from Russia: VNIIM;
- CCQM-K133/P170 “Phthalates in PVC”; Coordinator: NIM (China);
- CCQM-P164 “HGH quantification in serum”; Coordinator: PTB (Germany);
- CCQM-K147/P186 “Vitamins in infant formula”; Coordinators: NIST (USA) and CENAM (Mexico);
- CCQM-K146/P185 “PAHs in olive oil”; Coordinator: NIM (China).

6. Report of Maksim S. Vonskiy and Tatyana V. Kulyabina on participation in the meeting of the Working Groups on Nucleic Acid Analysis (NAWG) and the Working Group on Cell Analysis (CAWG) in April 2017, Paris, where the status of the ongoing and scheduled comparisons in 2016-2017 on the following subjects was discussed:

Ongoing comparisons:

- CCQM-K86.b Key comparison “Relative quantification of Bt63 in GM rice matrix sample”;
- CCQM-P154 Pilot comparison “Absolute quantification of DNA”;
- CCQM-P155 Pilot comparison “Multiple cancer cell biomarker measurement”;
- CCQM-P123 Pilot comparison “measurement of the number and geometrical properties of cells adhered to a solid substrate”;
- CCQM-P165 “Quantification of CD34+ cell counts”;
- CCQM-K86.c/P113.4 Key Comparison “Relative quantification of genomic DNA fragments extracted from oil matrix (OSR)”;

Scheduled comparisons:

- CCQM-Pxxx Comparisons in the field of recounting of blood elements;
- CCQM-Pxxx Quantitative determination (and fractional composition) of genomic DNA isolated from high-protein tissue (meat/fish and qualitative/quantitative model);
- CCQM-Pxxx Comparisons of methylated DNA.

Working Group on Cell Analysis determined the following priority areas of CMC development:

- recounting of cells using flow cytometry;
- determination of number of antibody binding sites per cell using flow cytometry;
- determination of concentration of CD4 and CD34 cells using flow cytometry;
- determination of cell density on 2D surface;
- culture confluence on 2D surface;
- measuring of relative cell parameters in a single cell culture or line;
- determination of stem cells dose delivered by a single therapeutic product;
- functional activity of stem cells delivered by a separate therapeutic product;
- characteristics of interaction between nanoparticles and cells;
- determination of number of living microbial cells.

7. Report of the Chairperman of TC 1.8/SC 3 “Pure inorganic substances” Egor P. Sobina, UNIIM, on activities of the Subcommittee and his proposals for future works.

7.1. Status of works on ongoing COOMET subjects with UNIIM as the Coordinator:

- 613/RU-a/13 Pilot comparison in the field of measuring of porosity properties (specific absorption, specific surface, specific pore volume, pore diameter) of nanoporous materials;
- 650/RU/14 Pilot comparison in the field of measuring of mass fraction of metals (Cu, Zn, Pb, Al, Ca, Sb, Mg, Au, Ag, As, Ni) in smelter slag (part II); participants: UNIIM, VNIIOFI, BelGIM, "Ukrmetrteststandart", KazInMetr;
- 645/RU/14 Pilot comparison in the field of measuring of mass fraction of copper and impurities in the oxygen-free copper wire rod KMb M1001b for determination of copper purity; participants: VNIIOFI, BelGIM, "Ukrmetrteststandart", TUBITAK UME;
- 672/RU/15 Pilot comparison in the field of measuring the mass fraction of Fe in high purity iron; participants: "Ukrmetrteststandart", UNIIM, VNIIOFI, BelGIM;
- 692/RU/15 Pilot comparison in the field of measuring of moisture mass fraction in wood; participants: "Ukrmetrteststandart", BelGIM, KazInMetr, INM;
- 696/RU/15 Pilot comparison in the field of measuring of mass fraction of Au and Ag in gold ore using high-precision methods: inductively coupled plasma mass spectrometry, inductively coupled plasma-atomic emission, etc.; participants: BelGIM, etc.

7.2. Participation of UNIIM in ongoing CCQM comparisons:

- CCQM-K136/CCQM-P180 Measurement of porosity properties of nanoporous Al_2O_3; Coordinator: UNIIM;
- CCQM-P167 Mass fraction of protein nitrogen in milk powder; Coordinator: UNIIM;
- CCQM-K130/P166 Nitrogen in glycine; Coordinator: UNIIM;
- CCQM-K128 "Heavy metals and organo-tin (tributyltin) in leather powder: Pb, Cd, Hg, As, Sb, Co, Cu, Ni in the concentrations range from 30 mg/kg to 200 mg/kg and tributyltin from 100 mg/kg to 300 mg/kg; Coordinator: NIM;
- SIM.QM-S7 Supplemental comparison "Metals in drinking water"; Coordinators: NRC/CENAM.

7.3. Report on UNIIM works in the framework of BIPM in 2017-2020:

- CCQM-K144 Analysis of elemental impurities in alumina powder; Coordinator: KRISS;
- CCQM-K143 Copper calibration solutions; Coordinator: NIST;
- CCQM-K34.2016 Assay of potassium hydrogen phthalate; Coordinator: NIM;
- CCQM-K145 Toxic and essential elements in bovine liver powder; Coordinator: NIM;
- CCQM-Kxxx Mass fraction of nitrogen in milk powder; Coordinator: UNIIM;
- CCQM-Kxxx Measuring of specific surface of white silica sand SiO_2; Coordinator: UNIIM;
- CCQM-Kxxx Measuring of porosity properties (specific absorption, specific surface, specific pore volume, pore diameter) of nanoporous zeolite; Coordinator: UNIIM;
- CCQM-Kxxx Measuring of pore size of nanoporous Al_2O_3; Coordinator: UNIIM;
- CCQM-Pxxx Pilot comparison "Measuring of porosity and openness of earth material"; Coordinator: UNIIM;
- CCQM-K/Pxxx Determination of mass fraction of base substabce of high-purity salts; Coordinator: UNIIM.

7.4. Report on status of Reference Materials in Russia and at international level, and on implementation of ISO 34.

8. Report of the Chairman of TC 1.8/SC 2 “Metals and alloys” Natalya P. Muravskaya offering to conduct Pilot comparison 618/RU/13 in the field of composition of nickel-based alloys; Coordinator: VNIIOFI, participants that are interested in the comparison: UNIIM, etc.
9. Report of the Chairman of TC 1.8/SC 1 “Electrochemistry” Vladimir I. Dobrovolskiy on the results of Pilot comparison 537/RU/12 in the field of measuring of number concentration of aeroions; Coordinator: VNIIFTRI, participants: VNIIM and VNIIFTRI.

   The following new comparisons were offered to be carried out in the field of electrochemistry:

   – xxx/RU/18 Pilot comparison in the field of pX measurements of sodium ions activity, pNa ≈ 1.1; Coordinator: VNIIFTRI;
   – xxx/RU/18 Pilot comparison in the field of pH measurements, pH ≈ 1.2; Coordinator: VNIIFTRI;
   – xxx/RU/XX Supplementary comparison in the field of pH measurements of phosphate buffer, pH ≈ 7.0; Coordinator: VNIIFTRI.
10. Report of TC 1.8/SC 1 “Electrochemistry” Chairman S.V. Prokunin on the first results of Pilot comparison 655/RU/15 “Pilot study on pH measurement of phosphate buffer pH ≈ 7.0 (Coordinator: VNIIFTRI; participants: PTB, KazInMetr, Center for Standardization and Metrology of the Ministry of Economy of the Kyrgyz Republic, NIM of Moldova, TUBITAK UME, GEOSTM, BelGIM, “Ukrmetrteststandart”).
11. Report of Ahmet Ceyhan Gören about activities of TUBITAK UME, its structure, achievements, and main goal, which is to produce reference materials. Ahmet Ceyhan Gören also proposed to organize pilot comparisons with TUBITAK as the Coordinator:
    “Measurement of C_{12}/C_{13} isotope ratio in sugar” and
    “Measurement of C_{12}/C_{13} isotope ratio in CO_2/N_2”.
12. Reports of TC 1.8 members from Belarus, Ukraine, Moldova, Kazakhstan, and Kyrgyzstan.

II. Following the discussion of the reports, participants of the Meeting of COOMET Technical Committee 1.8 “Physics-Chemistry” have decided:

1. to approve activities of COOMET TC 1.8 “Physics-Chemistry” during the reporting period and results of works in June 2016 – May 2017;
2. to approve activities of TC 1.8/SC 1 “Electrochemistry” during the reporting period;
3. to approve activities of TC 1.8/SC 3 “Pure Inorganic Substances” during the reporting period;
4. to continue works on the ongoing COOMET subjects; and
5. to approve the following suggestions:

<u>to transfer the date of reference materials distribution for the following comparisons in 2018:</u>

– xxx/RU/18 Key comparison “Natural gas”; Coordinator: VNIIM; participants: BelGIM, kazInMetr, “Ukrmetrteststandart”, etc.;
– xxx/RU/18 Key comparison “Ammonia in nitrogen, 10-30 ppm”; Coordinator: VNIIM; participants: BelGIM, KazInMetr, “Ukrmetrteststandart”, etc.

<u>to establish new COOMET subjects:</u>

– 618/RU/13 Pilot comparison in the field of composition of nickel-based alloys; Coordinator: VNIIOFI, participants that are interested in the comparison: UNIIM, etc.;
– xxx/RU/18 Pilot comparison in the field of pX measurements of sodium ions activity, pNa ≈ 1.1; Coordinator: VNIIFTRI;
– xxx/RU/18 Pilot comparison in the field of pH measurements, pH ≈ 1.2; Coordinator: VNIIFTRI;

- xxx/RU/XX Supplementary comparison in the field of pH measurements of phosphate buffer, pH ≈ 7.0; Coordinator: VNIIFTRI; participants: NIM of Moldova, etc.
- xxx/RU/18 Key comparison "CO_2 in air, atmospheric level"; Coordinator: VNIIM, participants that are interested in the comparison: BelGIM, KazInMetr, "Ukrmetrteststandart";
- xxx/RU/18 Supplementary comparison "Ethanol in aqueous solution, 1.2 mg/cm^3"; Coordinator: VNIIM, participants that are interested in the comparison: NIM of Moldova;
- xxx/RU/18 Pilot comparison "Ethanol in aqueous solution, mass fraction 40%"; Coordinator: VNIIM; participants that are interested in the comparison: NIM of Moldova;
- xxx/RU/18 Pilot comparison "Measurement of C_{12}/C_{13} isotope ratio in sugar"; Coordinator: TUBITAK UME; participants that are interested in the comparison: VNIIM;
- xxx/RU/18 Pilot comparison "Measurement of C_{12}/C_{13} isotope ratio in CO_2/N_2"; Coordinator: TUBITAK UME; participants that are interested in the comparison: VNIIM;

Participants that are interested in taking part in those comparisons should send a confirmation to the Coordinators and the Chairman of COOMET TC 1.8 not later than August 31, 2017.

Coordinators should prepare and register the corresponding COOMET subjects not later than December 31, 2017.

Elaboration of the project "Pilot comparison: electrolytic conductivity at 0.014 – 1.4 S/cm" (Coordinator: VNIIFTRI; participants that are interested in the comparison: Center for Standardization and Metrology of the Ministry of Economy of the Kyrgyz Republic, NIM of Moldova) should be further addressed.

6. D.I. Mendeleyev Institute for Metrology to prepare proposals on including the method of measuring urea and uric acid in human serum used for CCQM-K109/P148 comparison "Urea and uric acid in human serum – high-polarity analytes in biological matrix" as the reference method for JCTLM database.
7. To recommend "Ukrmetrteststandart" and BelGIM to submit in the next cycle the set of CMCs of the core components based on the comparison COOMET.QM-K111 type Track A "Propane in nitrogen, 1000 ppm" completed in 2017.
8. To support UNIIM's request to include the Institute in CCQM.
9. To schedule the next Meeting of COOMET Technical Committee 1.8 "Physics-Chemistry" in May 2018 at VNIIM (St. Petersburg).

TC 1.8 participants were given the following documents:

- TC 1.8 "Physics-Chemistry" Work Programme
- TC 1.8 "Physics-Chemistry" Annual Work Report
- Draft Protocol of the Meeting of COOMET Technical Committee 1.8 "Physics-Chemistry"
- COOMET Recommendation "Regulations for Comparison of Measurement Standards from the National Metrology Institutes of COOMET"

Chairman of COOMET TC 1.8 "Physics-Chemistry" L.A. Konopelko