

IMPROVING METROLOGY IN CHEMISTRY INFRASTRUCTURE IN ROMANIA VIA IRMM-JRC-EC COLLABORATION

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*IRMM - GEEL, ** INM, ROMANIA, ***BRML ROMANIA

Rezumat. Articolul prezintă câteva activități inițiate de Institutul de Măsurări și Materiale de Referință - Centru Reunit de Cercetare al Comisiei Europene (Institute of Reference Materials and Measurements- Joint Research Center-European Commission (IRMM-JRC-EC)) și Institutul Național de Metrologie – Biroul Român de Metrologie Legală (INM-BRML) pentru îmbunătățirea infrastructurii de metrologie în chimie în România. În perioada 2002-2008, IRMM-JRC-EC, institutul european de metrologie și INM-BRML, autoritatea națională de metrologie au fost implicate în diferite acțiuni comune, cum ar fi elaborarea de studii și strategii pentru metrologia în chimie, diseminarea cunoștințelor pentru mai buna înțelegere și aplicare a principiilor metrologiei în chimie în practica analitică chimică, coordonarea laboratoarelor de chimie naționale pentru participarea lor în Programul Internațional de Evaluare a Măsurării (International Measurement Evaluation Programme (IMEP)) și alte activități legate de metrologia în chimie, care sunt descrise pe scurt în articol. Folosind facilitățile tehnice ale IRMM, s-a realizat transferul de know-how precum și expertiza de înalt nivel în domeniul metrologiei în chimie de către personalul național. Au fost realizate câteva proiecte de cercetare în legătură cu activitățile CCQM/EURAMET, certificarea compoziției chimice și izotopice a materialelor de referință IRMM candidate și au fost explorate, studiate și efectuate activități de cercetare pentru susținerea problemelor de management de mediu.

Cuvinte cheie: metrologie în chimie, instruire, TrainMiC®, AcadeMiC®, IMEP.

Abstract. This paper presents some activities initiated by Institute of Reference Materials and Measurements- Joint Research Center-European Commission (IRMM-JRC-EC) and National Institute of Metrology- Romanian Bureau of Legal Metrology (INM-BRML) for improving the infrastructure of metrology in chemistry in Romania. During the period 2002-2008, IRMM-JRC-EC, the European metrology institute, and INM-BRML, the national authority of metrology, were involved in different common activities, such as elaboration of studies and strategies of metrology in chemistry, knowledge dissemination for the better understanding and application of metrology in chemistry principles in chemical analytical practice, coordination of national chemical laboratories for their participation in the International Measurement Evaluation Programme (IMEP) and other related metrology in chemistry activities, that are shortly described in this paper. Using the IRMM technical facilities, know-how transfer and high level expertise in chemical metrology field were achieved by the national staff. Some research projects were carrying out in respect of CCQM/EURAMET activities, certification of chemical and isotopic composition of the IRMM candidate reference materials as well as research activities in support of the environmental management issues were explored, studied and carried out.

Key words: metrology in chemistry, training, TrainMiC®, AcadeMiC®, IMEP.

INTRODUCTION

On the basis of quantitative chemical measurements many important decisions are made in support of legislation or for industrial processes or social aspects. For this reason it is important to have a good quality of chemical measurement results, to improve them continuous and to make them comparable and acceptable everywhere.

IRMM-JRC-EC is the European metrology institute which promotes a common and reliable European measurement system in support of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of

the Member States, while being independent of special interests, whether private or national.

INM-BRML is a specialised research and development institute in the field of metrology which ensure the uniformity, accuracy and precision of measurements all over the country. These objectives are achieved by developing and maintaining the national and calibration standards, which, by calibration operations, are used to disseminate the units of measurement to the laboratories belonging to BRML or other economic operators.

In the actual context of the chemical measurements importance, measurements not very well developed in many national metrology institutes, IRMM-JRC-EC set up in 2001, in the framework

of EU enlargement, a programme to improve the scientific basis for metrology in chemistry.

Romania, via INM-BRML was part of this programme since its beginning in 2001. Nowadays this programme becomes a wide European programme for many EU countries; several activities are initiated such as elaboration of strategies based on the documentation studies, knowledge dissemination and training, fellowships, seminars, conferences or inter-laboratory comparisons, know-how transfer and other. Summary of some these activities are presented below.

1. STUDIES AND STRATEGIES ON THE METROLOGY IN CHEMISTRY IN ROMANIA

As the first step for future improvement process on metrology in chemistry infrastructure, IRMM's initiative was to assist the national organisations (i.e. metrology institutes or universities) to document the status of metrology in chemistry in the country, then, to write about the main activities, responsibilities and further preoccupations of the major stakeholders which are dealing with chemical measurements. The Status of metrology in chemistry in Romania was documented and elaborated. The activities of more 65 institutions were described and more than 25 people contributed to collect the relevant information contained in this report. It was published as EUR 20840 EN [1] and it is distributed, at the national level as well as at the European level [2].

The Status Report of Metrology in Chemistry in Romania consists in more chapters shortly described below. In the *Abstract* three types of key institutions dealing with chemical measurement issues-governmental, non-governmental and private institutions-and their main activities and responsibilities related to chemical measurements are identified. These institutions are specialised metrology organisations, some ministries and ministries's agencies, accreditation body, standardisation body, public or private chemical laboratories and some universities and associations.

The chapter *Governmental bodies* describes the main activities performed by metrology organisations as well as accreditation activities, standardisation issues, consumer protection activities as well as research activities carried out by the institutes coordinated by or under the Romanian Academy. For each body their responsibilities are shortly described, their tasks as well as the links with other bodies are indicated.

The *National metrology system* is shortly described in this chapter. The policy in the field of

scientific, legal and industrial metrology is in the competence of BRML. In the structure of BRML there are eight Regional Directions for Legal Metrology (DRML) which are involved in metrological control of measuring instruments. INM acts, under BRML coordination, as the research institute on metrology at national level. The basic Law of Metrology (the first law was adopted in 1978, later it has been several times amended, the last amendment was made in 1999), together with related metrological acts, regulate all aspects of measurements all over the country, such as, measurement units, standards, measuring instruments and their type approval, verification, calibration and other metrological services. BRML and INM represent Romania in the scientific international and regional organisations, such as BIPM, OIML, EURAMET, WELMEC, and other.

Various *Non-governmental* institutions are recognized by Romanian Government as national bodies for accreditation and conformity assessment-Romanian Accreditation Association (RENAR), for standardization-Romanian Standard Association (ASRO), for consumers protections-National Authority for Consumers Protection (ANPC) or for dangerous substances and chemicals-National Agency for Dangerous Substances and Preparations (ANSPP). Including all these institutions at the beginning of the report, even they are not operated under the same law or under the same ministry, is justified by their important role in the national measurement system at the country level. Activities under responsibility of the Non-governmental bodies, described in this chapter, underlines the main activities to disseminate information, promote scientific news or other educational activities (conferences, seminars, inter-laboratory comparisons etc.).

Different activities relevant to chemical measurements are taking place in various *Institutions acting under the responsibility of various ministries* that are also presented in the report: (i) industry, economy and commerce, (ii) education and research, (iii) agriculture, forest, water and environment and (iv) health. The research and development institutes, companies, inspectorates, directorates or state agencies or other departments are responsible for strategic activity, monitoring, scientific and applied research in each field. For each body their responsibilities are described, the means to fulfilling their tasks as well as the links with other bodies are underlined. It is important to stress that only few companies are presented in this part of the report, which meanly could have a big impact on chemical measurement area.

In the chapter *Laboratories* the developed Romanian infrastructure of testing chemical laboratories, public and private, their functions and activities and sometimes their status concerning accreditation process are described. The applicable measurement methods and the available equipments are mentioned, the main research programmes-national and international are shortly presented as well as the inter-laboratories rounds where the respective laboratories have been participated.

Concerning *Education and training* in the chemical field, the Romanian universities have long tradition in teaching chemistry. A lot of specialization studies, postgraduate courses in many domains, distance learning courses influenced by the European integration process, research activities as mandatory components for the academic staff are offered by chemical faculties. Many universities offer the education either in the chemistry departments or in other departments (geology, biology, pharmacy or environmental studies) where the chemistry is one of the essential subjects. Some research centres for fundamental or applied research under the responsibility of universities there are also involved in training and education activities. However, in the academic curriculum the metrological aspects of chemical measurements are not included.

Important conclusions are pointed out in the *Summary* of this report. They are concerning the need of participation of chemical laboratories in inter-laboratories comparisons, proper use of certified reference materials and educational aspects needed in the field of chemical measurements.

The Appendix contains also several spreadsheets presenting the summary on the present state in some important area such as environmental, food and clinical sectors as well as the main tasks of the representatives institutions from these sectors. The spreadsheet contains as well a brief description of the monitoring status for wastewater, soil, air and pesticides, from which it is possible to conclude who is currently responsible for what at the national level. Some specific information concerning the organisation and participation of Romanian laboratories in the different inter-laboratory comparisons as well various kinds of reference materials produced at national level are also annexed.

Based on this documented study of the present status on metrology in chemistry at national level, some *Conclusions* were summarised, mainly related to the measurement/calibration capabilities at the national level, the participation of chemical laboratories in inter-laboratory comparisons, the traceability dissemination, training and education in metrology in chemistry and other activities.

Taking into account the main conclusions of Status Report of Metrology in Chemistry, as a *process to document* the current status of metrology in chemistry and as the *first step of the future improvement process*, new priorities at national level are identified. They are mentioned in the above status report of Metrology in Chemistry in Romania. The need to use the national measurement capabilities (human resources, equipments, knowledge etc.) in the important areas of interest/competence for industry, environmental, food, clinical, consumer protection etc. is considered as an important issue for improving metrology in chemistry in Romania.

Later on, in 2004, as a next step for improvement process of metrology in chemistry infrastructure, a *strategy paper* for a national metrology organisations was prepared and published [3]. This paper describes how to set up a cost effective distributed metrology infrastructure for chemical measurements. A national measurement infrastructure is defined as a collection of various measurement services (testing, calibration and reference laboratories) and the communication between these services. In this paper the distributed metrology infrastructure covers those organisations that are involved in dissemination measurement traceability (i.e. the national metrology institute, universities, research centres or reference laboratories acting as national reference standard holders).

In particular, when proven competence is available, the national metrology institute itself, can and should act as national reference standard holder in the field of chemical metrology, and as a consequence, to be able to declare their measurement capabilities recognised under the CIPM-MRA.

However, the new trend, for a better co-operation and particularly a better understanding of what the future role and activities of each of the stakeholders should be in the field of chemical measurements, is to bring together, at national level, the metrology and chemical experimental expertise in those areas in which the metrology institutes don't have enough experimental measurement capabilities in the area where the quality of measurement results play the most important role in the country.

The conclusions on the Status report Metrology in Chemistry in Romania and the strategy presented in the open literature publication presented above, were discussed and transferred to the national metrology level. As a consequence, some idea were took over and, at the moment, INM-BRML in collaboration with two Romanian partners [4], runs a project to develop new national measurement capabilities in a new area of chemical expertise.

2. KNOWLEDGE DISSEMINATION VIA TRAINMiC® AND ACADEMiC® PROGRAMMES

The demand for reliability of chemical measurement results is continuously growing in the last decade. This needs better understanding of the chemical measurement procedures and the well applied metrological concepts. As a consequence the knowledge dissemination concerning the science of chemical measurements is needed.

TrainMiC® - Training for Metrology in Chemistry has been set up as a training platform for experts from all types of organisations. Via the TrainMiC® platform [5], a set of training presentations and examples had been elaborated that provides better understanding in basic measurement matters, which apply to measurements across different sectors such as food, environmental, clinical, industrial, research or routine measurements.

TrainMiC training material contains theoretical presentations covering the important information on metrology of chemical measurements. The main topics are: (i) General introduction to Metrology in Chemistry; (ii) Traceability of chemical results; (iii) Uncertainty of measurements results; (iv) Validation of measurement procedures; (v) Use of certified reference materials; (vi) Inter-laboratory comparison; (vii) Applied statistics; (viii) Internal quality control; (ix) Sampling as a part of measurement procedure. TrainMiC examples contain practical applications for environmental, food, clinical and other chemical measurements. Examples are structured in three types of exercises: uncertainty estimation of chemical results, establishing of the traceability of chemical results and validation of the chemical analytical procedures. They are presented in an uniform manner all over the countries involved in this programme, to allow the lecturer and the audience to efficiently use the theoretical and practical knowledge accumulated during TrainMiC seminars.

A short description of the TrainMiC training material could be found on the internet [5].

Metrology in Chemistry - training and education activities in Romania

The TrainMiC programme has its beginning in Sinaia, Romania in 2001; nowadays it is well accepted and implemented at the international and national level as well. The training seminars are dedicated to the national needs, as a consequence, a large target audience of the national seminars

are practitioners from testing and calibration laboratories as well as end-users of measurement data.

A training national team is in place [5], the team members (3 out of 4 members are INM staff) are periodically trained at IRMM by TrainMiC editorial and management boards members. The TrainMiC presentations are translated into the Romanian language and are presented by the national team members in the native language.

From its beginning, at least 15 seminars were organised up to now at the national level. More than 350 participants from metrology institute, universities, accreditation body, ministries, chemical laboratories attended the TrainMiC seminars. The TrainMiC training courses were organised by the local public or private partners, such as national metrology institutes, universities or accreditation bodies, etc. The local hosting and organisation institutions were INM-BRML, RENAR, ROLAB, ANAR, ECOIND, Cantacuzino Institute, ICPAO and other. The TrainMiC events took place in Romania were periodically reported at the European level [5]. Particularly, in support of IMEP participants, an initiative was based on the IMEP-EDUC programme. As a consequence, a workshop with open discussion of basic problems of laboratory performance was held in Romania to help IMEP participating laboratories to improve the quality of their measurement results. Also some seminars were addressed to European directives implementation in the environmental, food or clinical fields.

In collaboration IRMM, the TrainMiC programme brings the opportunity to work together with different Romanian organisations and to share experience on the related topics. The seminars offered the opportunity to all involved to deal with theoretical principles and practical applications of chemical measurements, and as a consequence, to meet the requirements of the '*acquis communautaire*'.

Another knowledge dissemination activity supported by INM-BRML is via *AcadeMiC®: Metrology in Chemistry for Academia*. The idea of AcadeMiC® originated in 2002 at IRMM-JRC-EC. From the beginning, the vision of AcadeMiC® was to foster the advancement of education in metrology in chemistry and related topics (e.g. method validation, traceability, measurement uncertainty) on the generic measurement issues that are addressed in the standard ISO/IEC-17025 [6]. It is important to provide good quality education to the new generation of university students, so they are equipped with the necessary competence

entering the job market. The implementation of this idea requires a forum [7] where people and organisations lecturing/training in these topics meet, exchange skills, share experience and materials.

More activities related to this topic are published somewhere [8, 9] by a large group of people bringing together their experience in the field. Later on, the effort was spent to create a Joint Degree Programme (JDP) on this topic. Nine universities, one from Romania, are now part of the consortium that applied to the European Chemistry Thematic Network Association (ECTNA). In this respect, INM-BRML acts, on behalf of Romania, as specialised metrology organisation to support the Romanian universities to joint this activity.

Further information and new developments are available on [5].

3. PARTICIPATION IN THE INTERNATIONAL MEASUREMENT EVALUATION PROGRAMME (IMEP)

To disseminate measurement traceability, IRMM provides with its International Measurement Evaluation Programme (IMEP) an inter-laboratory tool to enable the benchmarking of laboratory performance. IMEP emphasizes the metrological aspects of measurement results, such as traceability and measurement uncertainty. In this way it became a publicly available European tool for metrology in chemistry. The aim of IMEP is to demonstrate objectively the degree of equivalence and the quality of chemical measurements by comparing participants' measurement results with these external reference values, which are completely independent from the participants' results. The reference values are obtained by metrological work of recognized reference laboratories with demonstrated measurement capability at international level. Par-

ticipating laboratories can use this value to assess the quality of their results and to support their measurement capabilities claims on an international forum. IMEP is focused as much as possible on 'real – life' samples and has as the main objective to enable assessment of measurement capability linked to implementation of European Directives in the fields where chemical measurements are important, such as consumer protection and public health, single market, external trade, environment, research and technology or economic policy. IMEP is aiming to assist the establishment of an internationally structured measurement system for chemical measurement, and in this respect, interacts with different bodies as BIPM-CCQM, EUROMET, European Accreditation and others [10, 11, 12].

Participation of Romanian chemical laboratories in the IMEP inter-laboratory comparisons (ILCs) is detailed described in [13]. Romania has a developed infrastructure of testing chemical laboratories, most of them are accredited or under accreditation process. INM-BRML actively supports the participation of Romanian authorized and field laboratories in IMEP inter-laboratory comparisons. Even more, INM acts as regional coordinator for IMEP programme [2]. Since 2000, some interested laboratories from Romania used the opportunity to participate in this programme, they accounted for about 3-25% of the participants in IMEP ILCs (Fig. 1).

As an example, the distribution of participants from new member states (= former acceding countries), candidate countries and Western Balkan countries for IMEP-12 (Trace elements in water) in respect of EC Directive 98/83/EC on drinking water implementation [14] is given in Fig. 2; from a total of 348 results, 151 results are from former acceding and candidate countries, and 16 from Romanian participants.

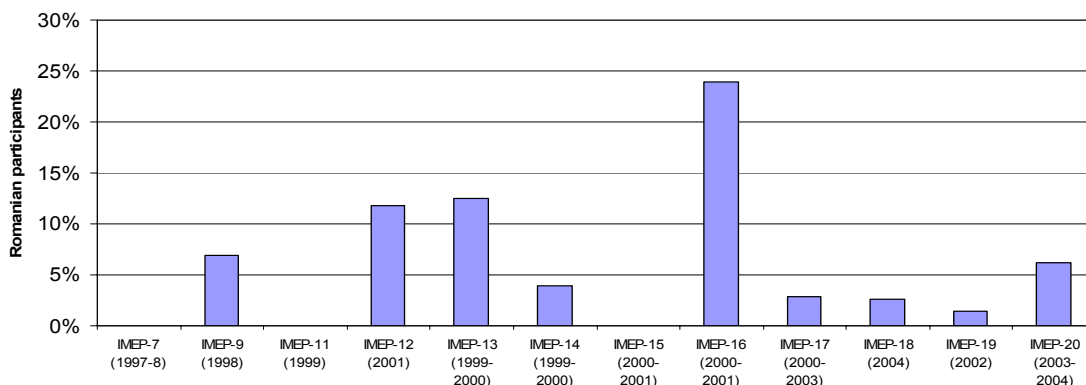


Fig. 1. Participation of Romanian laboratories in IMEP ILCs in percentage.

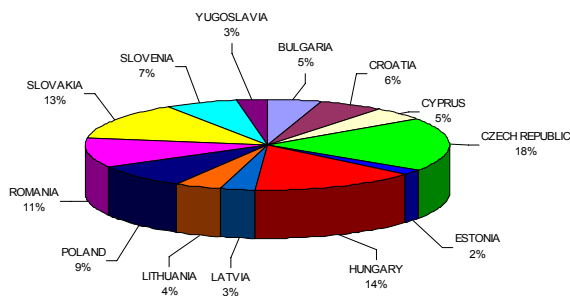


Fig. 2. IMEP-12: Trace elements in water.

The number of national interested laboratories was also considerable for IMEP-16 (Lead in wine), also the participation in IMEP-17 (Trace and Minor Constituents in Human serum) and IMEP-20 (Trace Elements in Tuna Fish) were representative, because of compliance of laboratories with relevant Romanian and EU legislation in each sector (food, clinical and consumer protection). In Fig. 3 the comparing measurement capabilities across the international measurement infrastructure with results from Romanian participants are presented, for IMEP, EURAMET and CCQM comparisons.

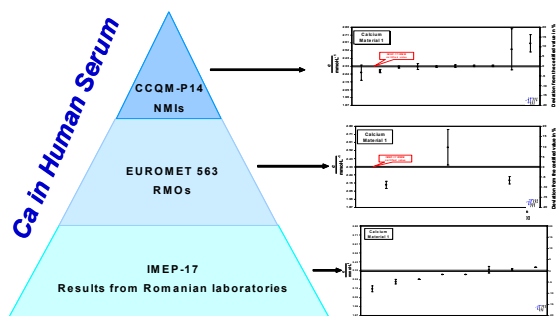


Fig. 3. Calcium in serum: IMEP, EURAMET and CCQM comparisons.

4. KNOW-HOW TRANSFER, THEORETICAL AND APPLIED TECHNICAL EXPERTISE

Using the technical facilities existing in the IRMM laboratories, equipped with TIMS, GC-MS, IRMS, TC-EA etc. the high level expertise in chemical metrology domain was achieved by Romanian staff working there. The international and multicultural environment from EC institution, brings a new perspective for the measurement chemical infrastructure, projects management and other important issues in the field.

By carrying out research projects in respect of **CCQM/EURAMET activities** the measurement methods as IDMS were studied and applied i.e. for CCQM-K14: Key comparison: Calcium amount content in human serum. The aim of CCQM-K14 was to measure the calcium amount content in unmodified frozen human liquid serum. The main task of this work was to establish and demonstrate the traceability and degree of comparability of calcium measurement results.

Thermal ionisation mass spectrometry (TIMS) became the preferred analytical technique for the determination of stable calcium isotopes (^{42}Ca , ^{44}Ca , ^{46}Ca , ^{48}Ca) because its good measurement performance characteristics. With this method SI traceable results with expanded uncertainties ($k = 2$) below 1% were obtained. Good results were reported in CCQM-K14 (see Fig. 4) on behalf of IRMM participation institution.

Related to the improvement process at the national level in respect of EURAMET/CCQM activities, some support was given to INM-BRML in its participation in EURAMET comparisons.

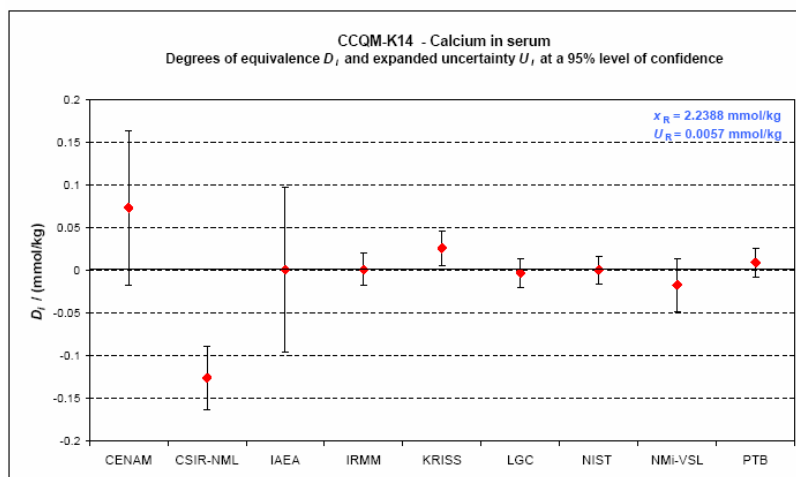


Fig. 4. CCQM-K14: Key comparison: Calcium amount content in human serum.

The *reference materials certification* expertise were reached by Romanian staff working there carried out the certification reference materials in the special clean environmental conditions of IRMM facilities. The chemical and isotopic composition of lithium reference materials IRMM-016, IRMM-015 and IRMM-615 were characterized. The certified amount content of IRMM-615 was estimated based on isotope dilution equation using specialized software. The corresponding isotopic composition of reference material was calculated in terms of amount fraction as well as mass fraction. The associated value of molar mass of lithium in IRMM-015 was determined. The re-certified isotope ratio of IRMM-016 was performed as well, and by comparing this value with the previous certified value of IRMM-016, more accurate re-certified value was reported. As a consequence of this work, the new certified values of lithium isotopic reference materials IRMM-615, IRMM-015, IRMM-016 and the corresponding certifications reports were published and they are included now in the IRMM isotopic reference materials catalogue [15].

The research activities in the context of some *European projects* were carried out. As an example, the exploratory and applicative researches for environmental protection were carried out for chemical and isotope data of $\delta^{15}\text{N}$, $\delta^{18}\text{O}$ and $\delta^{11}\text{B}$ in respect of source apportionment of suspected pollutants in different environmental samples. Special work was allocated for analytical aspects and boron isotopic measurements by TIMS. Boron isotope ratio measurements are tedious and time consuming due to low boron concentration in surface water samples (5-100 $\mu\text{g/L}$), due to easy contamination during entire process measurement and due to high boron volatility. The amount of boron required for isotopic ratios measurements by TIMS is above 1-2 $\mu\text{g B}$. At the moment the results are used for environmental management issues in respect of EU Directives implementation; the results are already published or presented at international conferences [16, 17, 18].

CONCLUSIONS

The activities presented above for improving metrology in chemistry in Romania via IRMM-JRC_EC and INM-BRML collaboration has a positive impact on the Romanian metrology infra-

structure. Many activities presented in these paper offer the opportunity to be faced and to take over at the national level some of the European expertise on the metrology in chemistry issues.

Some new priorities and some possible approaches at national level were identified, based on the studies and publications on the status of metrology in chemistry in Romania and on the proposed strategies for metrology in chemistry infrastructure in different countries. The need to use the national measurement capabilities (human resources, equipments, knowledge etc.) in the important areas of interest/competence for industry, environmental, food, clinical, consumer protection etc. already started to be considered as valuable approach in the context of the national metrology research activities.

The knowledge dissemination is considered as an important issue in the metrology in chemistry domain. As a consequence, TrainMiC® European training platform, in which Romanian laboratories were interested from its beginning in 2001 is well accepted and is running well in Romania; at the moment at least 15 events were organised, more than 350 people were trained in support of testing and calibration laboratories accreditation, to meet the requirements of the '*acquis communautaire*'.

The measurement traceability disseminate in the country via inter-laboratory comparisons, i.e. CCQM, EURAMET, IMEP, with INM acting as regional coordinator for IMEP in Romania and with some recognised CMCs for monoelemental standard solutions produces by INM, are useful tools for testing and calibration Romanian laboratories, to enable the benchmarking of the laboratories performance.

The technical expertise and the intercultural working environment experience accumulated by Romanian staff working at IRMM, brings also some performance indicators [4], such as report, publications, etc. for INM-BRML research activities.

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