

## SOME LATIN-AMERICAN EXPERIENCES CONCERNING TEACHING OF CHEMICAL METROLOGY

*M. Alvarez Prieto<sup>1</sup>, J. Jiménez Chacón<sup>2</sup>, I. Cortés Nodarse<sup>3</sup>, N. Martínez Alfonso<sup>4</sup>*

Institute of Science and Technology of Materials, University of Havana, Cuba. Zapata y G, s/n, Habana, Cuba.

<sup>1</sup> [malvarez@imre.oc.uh.cu](mailto:malvarez@imre.oc.uh.cu)

<sup>2</sup> [jjimenez@imre.oc.uh.cu](mailto:jjimenez@imre.oc.uh.cu)

<sup>3</sup> [isel@imre.oc.uh.cu](mailto:isel@imre.oc.uh.cu)

<sup>4</sup> [nancy@imre.oc.uh.cu](mailto:nancy@imre.oc.uh.cu)

**Abstract:** Some acquired experiences concerning the teaching of Chemical Metrology in Latin-America for almost fifteen years are presented. They include postgraduate and undergraduate activities developed in seven countries of this continent. The combination of theoretic and practical activities and the sequence of learning from metrological, statistical and chemometrical backgrounds up to practical activities in personal computers are basic and motivate the learning process. Care is taken in order to promote the metrological approach and thinking in Analytical Chemistry. The learning of computing techniques plays an important role, combining graphic and numerical techniques for data analysis. The role of examples during the teaching process is analyzed and recognized. The introduction of a general model of errors permits to approach different topics on a metrological basis. Similarly, linking of performance characteristics of analytical procedures and validation concepts is essential. The understanding of traceability prepares students for organizational actions. Proper understanding of interlaboratory studies is attained on a practical basis. The metrological approach of uncertainty based on the Theory of Errors permits to develop the topic. A better comprehension of written standards is obtained from practical examples. Undergraduate students acquire a basic metrological knowledge very well. Recommendations for undergraduate and postgraduate programs are pointed out.

**Keywords:** chemical metrology teaching, analytical chemistry learning, quality assurance teaching.

### 1. INTRODUCTION

Teaching of Chemical Metrology is mandatory nowadays on the basis of a modern approach. Classical approaches for Analytical Chemistry learning do not emphasize the necessary metrological fundamentals. Some efforts have been made to introduce new concepts and practices in undergraduate courses [1-3]. Additionally, postgraduate courses need to be provided with continuing professional development requirements [4]. To fill the gap of experienced or novel professionals, our staff prepared and has developed a complete series of courses which combines both theoretical and practical approaches.

Five courses concerning quality and Chemical Metrology as applied to analytical laboratories have been introduced in postgraduate teaching programs at the University of Havana during several years. The courses in logical order are: "Chemometrics for Quality", "Quality of Analytical Laboratories", "Sampling and Quality", "Computing and Quality" and "Accreditation of Analytical Laboratories". Two of them are included in two Master in Science programs and a Ph. D. program. The five integrate the Diploma "Quality of Analytical Laboratories". All or some of them have been taught in Argentina, Cuba, México, Panama and Uruguay. Derived short courses have been presented in Guatemala, Panama and Peru. So, some Latin-American experiences concerning the teaching of Chemical Metrology are presented and discussed in this article.

### 2. METHODS AND RESEARCH METHODOLOGY

Several methods have been used to attain the experiences presented in this work:

1) Direct inquiries at the end of postgraduate courses. This has been done by means of designed forms to fill or by means of verbal questions to students.

2) Inquiries to M.Sc. program providers, coordinators and professors. The exchange of experiences with colleagues related with other undergraduate or postgraduate courses has been very useful to sediment most of the ideas presented in this article.

3) Attendance at workshops and conferences on teaching. Other experiences from professors of different subjects and scientific branches have been useful, too, in order to include modern methods of teaching and learning.

4) Joint analysis in professors' periodic meetings. From the basis of a close relationship among the different courses, their logical sequence and treated topics, periodic meetings of professors have been essential in order to develop team work. For example, this has been basic to define detailed sequences of topics within each course.

5) Contacting industrialists, academics and specialists with interest on chemical metrology. These activities have been developed with the goal to know precisely the

necessities of students in different branches of economic or social activities. In some cases, some topics have been “modulated” (mainly, practical exercises) to adapt the courses to special interests (for the fields of drug manufacturing, Enology, etc.). This approach is very useful to satisfy expectations of students.

The main characteristics of students in different countries are the following:

- Age over 25 (average about 30, range 25-55).
- Mainly graduates in Chemistry, Pharmacy, Biochemistry, Pedagogical Sciences, Enology and Chemical Engineering.
- Mainly workers of analytical laboratories, industries, professors, specialists on quality and researchers.
- Equilibrium between sexes.
- About half of the students belong to Master in Science programs.
- Poor knowledge of Chemometrics, Chemical Metrology, Theory of Errors and statistical methods.

### 3. RESULTS AND DISCUSSION

The courses include several topics that are under development nowadays: Theory of Errors applied to Analytical Chemistry, metrological basis of Analytical Chemistry, uncertainty of measurements, validation of analytical procedures, traceability and ways to attain it, practical use of quality control techniques, routine application of control charts, role of interlaboratory studies to attain quality, good laboratory practices, practical use of ISO 17025 and other written standards, role of documentation in quality, and some other important topics. Comments about the main tendencies in the scientific literature are offered in class.

In general, the main interests of students are: 1) Theory of Errors and uncertainty of measurements and the ways to estimate it; 2) Single laboratory validation of analytical procedures; 3) the role of interlaboratory studies to attain quality and 4) the ISO/IEC 17025 standard and how it is applied in practice. Of course, these topics are very broad and students’ expectations can be satisfied by means of a combination of dissertations, discussion of examples and practical solution of problems in a computer lab.

Multiple experiences have been acquired by the staff of professors. For reasons of clarity, the experiences of the last fifteen years are discussed in the following paragraphs by blocks.

1. **An important motivation is the sequence of learning:**
  - i) metrological, statistics and chemometric backgrounds of different experimental techniques and tests applied for quality assurance and control;
  - ii) to learn their principles and practical role;
  - iii) to solve practical exercises in computers and
  - iv) as a complement, to discuss the international agreed guidelines for laboratory organization on the basis of former topics (Fig. 1). This sequence of learning layout allows

learning first the theoretical supports to understand later the experimental techniques and tests. These comprise techniques for sampling and quality assurance and control. After that, students face and solve real problems from actual life, to consolidate concepts and new knowledge on a practical base. To close the circle, the last course is intended to present all the above concepts approaching them from the point of view of the organizational level in a modern laboratory.

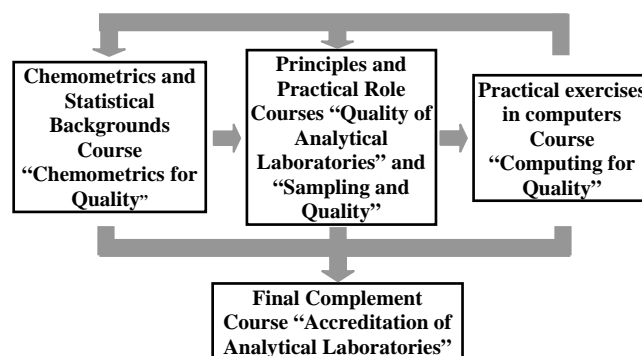


Fig. 1. General sequence of learning: role of precedence and complementary topics in the general learning process.

2. **It is proper to introduce the fundamental terms and concepts of General Metrology from the first activities.** Care is taken in order to promote the metrological approach and thinking in Analytical Chemistry. Terms and definitions are taken from the International Vocabulary of Metrology [5], The Guide for Uncertainty of Measurement [6] and some other important documents on vocabulary and basic concepts.
3. **The learning of computing techniques must play and indeed plays an important role.** It takes place by steps, incrementing complexity, but maintaining the necessary relationships between steps. Emphasis on analytical and metrological interpretation of results is done during the teaching process (Fig. 2).

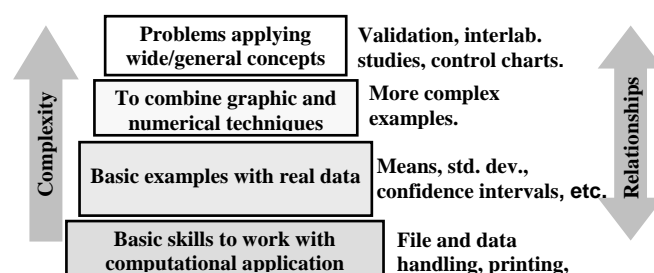


Fig. 2. Learning of computing techniques: from the basics to a general problem solve approach.

It starts with the learning of basic skills to work with computational programs (files and data handling, printing, plotting, etc.). It is followed with basic examples with real or simulated data (calculations of means, standard deviations, confidence intervals, etc.). After, the students combine graphical and numerical techniques for the analysis of real data with more complex examples. At the end, the students solve more complex problems applying more general and wider concepts (interlaboratory studies, validation of

analytical procedures, control charts, etc.). In general, the exercises are real examples taken from written standards, scientific articles, monographs and from our laboratory.

4. **Combination of graphic and numerical techniques for data analysis with computers allows to correlate results and to get a proper analytical interpretation of them.** Graphical representations of different techniques and test results are very useful because they make use of visual patterns. It is well known that the human brain is very well prepared to visually discern behaviors and characteristics of systems at hand. After that, exact numerical information is better interpreted. (Fig. 3)

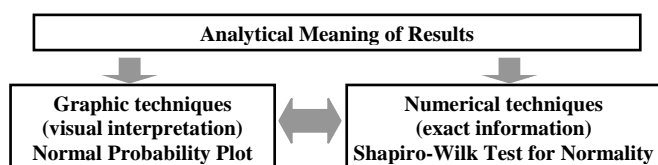


Fig. 3. Combination of graphic and numerical techniques of data analysis to interpret analytical meaning of results: visual interpretation combined with exact information.

5. **The role of examples during the teaching process is very important.** The key is to promote open discussions, starting with examples from professors, students or both. Examples come from different fields or areas, taking into account diversity of students' institutions. Orientation and leading is conducted by professors. Point apart is the discussion of written standards, where examples are fundamental to understand the general statements they contain. In that sense, particularization of concepts is important: from general concepts to particular cases, exemplified by concrete cases. On the other hand, the transition from examples (particular cases) transit to general concepts is another tool. (Fig. 4).

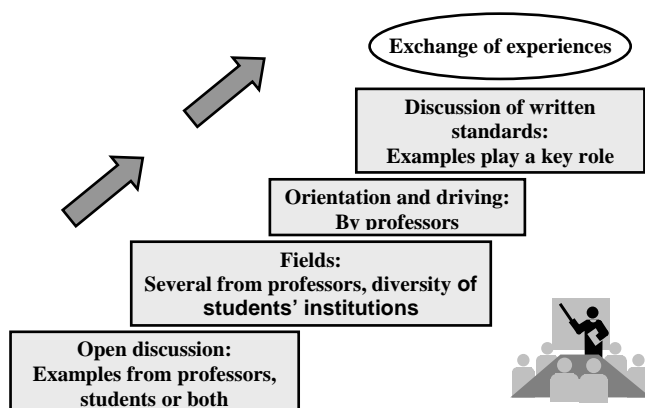


Fig. 4. Role of examples in the learning process: driven open discussions from different fields of Chemical Metrology where written standards take an important part.

6. **The introduction at the beginning of a general model of errors in chemical analysis [7] allows us to approach different problems and techniques on a metrological basis** (Fig. 5). Emphasis is done in the strategic principles to control errors. Behavior and nature of errors in Chemical Metrology is different if

compared with other fields like Physical Metrology and Microbiology. Thus, it seems that the matrix effects do not have analogies in other branches of Metrology. Additionally, the performance of measures in interlaboratory contexts could have its own singularities. So, the basis of a proper understanding of Chemical Metrology rest on a proper model of errors, applied on different concepts, experimental techniques and statistical approaches. On the other hand, the general model of errors precedes the understanding of the uncertainty concept and how it is applied in Chemical Metrology.

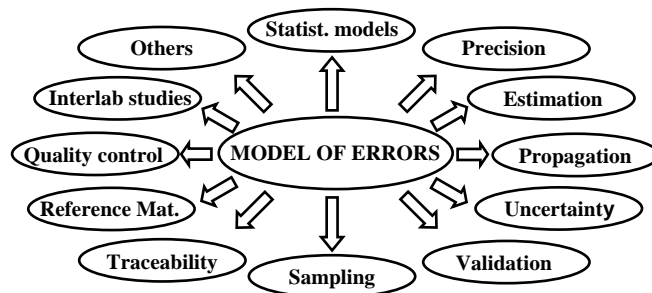


Fig. 5. Importance of starting from a general model of errors: implications on numerous topics.

7. **It is adequate to link performance characteristics of analytical procedures and validation concepts, both in theory and in practical exercises [8].** Nowadays, validation of analytical procedures is fully demanded by professionals of Chemical Metrology. It is a requisite of well organized laboratories, and accreditation bodies have established clear politics on the basis of the most recognized standard concerning general requirements for the testing and calibration laboratories [9]. So, validation of analytical procedures is a daily practice in analytical laboratories. Although conceptually, professionals can understand the essences of the validation process, the best way to get a complete understanding is to meet and solve practical examples from the living laboratory. To attain such goal, the learning process starts reviewing performance characteristics of analytical procedures and related concepts, complemented with practical demonstrative examples. After that, the principles, golden rules and general schemes of validation are discussed. Although validation is approached with a general view, emphasis of internal or single laboratory validation is done. This latter topic is dictated by practical necessities of analytical laboratories. At the end, the students solve practical problems in the course "Computing and Quality".
8. **The understanding of traceability and how it is properly applied to chemical measurements prepares students for organizational actions in the laboratory.** The concept of traceability is not new in Metrology [5]. It plays an important role in every field of measurement. For a long time the concept of traceability has been applied in Analytical Chemistry on the basis of intuition and professional judgment. However, the incorporation of that concept to chemical

metrology has been relatively recent. In order to develop general and particular organizational actions in a laboratory from a consistent point of view, it is necessary to understand the concept of traceability and how it is properly applied to chemical measurements. Nevertheless, traceability of Chemical Metrology has been a discussed topic in the literature. The concept of traceability, the singularities of traceability chains of Chemical Metrology and comparisons with centralized and other traceability chains are presented in the course "Quality of Analytical Laboratories". Additionally, the quality assurance measurements taken in order to attain and preserve traceability are discussed from the precedence of several other important concepts like errors, uncertainty, etc.

9. **Proper understanding of interlaboratory studies is attained on practical basis** [10]. It starts with the explanation of statistical basis; it follows with the understanding of principles and practical role and ends with exercises taken from recognized written standards (v.g., ISO 5725) [11]. Again, reiteration at different levels of complexity is used as a way to reaffirm concepts and practices. That topic is exposed at the end of the course "Quality of Analytical Laboratories". To attain a complete understanding of interlaboratory studies is necessary to know precedent concepts like traceability, certified reference materials, performance characteristics of analytical procedures, validation, etc.
10. Different approaches to estimate uncertainty of analytical measurements are discussed in the classroom with emphasis in comparison between them [6, 12-13]. The metrological approach of uncertainty based on the Theory of Errors is very useful to introduce the topic. Moreover, it has been updated several times in the courses during the last years, because it is under continuous development and improvement at international scale. **Presentation and discussion of different approaches to estimate uncertainty permit a general orientation for activities in the laboratory.**
11. Usually, students know sampling schemes on an empirical basis. **It is very attractive for them to know the principles and role of sampling in the chemical measurement process.** The scientific fundamentals of sampling, sampling plans and sampling protocols are introduced. Students discover the guidelines that support proper sampling processes, both outside or inside the laboratory. Quality assurance and control of sampling close the program to prepare students for practical activities at the field or in the laboratory.
12. **A better comprehension of ISO/IEC 17025 standard is obtained on the basis of practical examples of analytical laboratories** [9]. It is well known that the best way to get proper comprehension of organizational activities is to analyze practical situations presented in actual life. The explanation of a general layout of a quality management system is developed on the basis of examples of solutions and documents that belong to the author's laboratory at the University of Havana.

Although those examples are not be the general solutions for all the possible cases, they constitute proper examples to start useful discussions. Normally, students ask how is possible to adapt the general ideas to their own laboratories, in the search of solutions to be applied in practice. The final result is a very constructive exchange of experiences for everybody. The explanations concerning ISO/IEC 17025 are complemented taking into account the frame of recognized standards and international bodies operating in the mechanisms of multilateral recognition that play roles in the international context [14].

An important related topic with the implementation of ISO/IEC 17025 in the context of an analytical laboratory is the role of documentation in the quality management system. Starting from the ISO 9000 definition of document [15], ideas about conception, development and practical application of a documentation system are discussed in class. Those explanations are complemented with real documents taken from the professors' laboratory. Examples of records, standard operating procedures, manuals, checking lists and several other documents are presented. Emphasis is put on audits and their role in an organized laboratory.

13. **Both generalization and particularization of concepts play an important role in the learning process.** Thus, starting from particular concepts such as laboratory performance study, method performance study and certification study, it is possible to get proper ideas about the general layout and characteristics of interlaboratory studies. Three cases contribute to the general concept. Inversely, starting from a general concept like traceability, through explanations of main practical meanings and practical examples, students get a firm concept on traceability to be applied in practice. Practical examples contribute to understand the general concept and particular cases. (Fig. 6).

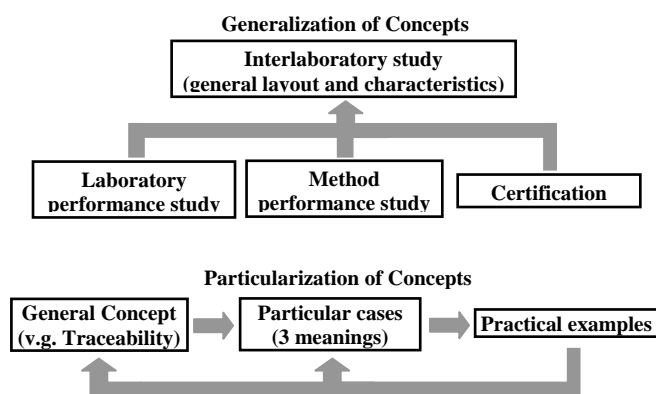


Fig. 6. Generalization and particularization of concepts in the learning process: examples.

14. **It is attractive for students who have received the complete set of courses to present a final dissertation on some subject.** It is possible to select from: i) a collection of practical exercises; ii) a short critical review on a selected topic and iii) a discussion on problems from the student's institution with proposals

of solutions. In general, the last option is the most selected by students. The final dissertations are programmed in a workshop, specially organized at the end of the courses. In this way, the students' experiences are exchanged.

15. An undergraduate course on Chemical Metrology has been introduced in the current curriculum of the Faculty of Chemistry of the University of Havana. It constitutes an optional course to complete the curriculum of Bachelor in Chemistry. It complements the basic courses on Analytical Chemistry, because it introduces the metrological approach. The course is an abbreviated version of the course "Quality of Analytical Laboratories", special for undergraduate students from fourth or fifth year. It is very interesting to know that **undergraduate students are able to understand quite well the metrological basis of Analytical Chemistry and the importance of laboratory management.** Topics like nature and behavior of errors in Analytical Chemistry, importance of validation of analytical procedures, fundamental quality assurance and control techniques and accreditation are very well understood.

#### 4. CONCLUSIONS AND RECOMMENDATIONS

The analyzed experiences could be useful for both undergraduate and postgraduate courses on Analytical Chemistry where Chemical Metrology and quality are important topics. They can contribute in the search of better pedagogic resources to introduce modern concepts and practices concerning quality of analytical laboratories.

Derived experiences from postgraduate teaching have been incorporated in undergraduate programs with good results by means of an optional course. Undergraduates are able to acquire basic knowledge with a metrological approach. However, this step should be conceived as intermediate. The next step could be the full incorporation of the metrological approach from the first course in Analytical Chemistry in future undergraduate programs. This includes, among others, to update undergraduate courses with modern approaches of Theory of Errors and Chemical Metrology and to introduce basic knowledge on quality assurance and laboratory management.

The efforts made to develop a new approach for the teaching of chemical metrology in Latin-America have been very useful. They have contributed to disseminate the metrological approach in our analytical community.

There are several appropriate teaching resources to be exploited in the near future so as to improve the attained levels. The first one could be to implement the courses on line, on the basis of the demand that has been observed. Nowadays, distance learning is an important tendency in higher educational institutions as an additional possibility. Another resource could be to produce computer assisted learning packages to facilitate the assimilation of particular topics. Coursewares could complement the standard approach to teaching Chemical Metrology, but do not

replace it. Its main disadvantage, lack of personal contact between the student and lecturer, should be avoided by means of direct exchanges. Internet should be better exploited to develop student expertise in the retrieval of scientific and technical information in order to solve practical problems. Sources like journals, databases, useful web sites, etc., should be known by practitioners.

#### REFERENCES

- [1] Bell, S.C., Moore, J., "Integration of quality assurance/quality control into quantitative analysis", *Journal of Chemical Education*, Vol. 75, No. 7, pp. 874, 1998.
- [2] Libes, S.M., "Learning quality assurance/quality control using U.S. EPA techniques", *Journal of Chemical Education*, Vol. 76, No. 12, pp. 1 642, 1999.
- [3] Valcárcel, M., "Principios de Química Analítica", Springer-Verlag Ibérica, Barcelona, 1999.
- [4] Houlgate, P., Prichard E., "Effective Ways of Contributing to Taught MSc courses in Analytical Science", *Valid Analytical Measurement (VAM) Report*, 1999.
- [5] NC OIML V2: 1995 Vocabulario internacional de términos generales y básicos de metrología.
- [6] ISO Guide to the expression of uncertainty of measurement, Geneva, 1993, ISBN 92-67-10188-9.
- [7] Thompson, M., "Towards a unified model of errors in analytical measurement", *The Analyst*, Vol. 125, pp. 2 020-2 025, 2000.
- [8] IUPAC, "Harmonized guidelines for single-laboratory validation of methods of analysis", *Pure and Applied Chemistry*, Vol. 74, No. 5, pp. 835, 2002.
- [9] ISO/IEC 17025:2005 Requisitos generales para la competencia de los laboratorios de calibración y ensayo. (Certified translation).
- [10] IUPAC, *Pure and Applied Chemistry*, "Nomenclature of interlaboratory analytical studies", Vol. 66, No. 9, pp. 1903, 1994.
- [11] ISO 5725:1994 Accuracy (trueness and precision) of measurement methods and results.
- [12] EURACHEM/CITAC Guide Quantifying Uncertainty in Analytical Measurements, 2<sup>nd</sup> Ed., 2000.
- [13] Analytical Methods Committee, "Uncertainty of measurement: implications of its use in Analytical Science", *The Analyst*, Vol. 120, pp. 2 303-2 308, 1995.
- [14] ISO/IEC 17000:2004 Evaluación de la conformidad. Vocabulario y principios generales. (Certified translation).
- [15] ISO 9000:2000 Sistemas de gestión de la calidad - Fundamentos y vocabulario (Certified translation).

#### ACKNOWLEDGEMENTS

The authors are grateful for the Organizer Committee of XVIII IMEKO World Congress and Prof. H. S. Brandi because of the facilities they offered so that this contribution could be presented.