the public understanding of chemistry.

So far, the ESDC has had one meeting (at the Royal Institution in London, arguably the historical origin of public understanding of chemistry). It quickly became clear at the meeting that there was one task we had to do if we were to compile a worthwhile report—we had to discover what the members of IUPAC wanted. There are already numerous educational initiatives underway throughout the world, and the ESDC wanted to avoid replication, inappropriate expenditure of effort, and—to express it directly—the treading on of toes. What is there special about IUPAC that can lead it to make a useful, effective, and welcome contribution to chemical education throughout the world? Which of its current activities are wasteful of volunteers' enthusiasm and effort?

In an attempt to gather our stakeholders' views, I have written to a large number of organizations. However, I know that lurking in the world are numerous good ideas. I am, therefore, using the pages of this news magazine to encourage anyone who has a view to write to me. I am particularly interested in imaginative *global* visions. An idea for developing an inexpensive synchrotron storage ring, reusable litmus paper, or whatever, can wait until the newly constituted CTC (if that is our recommendation) is in place; what the committee seeks are *strategic* ideas. Where should IUPAC's educational effort be directed? Where is its current effort wasted? How can it best reach the people who will benefit from its activities? How can IUPAC's activities mesh helpfully and constructively into the infrastructure of national and individual initiatives? Where should it step aside? Where would it be most welcome? Is there a role for IUPAC in contributing to the public understanding of science? How do we deploy the new media? What new media should we anticipate?

In considering these questions (and others like them), we have in mind two sets of slices through our stakeholders. One set divides our constituency into three horizontal bands: secondary education, tertiary education, and the general public (to cover public understanding of chemistry). The second set divides our domain into the developed world, the developing world, and global issues. We are aware, for instance, that in some developed countries, there is a worrying drift away from science and from chemistry in particular. In developing countries, the principal object of concern is perhaps the expansion of the technological base through education. The most obvious global issues are the protection and reclamation of the environment and the encouragement of sustainable development. Views on any aspect of our task—or entirely different ways of approaching the problem—would be most welcome.

The committee is already working hard on a number of issues that we have identified. It will meet again in July, when we hope to be able to work toward compiling at least an interim report. That report will be infinitely more valuable if it includes ideas that reflect what the world really wants rather than what we think it needs. Please write to me or pass on your comments to other members of the committee (see the web site) by the end of May 2000.

Report on FAO/IAEA/AOAC International/IUPAC International Workshop on Principles and Practices of Method Validation, 4–6 November 1999, Budapest, Hungary

Dr. Ales Fajgelj [Quality Assurance Supervisor, International Atomic Energy Agency (IAEA) Laboratories, A-2444 Seibersdorf, Austria; E-mail: A.Fajgelj@iaea.org], Chairman of the IUPAC Interdivisional Working Party on Harmonization of Quality Assurance Schemes for Analytical Laboratories, and Dr. Árpád Ambrus (FAO/IAEA Training and Reference Centre for Food and Pesticide Control, FAO/IAEA Agriculture and Biotechnology Laboratory, P.O. Box 100, A-1400 Vienna, Austria), Chairman of the Scientific Committee, have submitted the following report:

This workshop resulted from the internationally recognized fact that full method validation carried out through an interlaboratory method performance study is an expensive but also a limited exercise. It is impossible to organize interlaboratory studies for all analytical methods in use for determination of analytes in various analyte/matrix combinations. A formal basis for the organization of the workshop was provided by the following:

- Recommendations of the FAO/IAEA Consultants Meeting on Validation of Analytical Methods for Food Control, IAEA, Vienna, 1997,
- IUPAC Project 5/97/8, "Protocol for In-House Method Validation" (Coordinators: R. Wood, M. Thompson, and A. Fajgelj), and
- IUPAC Project 5/2/99, "Preparation and Harmonization of Internationally Harmonized Guidelines for In-House Method Validation" (Coordinators: A. Fajgelj and A. Ambrus).

In all three cases, in-house method validation (single-laboratory method validation) is scientifically and technically presented as an alternative to current internationally accepted method validation practices, namely interlaboratory method performance studies. In-house method validation is described in the IUPAC, AOAC International, and ISO guidance developed in 1988.[1,2] In this respect, the present workshop might be seen as an important event because it actually discussed and established technical guidelines to be followed within

a single laboratory performing method validation. The process makes it necessary to elaborate all technical details; to change the philosophy and, consequently, the international legislation may take some years. In this process, the present workshop was an important milestone.

The aim of the workshop was to bring together scientists and representatives of different agencies, governments, standardization organizations, and accreditation bodies involved in method validation in general or in the acceptance of analytical methods for legislative purposes. Around 120 participants from 34 countries attended the workshop. International organizations [AOAC International, Food and Agriculture Organization of the United Nations (FAO), IAEA, IUPAC, European Commission, EURACHEM, etc.] were also formally represented. Fourteen participants received IUPAC financial support to attend the meeting, and they all actively participated in preparation of the workshop documents or delivered a presentation (oral or poster).

The first day of the workshop was dedicated to presentations (lectures and posters), while on the second day two draft documents were introduced and explained. Both of these documents are available upon request of the following titles:

- *IUPAC Harmonized Guidelines for In-House Validation of Methods of Analysis* (Technical Report), prepared by R. Wood, M. Thompson, and S. Ellison; and
- *Practical Procedures to Validate Method Performance and Results of Analysis of Pesticide and Veterinary Drug Residues, and Trace Organic Contaminants in Food*, a discussion document, prepared by Á. Ambrus, FAO/IAEA.

The third day of the workshop was dedicated to general discussion regarding the quality requirements to be met when analytical methods are validated and specifically to comments and recommendations regarding the two draft documents presented. For logistical reasons, the discussion focused principally on methods for pesticide and veterinary drug residues and for trace organic contaminants in food. In these fields, the use of standardized methods has a strong legislative basis. Nevertheless, the single-laboratory method validation approach is also important for all other analytical methods. In this respect, specific guidance on minimum quality criteria and other requirements will need to be prepared.

Major topics related to validation and subsequent use of analytical methods discussed at the present workshop included the following:

- IUPAC "harmonized guidelines",
- "practical procedure",
- proficiency testing,
- use of collaboratively studied methods,
- uncertainty of analytical measurement, and
- role of level of detection/level of quantitation (LOD/LOQ).

The following recommendations resulted from the workshop.

IUPAC "Harmonized Guidelines" for In-House Validation of Methods of Analysis

- The term "single-laboratory validation" is preferred to "in-house validation".
- Validation criteria recommended should be the minimum necessary to assure method performance for the intended purpose.
- A single-laboratory validation cannot assess between-laboratory variation and will provide an optimistic assessment of interlaboratory variability.
- Quality control (QC) procedures should be used within a laboratory to monitor ongoing conformance to the performance characteristics estimated during validation. These results can be used to refine the estimated performance characteristics of the method.
- Participation in interlaboratory studies enhances method validation and supports comparability of analytical results.
- Analysis of fortified test portions provides an estimate of precision and bias of the analytical method.
- Analysis of samples containing incurred residues provides an estimate of analyte homogeneity after sample processing.
- Where certified reference materials containing incurred residues are not available, determination of efficiency of extraction is beyond the capability of most laboratories.
- Laboratories should agree with clients on method performance to be achieved, including reporting limits.

The basic concept of the document and the approach of in-house method validation based on evaluation of uncertainty sources associated with each specific analytical method were largely accepted, and after revision the document will be sent to IUPAC, AOAC International, International Organization for Standardization (ISO), EURACHEM, and Cooperation on International Traceability in Analyical Chemistry (CITAC) for endorsement. Its publication in *Pure and Applied Chemistry* is expected at the end of 2000, but this document will not represent the end of the complete process. The adoption of this new approach in laboratories and its acceptance by legislative authorities will require some more time.

"Practical Procedure"

In addition to the "harmonized guidelines" and the comments given above, the following points are to be considered by FAO/IAEA expert consultation regarding the "practical procedure":

- The redraft should contain a generic approach to single-laboratory method validation for organic trace analysis.
- Specific aspects relating to pesticide and veterinary drug residues will be contained in appendices.
- The minimum list of analytes will be reconsidered to determine the most appropriate analytes to be included in multiresidue method validation (e.g., compounds that have caused problems in trade).
- The following issues should be considered within the context of the intended use of a method: parameters to be studied, criteria to be used, and number of determinations required to meet criteria.
- The terminology used in the document should be consistent with Codex, ISO, and IUPAC terms, insofar as practical.
- "Practical procedure" is subject to further elaboration by the expert group of the FAO/IAEA. Publication of the document is foreseen during the first half of 2000.

Proficiency Testing

- The "International harmonized protocol for proficiency testing of (chemical) analytical laboratories" defines the criteria for design and evaluation of proficiency tests.
- The participants raised some general concerns regarding the use and interpretation of proficiency test results and requested that these concerns be brought to the attention of accreditation authorities. These items were not intended for inclusion in either the "harmonized guidelines" or the "practical approach" final documents.
- Properly designed proficiency tests can provide information to reduce the necessity for collaborative studies of methods. If laboratories can demonstrate accurate measurement of test analytes in common samples, acceptable method performance and equivalency of methods can be inferred.
- Proficiency testing and QC are distinct procedures and cannot take the place of each other.
- Proficiency test samples should represent the types of samples encountered in actual practice, to the extent that is possible and practical.
- The scale of proficiency testing should be cost-effective for each participating laboratory.
- Coordination of national testing plans or sample exchange schemes can provide a greater range of samples and analytes for proficiency testing. Such coordination is encouraged.
- Careful evaluation of proficiency tests is required to minimize the possibility of misinterpretation of results. It is critical that the limitations of proficiency testing be recognized because registration and accreditation organizations may use results as criteria of laboratory credibility.

Use of Collaboratively Studied Methods

Collaboratively studied methods should be used when such methods are already available, suitable for the purpose intended, and required by clients or regulations.

Uncertainty of Analytical Measurement

- A clear and unambiguous definition of uncertainty of measurement in analytical chemistry is needed.
- Well-defined practical methodology is needed on how to develop meaningful data to assess uncertainty in trace analysis.
- It is especially important that the lay public and laboratory clients understand what uncertainty *means* and *does not mean* in analytical measurement.
- Measurement uncertainty should be estimated, if required, and be available to clients.

Use of LOD/LOQ

LOD and LOQ are variable estimates, the values of which depend on the conditions of measurement and the experience of the analyst. The use of these estimates in client reports can be misleading. In view of this problem, participants requested that the FAO/IAEA expert consultation following the workshop consider, as an alternative, that the lowest calibrated level of the analysis be used in client reports.

Proceedings of the workshop, including most of the presentations (lectures and posters) will be published in a special proceedings series book by the Royal Society of Chemistry (RSC) during the first half of 2000 and can be ordered directly from the RSC.

The decision of the local organizers at the Plant Health and Soil Conservation Station of Budapest to hold the workshop in the amenable environment of the Hungarian Academy of Sciences resulted in three intensive and productive days of meetings.

[1]W. Horwitz. *Pure Appl. Chem.* **60**, 855–867 (1988).
[2] W. D. Pocklington. *Pure Appl. Chem.* **62**, 149–162 (1990).