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For a laboratory to produce consistently reliable data, it must implement an appropriate program of quality-assurance and performance-monitoring procedures. Proficiency testing is one of these procedures. The usual format for proficiency testing schemes in analytical chemistry is based on the distribution of samples of a test material to the participants. Participating laboratories ("participants") generally know the test material has been sent by a proficiency scheme provider, but occasionally the material may be received "blind" (i.e., it is received from a normal customer of the laboratory). The participants analyze the material without knowledge of the correct result and return the result of the measurement to the scheme provider. The provider converts the results into scores that reflect the performance of the participant laboratory. This alerts the participant to unexpected problems that might be present and spurs the management to take whatever remedial action is necessary.

The ethos of this Harmonized Protocol is that proficiency testing should provide information on the fitness-for-purpose of analytical results provided by participants, to assist them in meeting requirements. This can be achieved when:

- criteria for assessing results take fitness-for-purpose into account, so that scores inform participants when they need to improve their performance to satisfy customer (or stakeholder) needs
- the circumstances of proficiency testing are close to those prevailing during routine analysis, so that the outcome represents "real life"
- the method of scoring should be simple and, where at all possible, consistent over the whole realm of analytical measurement, to ensure ready interpretation by participants and customers

While the first consideration of proficiency testing is to provide a basis for self-help for each participant, it would be disingenuous to ignore the fact that other uses are made of proficiency testing results. Participants commonly use their scores to demonstrate competence to potential customers and accreditation assessors, and this has the unfortunate effect of pressurizing analysts to excel in the proficiency tests rather than simply to assess routine procedures. Participants should make every effort to avoid such a tendency as, for the most part, it is impossible for scheme providers to detect or eliminate it. Participants must also be diligent in avoiding any misinterpretation of accumulated scores.

The international standardizing organizations—International, ISO, and IUPAC—cooperated to produce the International Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories. The working group that produced the protocol agreed to revise that protocol in light of recent developments and the experience gained since it was first published in 1993. This revision has been prepared and agreed upon in light of comments received following open consultation.

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Reference Data for the Density and Viscosity of Liquid Aluminum and Liquid Iron

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The available experimental data for the density and viscosity of liquid aluminum and iron have been critically examined with the intention of establishing a density and a viscosity standard. All experimental data have been categorized into primary and secondary data according to the quality of measurement specified by a series of criteria. The proposed standard reference correlations for the density of the aluminum and iron are characterized by standard deviations of 0.65% and 0.77% at the 95% confidence level, respectively. The overall uncertainty in the absolute values of the density is estimated to be one of ±0.7% for aluminum and 0.8% for iron, which is
worse than that of the most optimistic claims but recognizes the unexplained discrepancies between different methods. The standard reference correlations for the viscosity of aluminum and iron are characterized by standard deviations of 13.7% and 5.7% at the 95% confidence level, respectively. The uncertainty in the absolute values of the viscosity of the two metals is thought to be no larger than the scatter between measurements made with different techniques and so can be said to be ±14% in the case of aluminum and ±6% in the case of iron.

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How to Access Structure and Dynamics of Solutions: The Capabilities of Computational Methods (Special Topic Article)

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Every experimental result is only as good as the theoretical model employed for its interpretation. Usually there is a complicated way from the actually measured data to the final results; for example, the determination of a structure: A theoretical model has to be defined, to which the measured data are fitted until the “best possible” agreement is achieved, mostly within a few percent of deviation. Though not too error-prone in the case of highly regular solids, this procedure becomes more difficult with gases (with their high mobility of components) and with liquids (where high mobility is combined with a density similar to solids). Any a priori postulated models can be much too simplified.

The quality of theoretical models plays a pivotal role in the determination of structural parameters, and even more, when other physicochemical phenomena such as reaction dynamics and mechanisms (where all interpretation of measurements depends on a correct structural model plus corresponding mechanistic models) are evaluated.

In this article, the progress of computational chemistry in the treatment of liquid systems is outlined. Emphasis is on the combination of the statistical methods—Monte Carlo and molecular dynamics—with quantum mechanics as the main foundation of this progress. The difficulties of experimental studies of liquid systems without having obtained sophisticated theoretical models describing the structural entities and the dynamical behavior of these liquids demonstrate that chemistry research is in a transition phase, where theory and high-performance computing have not only become a valuable supplement but an essential and almost indispensable component to secure a correct interpretation of measured data.

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One of the ways in which simulations are superior to experiments is that they offer the possibility of easily evaluating any kind of atom–atom pair distribution. In more complex systems (e.g., mixed solvents and solutions simultaneously containing several solute species), this is an enormous advantage over spectroscopic approaches, where only averaged data (e.g., atom-atom distances) can be “seen.” The example shown in this figure illustrates the overlay of various atom–atom radial distribution functions for Ca(II) ion in aqueous ammonia (from A. Tongraar, K. Sagarik, B.M. Rode. Phys.Chem. 4, 628 (2002)).
XML-Based IUPAC Standard for Experimental, Predicted, and Critically Evaluated Thermodynamic Property Data Storage and Capture (ThermoML) (IUPAC Recommendations 2006)

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ThermoML is an Extensible Markup Language (XML)-based new IUPAC standard for storage and exchange of experimental, predicted, and critically evaluated thermophysical and thermochemical property data. The basic principles, scope, and description of all structural elements of ThermoML are discussed. ThermoML covers essentially all thermodynamic and transport property data (more than 120 properties) for pure compounds, multicomponent mixtures, and chemical reactions (including change-of-state and equilibrium reactions). Representations of all quantities related to the expression of uncertainty in ThermoML conform to the Guide to the Expression of Uncertainty in Measurement. The ThermoMLEquation schema for representation of fitted equations with ThermoML is also described and provided as supporting information together with specific formulations for several equations commonly used in the representation of thermodynamic and thermophysical properties. The role of ThermoML in global data communication processes is discussed. The text of a variety of data files (use cases) illustrating the ThermoML format for pure compounds, mixtures, and chemical reactions, as well as the complete ThermoML schema text, are provided as supporting information.

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Letters in Support of the ThermoML Standard

In a linking agreement between Elsevier and the Thermodynamic Research Center at NIST (USA), the ThermoML standard is used to make freely available thermochemical and physical property data connected to published articles. It concerns articles from The Journal of Chemical Thermodynamics (since 2004), Fluid Phase Equilibria, and Thermochimica Acta (since 2005). Thanks to the ThermoML standard, this data is now available in an as universal-as-possible format, to scientists and engineers in industry and university laboratories. A link to the ThermoML record is available with the journal article on <ScienceDirect.com>, while the respective journal article in which the data was reported can be found following a link on the data record. The editors of the journals and Elsevier consider this a major addition to the published content, and so are our authors who are pleased and eager to submit.

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ThermoML will continue to have a positive impact on data activities, including scientific publishing (it is already used by five major journals), development of properties databases, and development of tools to correlate or to accurately predict thermodynamic quantities. I would anticipate that the proposed IUPAC standard will have an important role in the sharing of data between research groups as well as the storage and retrieval of such data. In addition, the standard addresses uncertainties of the data in a way that is consistent with the comprehensive reference document Guide to the Expression of Uncertainty in Measurement published by the International Organization for Standardization. The power of this concept lies in the fact that a complete knowledge of a physical quantity consists of a numerical value, its units, its uncertainty, and its provenance. This concept can help to create information and knowledge management systems that could carry the field of thermodynamics to the next level of intelligent predictive tools.

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JCAMP-DX for Electron Magnetic Resonance (IUPAC Recommendations 2006)

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The Joint Committee on Atomic and Molecular Physical data-Data eXchange (JCAMP-DX) is an evolving, open-ended, machine-independent, self-documenting file format for exchanging and archiving data from computerized laboratory instruments such as spectrometers and diffractometers whose output is commonly represented as spectral (profile) plots, contours, or peak tables. The first JCAMP-DX protocol was designed to meet the need for exchanging infrared spectra between similar instruments of different manufacturers. The present document is the result of ongoing efforts by users and manufacturers to extend JCAMP-DX to other types of instrumental data. This version of JCAMP-DX provides for a description of the file structure to be used to accommodate a very wide range of electron magnetic resonance applications. As much as it is very desirable for instrument data systems to be able to read and write files in a standard format directly, instrument vendors are encouraged to develop JCAMP-DX software for the instruments they currently support. This standard of the JCAMP-DX was further extended to cover Y2K-compatible date strings and good laboratory practice, and the next release will cover the information needed for storing n-dimensional data sets.

Uncertainty Estimation and Figures of Merit for Multivariate Calibration (IUPAC Technical Report)

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This report gives an introduction to multivariate calibration from a chemometrics perspective and reviews the various proposals to generalize the well-established univariate methodology to the multivariate domain. Univariate calibration leads to relatively simple models with a sound statistical underpinning. The associated uncertainty estimation and figures of merit are thoroughly covered in several official documents. However, univariate model predictions for unknown samples are reliable only if the signal is sufficiently selective for the analyte of interest. By contrast, multivariate calibration methods may produce valid predictions also from highly unselective data. A case in point is quantification from near-infrared spectra. With the ever-increasing sophistication of analytical instruments inevitably comes a suite of multivariate calibration methods, each with its own underlying assumptions and statistical properties. As a result, uncertainty estimation and figures of merit for multivariate calibration methods has become a subject of active research, especially in the field of chemometrics.
Guidelines for NMR Measurements for Determination of High and Low pKa Values (IUPAC Technical Report)

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Factors affecting the NMR titration procedures for the determination of pK_a values in strongly basic and strongly acidic aqueous solutions (2 > pH > 0 and 14 > pH > 12) are analyzed. Guidelines for experimental procedure and publication protocols are presented in this report. These include:

- calculation of the equilibrium H^+ concentration in a sample
- avoidance of measurement with glass electrode in highly acidic (basic) solutions
- exclusion of D_2O as a solvent
- use of an individual sample isolated from air for each pH value
- use of external reference and lock compounds
- use of a medium of constant ionic strength with clear indication of the supporting electrolyte and of the way the contribution of any ligand to the ionic strength of the medium is accounted for
- use of the NMR technique in a way that eliminates sample heating to facilitate better sample temperature control (e.g., ^1H-coupled NMR for nuclei other than protons, GD-mode, CPD-mode, etc.)
- use of Me_3NCI/Me_3NOH or KCl/KOH as a supporting electrolyte in basic solution rather than sodium salts in order to eliminate errors arising from NaOH association
- verification of the independence of the NMR chemical shift from background electrolyte composition and concentration
- use of extrapolation procedures

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There is no formal terminology used to describe the scope and use of microtechnology in the clinical laboratory. For many laboratory scientists, the word “microchip” is synonymous with high-density microarrays used primarily for investigating gene expression. This document proposes a system of “categories” and “descriptors” that facilitate the classification of a microfabricated device (MFD) in a way that communicates details of its function and analytical role, and describes the analytical principle involved and the methods and materials used for its manufacture. Adoption of this system would enable scientists to employ four descriptors that clearly delineate the function, analytical role, and chemical or physical principle involved in the device.

www.iupac.org/publications/pac/2006/7803/7803x0677.html

NanoChip® Electronic Microarray, Nanogen Corp., San Diego, CA, USA

Agilent 2100 Bioanalyzer®, Agilent Technologies, Palo Alto, CA, USA

Examples of microfabricated devices (MFD) and descriptions. The MFD categories are: A, microfluidic; B, micro-electronic; C, microarray; D, chemically reactive component-based; E, individually addressable; and F, micro-electromechanical. See full text for details.