Pure Appl. Chem., Vol. 75, No. 8, pp. 1097–1106, 2003.

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

CHEMISTRY AND THE ENVIRONMENT DIVISION
COMMISSION ON SOIL AND WATER CHEMISTRY*

MINIMUM REQUIREMENTS FOR REPORTING ANALYTICAL DATA FOR ENVIRONMENTAL SAMPLES

(IUPAC Technical Report)

Prepared for publication by
HANS EGLI[1,‡], MANOS DASSENAKIS[2], HEMDA GARELICK[3], RENÉ VAN GRIEKEN[4], W. J. G. M. PEIJNENBURG[5], LEO KLASINC[6], WERNER KÖRDEL[7], NICK PRIEST[3], AND TANIA TAVARES[8]

[1]Syngenta Crop Protection AG, CH-4002 Basel, Switzerland; [2]University of Athens, Department of Chemistry, GR-15 771 Athens, Greece; [3]Middlessex University, School of Health and Social Sciences, London, N11 2NQ, UK; [4]Universitaire Instelling Antwerpen, Departement Scheikunde, B-2610 Antwerpen, Belgium; [5]RIVM, Laboratory for Ecotoxicology, NL-3720 BA Bilthoven, The Netherlands; [6]Rudjer Boskovic Institute, HR-10001 Zagreb, Croatia; [7]Fraunhofer Institute for Molecular Biology and Applied Ecology, D-57377 Schmallenberg, Germany; [8]Universidade Federal da Bahia, Instituto de Química, 40.170-290 Salvador, Bahia, Brasil

*Membership of the Commission during the preparation of this report (1992–2001) was as follows:

***Titular Members*:** Y. Shevah (Chairman, 1991–2001); W. J. G. M. Peijnenburg (Secretary, 1989–2001); A. Sabljic (1998–2001); H. Garelick (2000–2001); ***Associate Members*:** H. Egli (1998–2001); W. Kördel (1994–2001); J. Lintelmann (1994–2001); A. Wenzel (2000–2001); ***National Representatives*:** A. Rodrigues de Aquino (Brazil, 1994–2001); P. Schejbal (Czech Republic, 1996–2001); B. M. Mishra (India, 1996–2001); G. Becher (Norway, 2000–2001); A. da Costa Duarte (Portugal, 1994–2001); M. Boybay (Turkey, 2000–2001).

‡Corresponding author

Minimum requirements for reporting analytical data for environmental samples

(IUPAC Technical Report)

Abstract: In view of the significance of environmental analytical data, it is essential that the quality of both sampling strategy and analysis be assured and that procedures used, as well as all relevant additional information, are reported. There is a minimum level of information required in order to guarantee the fitness-for-use of the data. Emanating from discussions on the fundamental problems of the analysis of environmental samples for chemical or biological contaminants, a general guidance is given regarding the minimum information that should be provided to adequately describe the sampling strategy, method of sampling, sample properties, handling between sampling and analysis (including storage conditions, pretreatments, homogenization, subsampling), and the analytical methodology (including calculation and validation procedures). Special attention and specific guidance are given for the environmental compartments soil, pore water, groundwater, inland surface water, sediment, seawater, precipitation water, and air.

1. INTRODUCTION

Environmental analytical data are of major importance as they serve many purposes. They are generated to investigate how human activities influence the environment; to develop, calibrate, and validate environmental models; to test whether standards or quality criteria are exceeded; and to deduce whether there is a potential or actual risk to ecosystems. Given the significant implications of environmental data, it is essential that their quality is assured and supplementary information is published in addition to the analytical results, documenting the data quality and relevant aspects of the sampling and analytical procedures. Appropriate use of the data is possible only with sufficient additional information.

This paper was prepared as a guide for reporting analytical data of environmental samples. In particular, the recommendations list the information that should be provided about sample history and the circumstances that might have affected the results. As will be discussed in more detail in the next section, the overall goal of the additional information to be provided is to enable the reader to judge whether the results are accurate and representative, compare results of different origin, and interpret the results. While the recommendations focus on analytical data of anthropogenic chemicals in the environment (i.e., mainly contaminants or pollutants, resulting from analyses of field samples), they also apply to measured data of other analytes such as radionuclides and biological parameters.

The recommendations list the minimum requirements for reporting considered indispensable by IUPAC irrespective of the analyte(s) and the goal of the study. The given minimum requirements may not be considered sufficient in many cases. If so, the reporting analyst should provide all the information deemed necessary to adequately meet the general principles.

The scope of this guidance paper does not directly address summary papers (i.e., compilations of data that make reference to data published elsewhere). Instead, the paper aims at facilitating such compilations, as all necessary information will be found in the source papers if the proposed recommendations are followed. Thus, the authors of summary papers are encouraged to consider reported original data only if they contain the recommended information.

Editors of journals and reviewers of papers submitted for publications may also take into consideration this guidance paper. They are key players in the entire publication process and, in taking their responsibility for a high scientific standard of published articles seriously, should consider refusing papers containing environmental–analytical results if minimum requirements are not met.

2. BASIC CONSIDERATIONS

Trace analysis in environmental samples is complex and requires considerable skills and experience. All steps of the used methodology must be regularly revalidated to ensure that each result is of acceptable quality (i.e., lies around the true value within a known range of uncertainty). The frequency distribution of the uncertainty must be known, at least roughly. With this in mind, the determination of the fitness-for-use of any analytical method is an important part of any quality assurance system. Published environmental data often attract high attention from the general press and public and may even be used to support advisory or regulatory measures. It is, therefore, in the interest of all involved parties to endeavor to publish only data with a proven quality, known uncertainty, and sufficient additional information about the sample history.

Each analysis starts with taking samples from a much larger environmental entity. Analytes and, in particular, living organisms are seldom either uniformly or randomly distributed in the environment. Instead, their distribution follows patchy or clumped patterns. Hence, all environmental compartments are considerably inhomogeneous, both spatially and temporally, and the inter-sample variability is usually large. To some extent, sampling procedures and analysis of replicate or composite samples may smooth out this "natural variability". Nevertheless, the sampling process has a crucial impact on the final analytical result, and an adequate interpretation of the result cannot be made without knowledge of the sampling process. Between sampling and analysis, the analytes and other parameters can be subjected to alterations which may be influenced by transport and storage conditions as well as the sample preparation procedures (e.g., filtering of water and sieving or drying of soils do influence the subsequent analytical result). Therefore, information concerning all the factors that might have had an impact on the results and/or are relevant for the interpretation of the results is important.

Based on these considerations, the definition of minimum requirements for reporting of environmental analytical data aims to:

- provide guidance about the information to be reported about the samples and the way they were collected, in order to unequivocally document their origin and important characteristics;
- give advice on which details should be reported about the analytical and computational procedures (including all treatments since sampling from the field, that are necessary to demonstrate the validity of the results, including an adequate estimate of the uncertainty);
- define which information and parameters should be recorded and reported and are considered necessary for a sufficient sample description; and
- propose that all factors that may have had an impact on the true or determined concentration of the analyte are reported.

Owing to the variability in the aims and purposes of research projects that involve the analysis of environmental samples for chemical parameters, these recommendations cannot fully and unequivocally include all cases. They are, rather, meant to cover the most common environmental compartments and project types. Other compartments not explicitly covered here, such as sediment water, fog, snow, ice, etc., need a suitable adaptation of these minimum requirements, as do special project types, resulting in more information required in some cases, less in others. More detailed and tailor-made information would be needed if the analyses are part of a specific and comprehensive research project, and less information would suffice in case of emergency situations, for instance, in an oil spill.

3. GENERAL RECOMMENDATIONS

The general principles discussed in this section apply to all results, irrespective of the compartment of origin of the sample, the chemical or biological nature of the analyte, and the method of analysis. In this section, the general recommendations are grouped into two subsections. These are: (1) the sample description subsection, which includes information about the sample and its history prior to collection and (2) the sample analysis subsection, which includes information about all actions conducted on the samples between collection and generation of the final result of the analysis.

3.1 Sample description

The following information should be provided:

Justification of sampling strategy
Before any environmental monitoring program can actually start, the researcher must develop a suitable sampling strategy according to the goal of the study. The selection of sampling sites and times is an important part of this strategy, and should, therefore, be based on rational and explainable principles.

Hydrogeological, topographical, agricultural, industrial, and other aspects may be used for this purpose; the mere accessibility of the site may also be of importance; depending on the aim of the study, emphasis would be put on typical sites or worst-case situations. All considerations made at this stage are relevant and should be reported.

Adequate description of the sampling site location
In addition to the rationale of their selection, the clear location of sampling sites must be reported, preferably by indication of their longitude and latitude. A map may be very useful to visualize the sites. Additionally, for most sample types, the exact spatial sampling site must be specified by indicating depth or height with reference to the surface or the sea level.

Adequate description of the time point and frequency of sampling
As a minimum, the sampling day must be reported. The time of the day may be indicated depending on the goal of the study.

Description of the method of sampling
A description of the technique used for sampling is mandatory, including a description of the equipment and the type of samples (replicate, grab, spatially or temporally composite samples).

This point does not apply in the case of flow-through measurements of concentration with sensors, as no physical sample is taken. In such cases, an adequate description of the experimental set-up is required.

Size of samples
The sample size refers to that portion that was removed from the environmental compartment under investigation.

Given the fact that hardly any environmental compartment is homogeneous, the sample size has to be chosen carefully depending on whether the sampling should reflect or "average-out" the lack of homogeneity. The larger the sample size, the lower is the sample-to-sample variability. Reporting the sample size is, therefore, indispensable.

In case of on-line preconcentration during sampling (e.g., an adsorbance onto a media surface), the efficiency and capacity of the sampling device should be evaluated and reported.

Additional information
Depending on the nature of the analyte and the goal of the study, other site-specific factors may either affect the analytical data or be relevant for their interpretation. Information about such factors should also be reported. This may include climatic conditions (e.g., typical precipitation, and wind direction

and velocity for air studies), meteorological conditions prior to and during sampling, particular topographical situations, type and stage of tide in relation to yearly maximum and minimum (for coastal marine studies), or relevant anthropogenic activities adjacent to the sampling site (land use and agricultural practices, sewage systems, transport facilities, exploitation of resources, industries, waste treatment facilities) of the region.

3.2 Sample analysis

The following information should be provided:

Storage conditions between sampling and analysis
The time and conditions of sample storage between sampling and analysis must be described. This should include the material of the sample containers and preceding cleaning procedures. Moreover, some evidence for sufficient stability of the analyte(s) and important parameters over the storage period must equally be provided.

Pretreatment
Any pretreatment of chemical (addition of buffers, preservatives, etc.) or physical nature (adsorption of the analyte onto cartridges, filtration or centrifugation, sieving, drying of the sample, etc.) must be reported.

Procedure of sample homogenization and subsampling
This point applies to nonhomogenous samples such as soil samples. Reported details of the method used for homogenization and taking subsamples must be sufficient to assess their representativeness. Details of tests for homogeneity, if conducted, should be described.

Description of the analytical methodology
The entire analytical method must be described (if published methods are used, referencing these is sufficient); practical details including description of the equipment should be provided as appears reasonable (i.e., inclusion of details relevant to the outcome of the measurement).

Quantitation and validation procedures
The description of the analytical methodology must include a sufficiently detailed description of the quantitative aspects and the performance of the method. In addition to a proper calibration of the quantitation system, which does not normally include the extraction, purification, concentration, and derivatization steps, validation tests must regularly be conducted on the entire analytical procedure. Such tests (e.g., recovery experiments or analysis of certified reference materials) must be designed to prove that the method delivers accurate results within the uncertainty limits. The following information on the quantitative aspects is necessary:

- a description of the calibration of the analytical system (concentration and chemical nature of internal and external standards, surrogate or isotopic tracers, number of calibration points, standard deviations);
- details of the mathematical procedures used to calculate and statistically interpret the results must be reported;
- number of replicate measurements of the samples;
- the limit of detection of the detector *and* the limits of determination or quantitation of the entire analytical procedure, including the method of its calculation or estimation; and
- an estimate of the uncertainty of the final analytical result including both precision (random errors) and accuracy (i.e., results of recovery tests).

The specific requirements in this section are method-dependent and must be adapted accordingly. For example, a specific counting error should be reported for counting procedures employed (e.g., for radionuclides).

The following rules should be observed for the reporting of analytical data:

Units

SI units, or internationally accepted units derived hereof, should be used, and traditional units such as pounds, acres, inches, and Curies avoided, even in countries where these units are still in common use. Concentrations should be expressed in units such as mol/m^3, ng/dm^3, or ng/L rather than given as fraction numbers such as %, ppm, or ppb. As fractions are not always used in a correct way, they are ambiguous and may lead to misunderstandings.

Rounding

Figures should be rounded to a significant number of digits so that no more than the last of the given digits should be uncertain due to the variability of the method.

Replicates

A clear indication whether the reported results are single measurements or mean averages of replicates is necessary, as well as a clarification of the type of replicates (i.e., replicate samples, replicate analysis, or replicate determination).

Where mean results are reported, the range should be included, and, where the reported result is based on three or more replicates, the standard deviation should be given.

4. SPECIFIC RECOMMENDATIONS

In addition to the general requirements listed above, the following specific recommendations apply to samples from different environmental compartments. For compartments not listed below, the author should adequately adapt the general requirements.

The requirements include procedural descriptions as well as environmental parameters relevant for the assessment and interpretation of the results. As some of the parameters may change during storage, their measurement must either take place immediately after sampling or their stability must be proven as outlined for the analytes in Section 3.2.

4.1 Soil

The following information should be collected and reported in addition to that listed in Section 3.1:

- soil horizon sampled;
- soil texture (particle size range or % clay, % silt, % sand);
- pH, organic carbon content, water content;
- land use (arable, pasture, forest, industry, etc); and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as cation-exchange capacity; redox conditions; water-holding capacity; biological activity; content in sorptive phases such as iron, aluminum, or manganese; type of clay mineral.

4.2 Pore water

The following information should be collected and reported in addition to that listed in Section 3.1:

- depth of origin;
- pH;
- sampling method (pressure, centrifugation, suction cups); and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as dissolved organic carbon (DOC), conductivity, macro-nutrients, redox conditions.

4.3 Groundwater

The following information should be collected and reported in addition to that listed in Section 3.1:

- depth (i.e., distance from soil surface) of water table and depth of the sampling point;
- pH and temperature of the water;
- description of the sampling installation or bore-hole construction;
- details on water treatment (if applicable);
- description of the sampled aquifer (confined/nonconfined, porous rocks/fissured or karstified rocks, permeability); and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as electrolytic conductivity, color, odor, turbidity.

4.4 Inland surface water (including suspended matter)

The following information should be collected and reported (where applicable) in addition to that listed in Section 3.1:

- size of the water body;
- actual and average flow rate in the case of river water;
- precipitation during the study period, notably during the days immediately preceding sampling;
- sampling depth and position;
- pH and temperature of the water;
- separation method, if suspended material is separated (including filter type in case of filtration) or specification if no separation was made; and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as suspended particles, DOC, electrolytic conductivity, ionic strength, concentration of anions and cations, redox potential or concentration of dissolved oxygen, position of main picnocline (thermocline) for lake if known.

4.5 Sediment

The following information should be collected and reported in addition to that listed in Section 3.1:

- "horizon" sampled, height of overlaying water column;
- sediment texture (particle size range or % clay, % silt, % sand);
- organic carbon content, dry matter content; and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as pH, redox potential, organic and/or inorganic carbon content, cation-exchange capacity, biological activity, content in sorptive phases such as iron, aluminum, or manganese, type of clay mineral.

4.6 Seawater

The following information should be collected and reported (where applicable) in addition to that listed in Section 3.1:

- sampling depth and total sea depth at the sampling site;
- distance from shore, longitude, and latitude;
- salinity;
- temperature;
- separation method, if suspended material is separated (including filter type in case of filtration), or specification if no separation was made; and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as suspended particles or transparency, DOC, pH, dissolved oxygen, a statement on the level of biological activity (e.g., concentration of chlorophyll-A), position of main picnocline (thermocline or halocline).

4.7 Rain water

The following information should be collected and reported in addition to that listed in Section 3.1:

- length of time period of collection of water;
- information of whether dry deposition was excluded;
- weather conditions at and during the relevant time period before sampling;
- pH and temperature; and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as suspended particles, DOC, concentration of anions and cations, length of time between rain event.

4.8 Air

The recommendations focus on ambient air analysis, less so on measurements following specific reference methods such as determinations of chemicals in air at workplace or determinations of regulated pollutants at points of emission.

The following information should be collected and reported in addition to that listed in Section 3.1:

- location of sampling point (longitude, latitude, height above ground level, height above sea level);
- air temperature, barometric pressure, relative humidity, wind speed and direction;
- precipitation during the study period, notably during the days immediately preceding sampling;
- sampling devices (container or flow-through equipment, adsorbents or absorbents), including dimensions and technical description;
- for particulate matter: cut-off of the sampling device, filter or impactor type;
- specification of collection efficiency and capacity;
- presampling processes (passage through scrubber, filter, drier);
- duration and rate of sampling; and
- other parameters if known or assumed to impact on the analyte to be determined or relevant for the specific goal of the research project, such as duration of sunshine, local point sources, local airflow obstacles, any other local circumstances that affect the result.

5. REFERENCES

The following list of relevant literature includes some internationally accepted guidance documents and method descriptions for sampling, sample handling, and analytical methods. These guidelines should be taken into consideration when planning and performing a study. The list is not complete, however, it gives access to further documents.

5.1 General

European Commission. *Residues: Guidance for Generating and Reporting Methods of Analysis*, SANCO/3029/99 (1999). <http://europa.eu.int/comm/food/fs/ph_ps/pro/wrkdoc/wrkdoc12_en.pdf>

European Commission. *Guidance Document on Residue Analytical Methods*, SANCO/825/00. <http://europa.eu.int/comm/food/fs/ph_ps/pro/wrkdoc/wrkdoc08_en.pdf>

K. Grasshoff, K. Kremling, M. Ehrhardt (Eds.). *Methods of Seawater Analyses*, 3rd ed., Wiley-VCH, New York (1999).

IUPAC. *Compendium of Analytical Nomenclature*, 3rd ed., J. Inczedy, T. Lengyel, A. M. Ure, Blackwell Science, Oxford (1998). <http://www.iupac.org/publications/analytical_compendium>

T. Cvitas. "Quantities describing composition of mixtures", *Metrologia* **33**, 35–39 (1996).

U.S. EPA. *Ecological Effects Test Guidelines, Data Reporting for Environmental Chemistry Methods*, OPPTS 850.7100 (1996). <www.epa.gov/opptsfrs/OPPTS_Harmonized/850_Ecological_Effects_Test_Guidelines>

U.S. EPA. *Residue Chemistry Test Guidelines, Residue Analytical Method*, OPPTS 860.1340 (1995). <www.epa.gov/opptsfrs/OPPTS_Harmonized/860_Residue_Chemistry_Test_Guidelines>

USPHA. *Standard Methods for the Examination of Water and Wastewater*, 19th ed. Prepared and published jointly by: American Public Health Association, American Water Works Association, Water Environment Federation; Joint editorial board: Arnold E. Greenberg, Andrew D. Eaton, Lenore S. Cleseri, American Public Health Association, Washington, DC, (1995).

HMSO. *The Microbiology of Water 1994: Part 1. Drinking Water*. Report on Public Health and Medical Subjects (Report 71) (1994).

R. W. Weaver, S. Angle, P. Bottomley, D. Bezdicek, S. Smith, A. Tabatabai, A. Wollum (Eds.). *Methods of Soil Analysis: Part 2. Microbiological and Biochemical Properties*, American Society of Agronomy and Soil Science Society of America, Madison, WI (1994).

IUPAC. *Quantities, Units and Symbols in Physical Chemistry*, 2nd ed., I. Mills, T. Cvitas, K. Homann, N. Kallay, K. Kuchitsu. Blackwell Science, Oxford (1993).

P. E. Greeson T. A. Ehlke, G. A. Irwin, B. W. Lium, K. V. Slack. *Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples.* U.S. Geological Survey Techniques of Water-Resources Investigations (1990).

A. Klute (Ed.). *Methods of Soil Analysis: Part 1. Physical and Mineralogical Methods*, American Society of Agronomy and Soil Science Society of America, Madison, WI (1986).

5.2 Sampling and handling of samples

International Organization for Standardization (ISO) Geneva (<www.iso.org>):

Standards "soil quality", notably

ISO 10381 (2002) Soil quality—Sampling—Part 2: Guidance on sampling techniques.

ISO 10381 (2001) Soil quality—Sampling—Part 3: Guidance on safety.

ISO 10381 (1993) Soil quality—Sampling—Part 6: Guidance on the collection, handling and storage of soil for the assessment of aerobic microbial processes in the laboratory.

ISO 11259 (1998) Soil quality—Simplified soil description.

ISO 11464 (1994) Soil quality—Pretreatment of samples for physicochemical analyses.

ISO 15709 (2002) Soil quality—Soil water and the unsaturated zone—definitions, symbols and theory.

Standards "water quality", notably

ISO 5667 (1980/2001) Water quality—Sampling—Parts 1–18: Guidance for various waters and sediment.

Standards "air quality", notably

ISO 8756 (1994) Air quality—Handling of temperature, pressure and humidity data.

ISO 9359 (1989) Air quality—Stratified sampling method for assessment of ambient air quality.

ISO 11222 (2002) Air quality—Determination of the uncertainty of the time average of air quality measurements.

5.3 Quality of analytical methods

EURACHEM/CITAC Guide: Quantifying Uncertainty in Analytical Measurement, 2nd ed., S. L. R. Ellison, M. Rösslein, A. Williams (Eds.) (2000). <www.eurachem.bam.de/guides/quam2.pdf>

EAL/EUROLAB Permanent Liaison Group (PLG). *Validation of Tests Methods: General Principles and Concepts* (1996).

International Organization for Standardization (ISO) Geneva (<www.iso.org>):

ISO 8466 (1990) Water quality—Calibration and evaluation of analytical methods and estimation of performance characteristics—Part 1: Statistical evaluation of the linear calibration function.

ISO 8466 (2001) Water quality—Calibration and evaluation of analytical methods and estimation of performance characteristics—Part 2: Calibration strategy for non-linear second-order calibration functions.

ISO/TR 13843 (2000) Water quality—Guidance on validation of microbiological methods.

ISO 9169 (1994) Air quality—Determination of performance characteristics of measurement methods.

ISO 6879 (1995) Air quality—Performance characteristics and related concepts for air quality measuring methods.

UNEP/IAEA/IOC. *Standard Chemical Methods for Marine Environmental Monitoring*, Reference Methods for Marine Pollution Studies 50, UNEP, Nairobi (1991).