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Minimum requirements for reporting analytical data from marine environmental samples

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 Tanja Tavares[8]

[1] Syngenta Crop Protection AG, CH-4002 Basel, Switzerland

[2] University of Athens, Department of Chemistry, GR-15 771 Athens, Greece
E-mail: edasenak@cc.uoa.gr

[3] Middlesex University, School of Health and Social Sciences, London, N11 2NQ, United Kingdom

[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-Institut für Umweltchemie und Ökotoxikologie, D-57377 Schmallenberg, Germany

[7] Fraunhofer Institute for Molecular Biology and Applied Ecology
D-57377 Schmallenberg, Germany

[8] Universidade Federal de Bahia, Instituto de Quimica
40.170-290 Salvador, Bahia, Brazil

Abstract

In view of the significance of marine environmental analytical data it is essential that the quality of both sampling 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 contaminants, a general guidance is given regarding the minimum information that should be provided to adequately describe the methods of sampling, handling between sampling and analysis and the analytical methodology including calculation and validation procedures.

Keywords: Sea water; Sediments; Sampling; Analysis; Quality.

Introduction

Environmental analytical data are of major importance as they serve many purposes. They often attract high attention from the general press and the public and may even be used to support advisory or regulatory measures. Therefore it is essential that their quality is assured and supplementary information is published in addition to the analytical results. Only with sufficient additional information an appropriate use of the data is possible. The IUPAC Subcommittee on

Chemistry of Environmental Compartments has published a guidance document about the minimum information that should be provided if such analytical data are published (Egli *et al.,* 2003). This paper was prepared as a guide for the reporting of analytical data of marine environmental samples.

Each analysis concerning marine environment starts with taking samples from a much larger physical entity. All environmental compartments are considerably inhomogeneous, both spatially and temporally, and the inter-sample variability is usually large. Nevertheless, the sampling process has a crucial impact on the final analytical result. Between sampling and analysis, the analytes and other parameters can be subjected to alterations due to transport and storage conditions as well as the sample preparation procedures (*e.g.* filtering of water, sieving or drying of sediments) (Strickland and Parsons, 1968; Greeson *et al.*, 1990; UNEP/IAEA/IOC, 1991; USPHA, 1995; Grasshoff *et al.*, 1999).

The present recommendations list the information that should be provided about sample history and the factors that might have affected the results (data and metadata). The overall goal of the additional information to be provided is to enable the reader to judge the accuracy and representativeness of results, to compare results of different origin and to interpret them (Garner *et al.*, 1992; Ott, 1995).

Due to the variability in the aims and purposes of research projects that involve the analysis of marine environmental samples for chemical parameters, these recommendations cannot fully and unequivocally include all cases. They are rather meant to cover the most common project types. More detailed information would be needed if the analysis is part of a specific and comprehensive research project and less information would suffice in case of emergency situations, for instance in an oil spill.

Recommendations

Some general principles 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. They are grouped into two parts: (a) the sample description part, which includes information about the sample and its collection; (b) the sample analysis part, which includes information about all actions conducted on the samples between collection and generation of the final result of the analysis.

Sample description

The following information should be provided:

Adequate location of the sampling site and of the time of sampling
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. Hydrogeological, topographical, agricultural, industrial and other aspects that were used for the selection of sampling sites may also be of importance and should be reported. The sampling day must also 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 (simple, replicate, composite etc.). The report of sample size is also useful as the larger the sample size, the lower is the sample-to-sample variability. In the case of non-homogeneous samples, such as coastal sediments, details of the method used for homogenization and taking sub-samples must be reported. In the case of on-line pre-concentration during sampling *e.g.*, an adsorbance onto a media surface, the efficiency and capacity of sampling device should be evaluated and reported (Keith, 1996; ISO, 2001).

Description of in situ *measurements*
In the case of *in situ* measurements with electrodes or sensors an adequate description of the experimental set-up is required together with the method of calibration and an estimation of the uncertainty of the final analytical result including both precision and accuracy (ISO, 1990 & 2001; Hunt and Wilson, 1986).

Additional information
Information about other site-specific factors that may affect the analytical data or be relevant for their interpretation should also be reported. This may include meteorological conditions prior to and during sampling, type and stage of tide in relation to yearly maximum and minimum or relevant anthropogenic activities adjacent to the sampling site (land use, agricultural practices, sewage systems, transport facilities, exploitation of resources, industries, waste treatment facilities, etc).

Sample analysis

The following information should be provided in the report:

Storage conditions between sampling and analysis
The time and conditions of sample storage must be described. This should include the material of the sample containers and preceding cleaning procedures. Moreover, evidence for sufficient stability of the analyte(s) and any pre-treatment (addition of preservatives, adsorption of the analyte onto cartridges, filtration or centrifugation,drying of the sample, etc.) must be reported. (Strickland and Parsons, 1968; Hunt and Wilson, 1986; UNEP/IAEA/IOC, 1991; Garner *et al.*, 1992; Grasshoff *et al.*, 1999).

Description of the analytical methodology
The entire analytical method must be described in details with the appropriate references. The description must include a sufficiently detailed description of the quantitative aspects and the performance of the method. The following information 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; number of replicate quantitations 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; an estimate of the precision and accuracy of the final analytical result (Hunt and Wilson, 1986; Grasshoff *et al.*, 1990; Greeson *et al.*, 1990; UNEP/IAEA/IOC, 1991; USPHA, 1995; Ellison *et al.*, 2000).

The requirements in this section are method depending and must be adapted accordingly.

In addition to the general requirements, the following specific information should be collected and reported (where applicable) in the cases of seawater and marine sediments:

Seawater
Depth; salinity; temperature; dissolved oxygen; separation method, if suspended material is separated (including filter type in case of filtration), or specification if no separation was made; other parameters if known or assumed to impact on the analyte to be determined such as suspended particles or transparency, dissolved organic carbon (DOC), pH, a statement on the level of biological activity, *e.g.* concentration of chlorophyll-A, position of main picnocline (thermocline or halocline) (UNEP/IAEA/IOC, 1991; Grasshoff *et al.*, 1999).

Sediments
'Horizon' sampled, sediment texture (particle size range or % clay, % silt, % sand); organic and inorganic carbon content; other parameters if known or assumed to impact on the analyte to be determined such as pH, redox potential, cation exchange capacity,biological activity,content in sorptive phases such as iron, aluminium or manganese, type of clay mineral. The results have to be presented as mg/g dry weight (UNEP/IAEA/IOC, 1991; MAP/UNEP, 1992).

The following rules should also 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 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, ng/L, or µg/kg rather than in ratio numbers such as %, ppm, or ppb. These latter pseudo-units are ambiguous and may lead to misunderstandings. The expression of results in mmol/L (nmol or µmol) has to be preferred in the cases of nutrients and trace metals, whereas the mg/L (ng or µg) is better for organic compounds (MAP/UNEP, 1992; Ellison *et al.*, 2000).

Rounding
Figures should be rounded to a significant number of digits; 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 at least three replicates, standard deviation should be given.

Conclusions

The analytical data about marine environment are of high significance as they serve many purposes and can be used to support advisory or regulatory measures. Therefore their appropriate use is possible only when their quality is assured and there is also sufficient additional information. This supplementary information may include detailed description of

sampling and analytical procedures. Specific information is needed for various types of marine environmental samples like seawater or sediments. Significant attention is also needed in the presentation of the results concerning units, accuracy and precision. This paper was prepared as a guide for the proper way of reporting analytical data concerning marine environment. It is part of a wider guide that includes all types of environmental data and shall be published by International Union of Pure and Applied Chemistry (IUPAC). We think that the follow-up of the recommendations included in this paper shall be very useful for the better evaluation and use of environmental data and thus for a better environmental management.

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