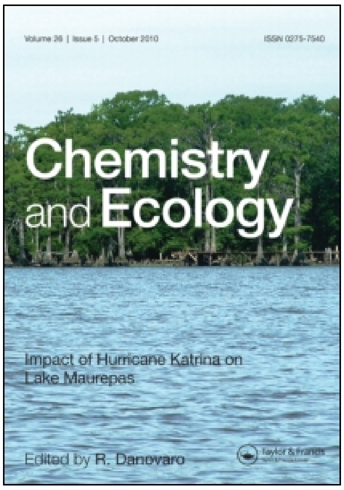

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The Use of Certified Reference Materials in Environmental and Ecological Elemental Analysis

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The Use of Certified Reference Materials in Environmental and Ecological Elemental Analysis

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INTRODUCTION

In the last decade the growth of environmental and ecological analytical chemistry has increased at an unprecedented rate. This can be witnessed by the numerous journals which have appeared covering all aspects of environmental pollution analysis in many interdisciplinary fields. Certainly, a lot of the analytical activity has been spurred by the ever-increasing awareness and concern of pollution, be it of the air, natural waters, marine or soil environments. As a result of this governments in almost all countries in the world have invested a lot of time, money and human resources to its various agencies and universities in trying to identify and control these pollutants.

Of particular significance in environmental monitoring has been, and will continue to be, trace element analyses. As the need increases to reach ultra-trace detection limits with good precision and accuracy the reliability of quality assurance of many older conventional techniques may suffer. Furthermore, the gaining of popularity of some newer analytical techniques (e.g. inductively coupled plasma-atomic emission spectroscopy, (ICP-AES) and proton-induced X-ray emission PIXE), shows a fundamental need to scrutinize their respective analytical limits and reliabilities.

One of the most convincing manners of establishing quality control is by employing certified reference materials prepared by reputable organizations.

It is the intention of this paper to address specifically the use or lack of use of certified reference materials in trace element analyses as it pertains to environmental and ecological monitoring. Previous articles which have discussed the role and merits of various reference materials have been largely written by several authors at the Office of Standard Reference Materials at the National Bureau of Standards (NBS) in Washington D.C. (Uriano and Gravatt, 1977; Alvarez, Raspberry and Uriano, 1982; NBS Special Publication 408, 1975; Cali and Reed, 1976 and all the references therein).

QUALITY CONTROL IN TRACE ELEMENT ANALYSIS

It is very evident that most chemical techniques, being essentially destructive in nature, need very careful evaluations of their respective instrumentation. However, at a time when so many automated systems are available on the chemical analysis market the training of proper researchers and technicians alike is not a trivial undertaking.

Analytical parameters such as stability of the system on a daily basis, precision, accuracy, blank values and limits of detection or limits of quantitation are important criteria which can vary very dramatically from laboratory to laboratory. Even when the same analytical systems are used optimal conditions may vary significantly. Taylor (1981) has effectively outlined the concepts of quality control and quality assessment, while the American Chemical Society (1980) has given very beneficial guidelines for trace element analysis. These include limits of detection and limits of quantitation. These concepts will not be discussed here.

It is unfortunate that results of many inter-laboratory comparisons are either never published or appear in reports which are not readily available. In many cases, results of these comparisons describing the salient features of the different methods used can give a better insight to the various inherent problems associated with different matrices.

It has been pointed out by Brzezinska (1980) that results from interlaboratory comparisons of marine samples conducted in Europe left a lot to be desired. Results of interlaboratory calibration exercises (Bewers *et al.*, 1981) attest that the determination of several key elements in open ocean waters suffers from problems with the analytical stage of the operation. This had led National Research Council of Canada Marine Analytical Chemistry Standards Program (MACSP) to certify a sea-water certified reference material (Berman *et al.*, 1983). At present it appears this is the only one of its kind. Other MACSP reference materials include marine sediments and lobster heptopancreas.

A more disturbing trend has arisen for the quality assurance of many biological samples undertaken by many laboratories for the International Atomic Energy Agency (IAEA). (Dybczynski, Veglia and Suschny, 1980; Parr, 1980; M'Baku and Parr, 1982; Pszonicki, Hanna and Suschny, 1983*a*; 1983*b* and Parr, 1983). IAEA has found out that it was common to have interlaboratory results for many key elements vary by more than order of magnitude. The problem is so acute that many environmental and ecological reference materials have not been certified and have been issued as uncertified reference material or as "information sheets" of trace elemental content.

ORGANIZATIONS FOR THE CERTIFICATION OF STANDARD REFERENCE MATERIAL

Alvarez, Rasberry and Uriano (1982) have pointed out that the National Bureau of Standards must certify more than 250 reference materials in the next five years. Those presently available for environmental, ecological and energy-related trace element research include oyster tissue, wheat and rice flour, brewers yeast, orchard, citrus and tomato leaves, pine needles, bovine liver, various coals, fly ash, water, river and estuarine sediments and urban particulate matter.

This is by far the largest (about nine hundred certified reference materials in all disciplines) but by no means the only organization in the world undertaking certification of environmental and biological reference materials. With so many organizations participating in the certification of various reference materials it is difficult for independent researchers to be aware of what is readily available. This is especially true when commercial advertisements do not always reach the potential customer. However, recently the International Organization for Standards (1982) has acted to publish an international directory of certified reference material producers which lists hundreds of certified reference materials available on a world-wide basis from just over three hundred organizations.

TABLE I

Organizations supplying certified reference environmental biological and ecological reference materials for elemental analysis

Organization	Certified Reference Material for Elemental Composition
Bureau National de Métrologie (B.N.M.) 8–10, rue Crillon 75194 Paris Cedex 04, FRANCE	fuel oil
Canada Centre for Mineral and Energy Technology c/o Coordinator, CANMET 555 Booth St. Ottawa, Ontario K1A O61, CANADA	various soils
Commission of the European Communities Community of Bureau of Reference (BCR) 200, Rue de la Loi B-1049, Brussels, BELGIUM	coal, fly ash, plants, milkpowder
Environmental Research Center 5–1, 1-Chrome, Marunouchi Chiyoda-Ku, Tokyo, JAPAN	pepperbush, pond sediment, mussel, chlorella
International Atomic Energy Agency Analytical Quality Control Services Laboratory Seibersdorf P.O. Box 590 A-1011 Vienna, AUSTRIA	soil, lake sediment, flour, milk-powder, animal bone and blood, muscle, cotton cellulose
National Bureau of Standards (USA) Office of Standard Reference Materials Washington, D.C. 20234, U.S.A.	coal, fly ash, fuel oil, urban particulate, water, river and estuarine sediments, oyster tissue, plant leaves, yeast, flour and bovine liver
The Japan Petroleum Institute 1–1, 1-Chrome, Minami-Aoyama Minato-Ku, Tokyo, JAPAN	fuel oil
Marine Analytical Chemistry Standards Program Chemistry Division National Research Council Montreal Road Ottawa, Ontario K1A OR9, CANADA or Atlantic Research Laboratory 1411 Oxford Street Halifax, N.S. B34 3Z1, CANADA	sea-water, marine sediments, lobster hepatopancreas
U.S. Environmental Protection Agency Environmental Monitoring and Support Laboratory Cincinnati, Ohio, U.S.A.	water

Undoubtedly, such a directory would be an extremely useful asset to researchers in many interdisciplinary studies. A comprehensive list of the organizations which have certified environmental, biological, ecological or energy-related reference materials with respect to trace element analysis is shown in Table I. It may well be that certain organizations may have been left out (e.g. Institute for Technical Research in Sao Paulo, Brazil and Centre for Reference Materials at the National Institute of Metrology in Beijing, China) but as yet it appears that no publicity has appeared regarding the certification of any environmental reference materials.

USE OF CERTIFIED REFERENCE MATERIALS

Gladney (1980*a*; *b*) has done a detailed compilation of elemental concentration data for NBS biological and environmental standard reference material. Concentration data on up to 76 elements in 19 NBS materials have been collected from 325 journal articles and technical reports. It is rather surprising to find out that the single most used technique was not a traditional chemical one, but rather neutron activation analysis. For the 19 reference materials analyzed the percentage of neutron activation analysis as compared to all the other methods ranged from 45%–100%.

Clearly, it appears that researchers employing better known methods such as atomic absorption, polarography, flame emission, ion selective electrodes, for example, have published few reports concerning the testing of their analytical procedures with certified reference materials. In fact, a perusal of many articles dealing with elemental analysis shows that the majority of authors do not subject their analytical techniques to a critical evaluation by using certified reference materials. It is true that in some cases such materials do not presently exist, but as seen in Table I many do.

It would appear that some pressure must be exerted by the editors of the various environmental, ecological and analytical journals to persuade researchers to use certified reference materials as an integral part of their research. As Taylor (1981) has so appropriately pointed out "Many laboratories consider that the 5%–15% extra effort ordinarily required for all aspects of quality assurance that it provides." In this regard it should be emphatically stated that the cost of many of these materials is not expensive. For instance, environmental reference materials supplied by NBS are often in the range between \$60–\$160 (U.S. dollars) or between £35–£100 (Sterling) for many grams of the sample. Similar prices are quoted by the National Research Council of Canada MACPS. IAEA has prices between \$40–\$80 (U.S.) for their materials. Certainly this is almost an insignificant amount compared to the thousands of dollars or pounds spent on equipment, technicians and for example, field trips.

CONCLUSIONS

In conclusion, it can be confidently stated that certified reference materials can be easily obtained by all researchers through the world, including those investigators in the poorer countries. It is therefore the onus of the editors in chief of the journals and referees of the various submitted articles to encourage and persuade (as best as possible) researchers to employ the certified reference materials as an integral part of their research. Perhaps, as a positive first step the editors can incorporate opening remarks at the beginning of each issue stating the policy of the journal as it pertains to the use of certified reference materials. As a final suggestion, the appearance of a journal dedicated exclusively to the publishing of certified reference materials and inter-laboratory comparisons of environmental, biological and ecological importance would be extremely beneficial to the researchers throughout the world. Such a publication now exists for geological reference materials; Geostandards Newsletter.

It has been the intention of the author to provoke constructive thought about the need and use of certified reference materials. Any critical analysis of this paper either in the form of letters either to myself or preferably as rebukes in article form submitted to this journal would be more than welcome. Only in such a way can we, as the research community, arrive at a reasonable and effective way of incorporating certified reference materials in our respective investigations.

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