

# METHODS FOR ENVIRONMENTAL TRACE ANALYSIS

**John R. Dean**

*Northumbria University, Newcastle, UK*





METHODS FOR  
ENVIRONMENTAL  
TRACE ANALYSIS

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**To Lynne, Sam and Naomi**





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# Series Preface

There has been a rapid expansion in the provision of further education in recent years, which has brought with it the need to provide more flexible methods of teaching in order to satisfy the requirements of an increasingly more diverse type of student. In this respect, the *open learning* approach has proved to be a valuable and effective teaching method, in particular for those students who for a variety of reasons cannot pursue full-time traditional courses. As a result, John Wiley & Sons Ltd first published the Analytical Chemistry by Open Learning (ACOL) series of textbooks in the late 1980s. This series, which covers all of the major analytical techniques, rapidly established itself as a valuable teaching resource, providing a convenient and flexible means of studying for those people who, on account of their individual circumstances, were not able to take advantage of more conventional methods of education in this particular subject area.

Following upon the success of the ACOL series, which by its very name is predominately concerned with Analytical *Chemistry*, the *Analytical Techniques in the Sciences* (AnTS) series of open learning texts has now been introduced with the aim of providing a broader coverage of the many areas of science in which analytical techniques and methods are now increasingly applied. With this in mind, the AnTS series of texts seeks to provide a range of books which will cover not only the actual techniques themselves, but *also* those scientific disciplines which have a necessary requirement for analytical characterization methods.

Analytical instrumentation continues to increase in sophistication, and as a consequence, the range of materials that can now be almost routinely analysed has increased accordingly. Books in this series which are concerned with the *techniques* themselves will reflect such advances in analytical instrumentation, while at the same time providing full and detailed discussions of the fundamental concepts and theories of the particular analytical method being considered. Such books will cover a variety of techniques, including general instrumental analysis,

spectroscopy, chromatography, electrophoresis, tandem techniques, electroanalytical methods, X-ray analysis and other significant topics. In addition, books in the series will include the *application* of analytical techniques in areas such as environmental science, the life sciences, clinical analysis, food science, forensic analysis, pharmaceutical science, conservation and archaeology, polymer science and general solid-state materials science.

Written by experts in their own particular fields, the books are presented in an easy-to-read, user-friendly style, with each chapter including both learning objectives and summaries of the subject matter being covered. The progress of the reader can be assessed by the use of frequent self-assessment question (SAQs) and discussion questions (DQs), along with their corresponding reinforcing or remedial responses, which appear regularly throughout the texts. The books are thus eminently suitable both for self-study applications and for forming the basis of industrial company in-house training schemes. Each text also contains a large amount of supplementary material, including bibliographies, lists of acronyms and abbreviations, and tables of SI Units and important physical constants, plus where appropriate, glossaries and references to literature sources.

It is therefore hoped that this present series of textbooks will prove to be a useful and valuable source of teaching material, both for individual students and for teachers of science courses.

*Dave Ando  
Dartford, UK*



# Preface

The field of environmental sample preparation has undergone a revolution in the last twenty five years. What was essentially a series of basic methods and procedures has developed (and continues to develop) into a new exciting area with a strong influence from instrumental approaches. This book essentially covers the traditional approaches of environmental sample preparation for both metals and organic compounds from a range of matrices.

The text is arranged into twelve chapters, covering the essentials of good laboratory housekeeping, through sampling and sample storage, and finally to the relevant sample preparation for inorganic and organic compounds from environmental matrices. A further chapter is devoted to the methods of analysis that can be used for quantitative analysis. To allow the user of the book to perform experiments in an effective manner, guidelines are also offered with respect to record keeping in the laboratory.

In Chapter 1, information is provided with regard to general safety aspects in the laboratory. In addition, specific guidance on the recording of numerical data (with the appropriate units) is provided, with examples on how to display data effectively in the form of tables and figures. Issues relating to sample handling of solids and liquids are also covered. Finally, numerical exercises involving the calculation of dilution factors and their use in calculating original concentrations in environmental samples are provided as worked examples.

Chapter 2 is concerned with the concept of quality assurance and all that it involves with respect to obtaining reliable data from environmental samples. Particular emphasis is placed on the definitions of accuracy and precision. Finally, details on the use of certified reference materials in environmental analysis are provided.

Chapter 3 involves the concept of sampling of representative sample systems. Specific details pertaining to the sampling of soil and sediment, water and air are

provided. Chapter 4 considers the issues associated with the storage and preservation of samples with respect to inorganic and organic pollutants. In particular, focus is given to the retention of chemical species information in environmental matrices.

Chapters 5 and 6 are focused on the specific sample preparation approaches available for the elemental analysis of pollutants from environmental matrices, principally soil and water. Chapter 5 is concerned with the methods available to convert a solid environmental sample into the appropriate form for elemental analysis. The most popular methods are based on the acid digestion of the solid matrix, using either a microwave oven or a hot-plate approach. The growing importance of chemical species information is highlighted with some specific examples. This is then followed by examples of methods to selectively remove the species without destroying its speciation. Details are provided on the methods available for the selective extraction of metal species in soil studies using either a single extraction or a sequential extraction procedure. In addition, a procedure to carry out a physiologically based extraction test on soil is provided. Finally, the role of a simulated gastro-intestinal extraction procedure for extraction of metals in foodstuffs is provided. Chapter 6 provides details of methods for the extraction of metal ions from aqueous samples. Particular emphasis is placed on liquid–liquid extraction, with reference to ion-exchange and co-precipitation.

The focus in Chapters 7 and 8 is on the specific sample preparation approaches available for the extraction of organic compounds from environmental matrices, principally soil and water. Chapter 7 is concerned with the role of Soxhlet, ultrasonic and shake-flask extraction on the removal of organic compounds from solid (soil) matrices. These techniques are contrasted with newer developments in sample preparation for organic compound extraction, namely supercritical fluid extraction, microwave-assisted extraction and pressurized fluid extraction. Chapter 8 is arranged in a similar manner. Initially, details are provided on the use of solvent extraction for organic compounds removal from aqueous samples. This is followed by descriptions of the newer approaches, namely solid-phase extraction and solid-phase microextraction.

Chapter 9 deals with the extraction of volatile compounds from the atmosphere. Particular emphasis is placed here on the methods of thermal desorption and purge-and-trap. Chapter 10 focuses on the methods used to pre-concentrate samples after extraction. In this situation, particular attention is paid to two common approaches, namely rotary evaporation and gas ‘blow-down’, although details of two other methods are also provided.

Chapter 11 details the relevant methods of analysis for both metals and organic compounds. For elemental (metal) analysis, particular attention is given to atomic spectroscopic methods, including atomic absorption and atomic emission spectroscopy. Details are also provided on X-ray fluorescence spectrometry for the direct analysis of metals in solids, ion chromatography for anions in solution, and anodic stripping voltammetry for metal ions in solution. For organic compounds,

particular attention is focused on chromatographic approaches, principally gas chromatography and high performance liquid chromatography. Details are also provided on the use of Fourier-transform infrared spectroscopy for the analysis of total petroleum hydrocarbons.

The final chapter (Chapter 12) provides examples of forms that could be used to record laboratory information at the time of doing the experiment. Guidelines are given for the recording of information associated with sample pre-treatment. Then, specific forms are provided for the recording of sample preparation details associated with inorganic or organic environmental samples. Finally, guidelines are given for the recording of information associated with the analysis of metals and organic compounds. This chapter concludes with a resource section detailing lists of journals, books (general and specific), CD-ROMs, videos and Web addresses that will act to supplement this text.

Finally, I should like to give a special mention to all of the students (both past and present) who have contributed to the development of interest in the field of environmental sample preparation. The achievements have been many and varied across a broad area of environmental sample preparation, but it has all been worthwhile.

*John R. Dean*  
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# Acronyms, Abbreviations and Symbols

AAS	atomic absorption spectroscopy
AC	alternating current
ACN	acetonitrile
ACS	American Chemical Society
A/D	analogue-to-digital (converter)
AE	alcohol ethoxylate
AES	atomic emission spectroscopy
AFS	atomic fluorescence spectroscopy
aMAE	atmospheric microwave-assisted extraction
ANOVA	analysis of variance
APDC	ammonium pyrrolidine dithiocarbamate
AsB	arsenobetaine
AsC	arsenocholine
ASE™	accelerated solvent extraction
ASV	anodic stripping voltammetry
atm	atmosphere (unit of pressure)
BCR	(European) Community Bureau of Reference
BOD	biochemical oxygen demand
BPR	back-pressure regulator (restrictor)
bpt	boiling point
BTEX	benzene–toluene–ethylbenzene–xylene(s) (mixture)
C	coulomb
CCD	charge-coupled device
CE	capillary electrophoresis
CEC	capillary electrochromatography
cGC	capillary gas chromatography

CI	chemical ionization
COD	chemical oxygen demand
COSHH	Control of Substances Hazardous to Health
CRM	Certified Reference Material
CVAAS	cold-vapour atomic absorption spectroscopy
ECD	electron-capture detector (detection)
EDTA	ethylenediaminetetraacetic acid
EI	electron impact
emf	electromotive force
ETAAS	electrothermal (atomization) atomic absorption spectroscopy
eV	electronvolt
EVACS	(automated) evaporative concentration system
FAAS	flame atomic absorption spectroscopy
FID	flame ionization detector (detection)
FL	fluorescence (detection)
FP	flame photometry
FPD	flame photometric detector (detection)
FTIR	Fourier-transform infrared (spectroscopy)
GC	gas chromatography
GFAAS	graphite-furnace atomic absorption spectroscopy
HASAW	Health and Safety at Work (Act)
HCL	hollow-cathode lamp
HPLC	high performance liquid chromatography
HyAAS	hydride-generation atomic absorption spectroscopy
IC	ion chromatography
ICP	inductively coupled plasma
ICP–AES	inductively coupled plasma–atomic emission spectroscopy
ICP–MS	inductively coupled plasma–mass spectrometry
id	internal diameter
IR	infrared
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
J	joule
LC	liquid chromatography
LDR	linear dynamic range
LGC	Laboratory of the Government Chemist
LLE	liquid–liquid extraction
MAE	microwave-assisted extraction
MBT	monobutyltin
MIBK	methylisobutyl ketone (4-methylpentan-2-one)
MIP	microwave-induced plasma
MMAA	monomethylarsonic acid
MS	mass spectrometry

MSD	mass-selective detector
MSPD	matrix solid-phase dispersion
NIST	National Institute of Standards and Technology
NMR	nuclear magnetic resonance (spectroscopy)
OCP	organochlorine pesticide
ODS	octadecylsilane
OPP	organophosphate pesticide
OT(s)	organotin(s)
PAH	polycyclic (polynuclear) aromatic hydrocarbon
PBET	physiologically based extraction test
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo- <i>p</i> -dioxin
PCDF	polychlorinated dibenzofuran
PFE	pressurized fluid extraction
pMAE	pressurized microwave-assisted extraction
PMT	photomultiplier tube
ppb	parts per billion ( $10^9$ )
ppm	parts per million ( $10^6$ )
ppt	parts per thousand ( $10^3$ )
psi	pounds per square inch
PTFE	polytetrafluoroethylene
QFAAS	quartz-furnace atomic absorption spectroscopy
RF	radiofrequency
rms	root mean square
rpm	revolutions per minute
RSC	The Royal Society of Chemistry
SAX	strong anion exchange
SCX	strong cation exchange
SD	standard deviation
SE	standard error
SFC	supercritical fluid chromatography
SFE	supercritical fluid extraction
SI (units)	Système International (d'Unitès) (International System of Units)
SIM	single-ion monitoring
SM&T	Standards, Materials and Testing
SOM	soil organic matter
SPDC	sodium pyrrolidinedithiocarbamate
SPE	solid-phase extraction
SPME	solid-phase microextraction
SRM	Standard Reference Material
TBT	tributyltin
TBTO	tributyltin oxide
TCD	thermal conductivity detector

TGA	thermal gravimetric (thermogravimetric) analysis
TLC	thin layer chromatography
TPH	total petroleum hydrocarbon
TPT	triphenyltin
URL	uniform resource locator
USEPA	United States Environmental Protection Agency
UV	ultraviolet
V	volt
vis	visible
VOA	volatile organic analyte
W	watt
WWW	World Wide Web
XRF	X-ray fluorescence (spectroscopy)
$A_r$	relative atomic mass
$C$	speed of light; concentration
$D$	distribution ratio
$e$	electronic charge
$E$	energy; electric-field strength; fraction of analyte extracted
$E_h$	redox potential
$f$	(linear) frequency; focal length
$F$	Faraday constant
$G$	gravitational constant
$I$	electric current
$K$	partition coefficient
$K_d$	distribution coefficient
$m$	mass
$M_r$	relative molecular mass
$p$	pressure
$Q$	electric charge (quantity of electricity)
$R$	molar gas constant; resistance; correlation coefficient
$R^2$	coefficient of determination
$t$	time; Student factor; statistical (theoretical) significance
$T$	thermodynamic temperature
$V$	electric potential
$z$	ionic charge
$\lambda$	wavelength
$\nu$	frequency (of radiation)
$\sigma$	measure of standard deviation
$\sigma^2$	variance



# About the Author

**John R. Dean, B.Sc., M.Sc., Ph.D., D.I.C., D.Sc., FRSC, CChem,  
Registered Analytical Chemist**

John R. Dean took his first degree in Chemistry at the University of Manchester Institute of Science and Technology (UMIST), followed by an M.Sc. in Analytical Chemistry and Instrumentation at Loughborough University of Technology, and finally a Ph.D. and D.I.C. in Physical Chemistry at Imperial College of Science and Technology (University of London). He then spent two years as a postdoctoral research fellow at the Food Science Laboratory of the Ministry of Agriculture, Fisheries and Food in Norwich, in conjunction with The Polytechnic of the South West in Plymouth. His work there was focused on the development of directly coupled high performance liquid chromatography and inductively coupled plasma–mass spectrometry methods for trace element speciation in foodstuffs. This was followed by a temporary lectureship in Inorganic Chemistry at Huddersfield Polytechnic. In 1988, he was appointed to a lectureship in Inorganic/Analytical Chemistry at Newcastle Polytechnic (now Northumbria University). This was followed by promotion to Senior Lecturer (1990), Reader (1994) and Principal Lecturer (1998). In 1995 he was the recipient of the 23rd Society for Analytical Chemistry (SAC) Silver Medal, and was awarded a D.Sc. (University of London) in Analytical and Environmental Science in 1998. He has published extensively in analytical and environmental science. He is an active member of the Royal Society of Chemistry (RSC) Analytical Division, having served as a member of the Atomic Spectroscopy Group for 15 years (10 as Honorary Secretary), as well as a past Chairman (1997–1999). He has served on the Analytical Division Council for three terms and is currently its Vice-President (2002–2004), as well as the present Chairman of the North-East Region of the RSC (2001–2003).



## Chapter 1

# Basic Laboratory Skills

### Learning Objectives

- To be aware of safety aspects in the laboratory.
- To be able to record, in an appropriate style, practical information accurately.
- To be able to record numerical data with appropriate units.
- To understand the importance of sample handling with respect to both solids and liquids.
- To be able to present data effectively in tables and figures.
- To be able to perform numerical exercises involving dilution factors.

## 1.1 Introduction

All scientific studies involve some aspect of practical work. It is therefore essential to be able to observe and to record information accurately. In the context of environmental analyses, it should be borne in mind that not all practical work is carried out in the laboratory. Indeed it could be argued that the most important aspects of the whole practical programme are done outside the laboratory in the field, as this is the place where the actual sampling of environmental matrices (air, water, soil, etc.) takes place. It is still common practice, however, to transport the acquired sample back to the laboratory for analysis, so knowledge and implementation of the storage conditions and containers to be used are important. Both sampling and sample storage are covered in Chapters 3 and 4, respectively.

## 1.2 Safety Aspects

No laboratory work should be carried out without due regard to safety, both for yourself and for the people around you. While the Health and Safety at Work

Act (1974) provides the main framework for health and safety, it is the Control of Substances Hazardous to Health (COSHH) regulations of 1994 and 1996 that impose strict legal requirements for risk assessment wherever chemicals are used. Within this context, the use of the terms *hazard* and *risk* are very important. A hazardous substance is one that has the ability to cause harm, whereas risk is about the likelihood that the substance may cause harm. Risk is often associated with the quantity of material being used. For example, a large volume of a flammable substance obviously poses a greater risk than a very small quantity. Your laboratory will operate its own safety scheme, so ensure that you are aware of what it is and follow it.

The basic rules for laboratory work (and, as appropriate, for associated work outside the laboratory using chemicals) are as follows:

- Always wear appropriate protective clothing. Typically, this involves a clean laboratory coat fastened up, eye protection in the form of safety glasses or goggles, appropriate footwear (open-toed sandals or similar are inappropriate) and ensure that long hair is tied back. In some circumstances, it may be necessary to put on gloves, e.g. when using strong acids.
- Never smoke, eat or drink in the laboratory.
- Never work alone in a laboratory.
- Make yourself familiar with the fire regulations in your laboratory and building.
- Be aware of the accident/emergency procedures in your laboratory and building.
- Never mouth pipettes – use appropriate devices for transferring liquids.
- Only use/take the minimum quantity of chemical required for your work.
- Use a fume cupboard for hazardous chemicals. Check that it is functioning properly before starting your work.
- Clear up spillages on and around equipment and in your own workspace as they occur.
- Work in a logical manner.
- Think ahead and plan your work accordingly.

### **DQ 1.1**

What is one of the first things that you should consider before starting a laboratory experiment?

*Answer*

*You should make yourself aware of the particular safety aspects that operate in your own laboratory. This includes the position of fire safety equipment, the methods of hazard and risk assessments for the chemicals*