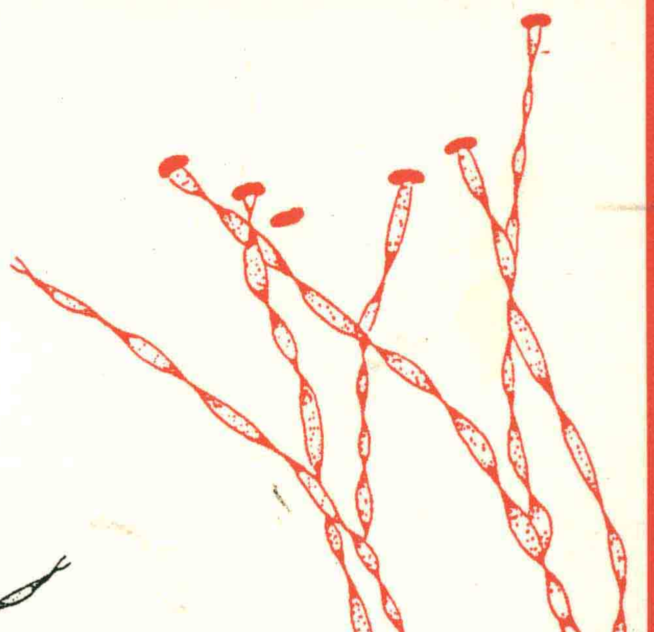
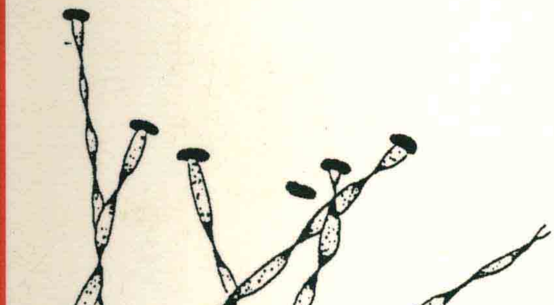
MARIA CSUROS

Environmental Sampling and Analysis for Technicians





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LEWIS PUBLISHERS
Boca Raton Ann Arbor London Tokyo

Library of Congress Cataloging-in-Publication Data

Csuros, Maria.
Environmental sampling & analysis for technicians / Maria Csuros.
p. cm.
Includes bibliographical references and index.
ISBN 0-87371-835-6
1. Pollution—Measurement. 2. Pollution—Measurement—Laboratory manuals. I. Title. II. Title: Environmental sampling and analysis for technicians.
TD193.C78 1994
628'.028'7—dc20 94-865
CIP

Lewis Publishers is an imprint of CRC Press

International Standard Book Number 0-87371-835-6
Library of Congress Card Number 94-865
Printed in the United States of America 3 4 5 6 7 8 9 0
Printed on acid-free paper

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Preface

A clean and healthy environment has always been a concern to most of us. Critical decisions in regard to the protection of our surroundings are based on data collected and derived from laboratory measurements of environmental pollutants.

During the past several years dramatic changes have taken place in environmental regulations, including enhanced laboratory performance and sample collection methods. Well-trained and knowledgeable technical staff is imperative for successful pollution control and environmental management; therefore, we have seen an increase in related educational programs, special training, and refresher courses.

Even though there are many detailed, discipline-specific texts, EPA and DER methodology releases, they give information only for selected areas of the necessary knowledge, and their language makes them unusable for teaching purposes. I have always had to rely on reprints, copies, and other source materials to supplement and support my lectures and laboratory classes. The lack of an extensive textbook in this field prompted me to compile all the information available, combined with my 30 years of laboratory experience, in a book form.

Although the primary audience of this publication is environmental laboratory technician students, I believe that it will be useful as a supplementary information source for more general courses in environmental studies and for a variety of job related training programs. As a practical handbook, it will also assist laboratory technicians in their everyday chores.

All in all, this book will provide a valuable advantage to environmental education and special training programs.

The text contains all required rules in environmental sample collection, related field activities, and proper sample custody. Special emphasis was placed on the philosophy and practical approach of quality assurance and quality control applied in analytical measurements, as established by the Florida Department of Environmental Regulations Quality Assurance Section ("DER Requirements for Quality Assurance" DER-QA-001/91). An overview of the occurrence, source, and fate of toxic pollutants and their control by regulations and standards will provide a helping hand to the readers in understanding analytical reports.

The Author

Maria Csuros is an environmental chemist with many years of varied experience. She received her Master of Science degree from Jozsef Attila University, Szeged, Hungary, in chemistry. She received a grant from the Environmental and Public Health Institution, Budapest, Hungary, for her postgraduate work specializing in environmental chemistry. Most of her professional life revolved around environmental testing laboratories and teaching.

She first served in Hungary at the Environmental and Public Health Laboratory as laboratory supervisor of the Water Department, with her main area of interest in the prevention and elimination of methaemoglobinaemia caused by high nitrate content of private well waters.

She spent six years in Benghasi, Libya, as part of an international team studying the health effects of brackish water quality drinking water.

After immigrating to the United States, she worked as a chemist and laboratory supervisor in private industry, and for the past four years she has dedicated her knowledge and time to Environmental Education. During these years, she has designed and developed a strong Environmental Science program, which is focused on Environmental Sampling and Analysis. Presently she is the coordinator of this program. She also teaches chemistry and environmental science courses.

She also appears at environmental educational seminars for local, state, and federal agencies as well as numerous regional companies.

She lives in Pensacola, Florida, with her husband, Csaba (a professor of microbiology and anatomy and physiology), and their two German shepherds.

She has two sons and four grandsons.

Acknowledgments

It has been a pleasure to thank those who have contributed in their own special way to the completion of this project. I begin with fond words of thanks to my husband, who has given me constant support, stimulating ideas and helpful suggestions in various ways. My warm and very special thanks to my sons, Geza and Zoltan, for their love, encouragement and technical assistance in the preparation of tables and figures.

I gratefully acknowledge the initiation, encouragement and support of Jon Lewis, Lewis Publishers. I appreciate his personal interest in this project, his cheerful spirit and helping hand. I also want to thank Susan Fox, Project Editor, for her careful work and patience, and Shayna Murry, Designer, for the design work.

To all of you, I thank you!

Dedication

To the memory of my mother

Table of Contents

Part 1

Collecting, Preserving, and Handling Environmental Samples

Chapter 1
General Considerations in Sampling

It is an old axiom, that the result of any test procedure can be no better than the sample on which it is performed.

The objective of sampling is to collect a portion of material that represents the actual sample composition. The quality of data depends upon six major activities:

1. formulating the particular objectives for a sampling program
2. collecting representative samples
3. proper sample handling and preservation
4. adhering to adequate chain-of-custody and sample identification
5. participating quality assurance and quality control (QA/QC) in the field
6. properly analyzing the sample

These areas are equally important for insuring that environmental data are of the highest validity and quality.

1.1 Objectives of a Sampling Program

Project Scope and Purpose

The purpose of a project is to be defined, clearly stated in the project plan, and hence in the related sampling program. Well-designed and well-implemented sampling programs are vital to pollution control.

The following six phases are essential to smooth project management:

- planning
- permitting
- compliance
- enforcement
- design
- research and development

Type of Sampling Program

The type of sampling program depends on the program objective, and accordingly, programs may be designed and characterized as follows:

- reconnaissance survey
- point source characterization
- intensive survey
- fixed station monitoring
- network monitoring
- groundwater monitoring
- special surveys

Sampling programs should contain all of the following objectives

- Site identification; include a site map that identifies the sampling locations. The *location of the sampling site is critical to obtain representative samples*
- Sample source (groundwater, drinking water, surface water, wastewater, sediment, soil, etc.)
- Number and matrix of the samples
- Duration of the survey
- Frequency of sampling (monthly, quarterly, etc.)
- Type of samples (grab or composite samples)
- Method of sample collection (manual, automatic)
- Needed analytical parameters with method numbers and references
- Field measurements
- Field quality control (QC) requirements
- Sample collector(s)

Type of Samples

Samples can be divided into four types:

Grab samples — Grab sample is an individual sample, collected at a particular time and place. This type of sample represents conditions at the time it was collected. Therefore, a grab sample should not be used as a basis for a decision about pollution abatement. However, some sources are quite stable in composition, and may be represented well by single grab samples.

Composite samples — Composite sample refers to a mixture of grab samples collected at the same point at different times. A series of smaller samples are collected in a single container and blended for analysis. The mixing process averages the variations in sample composition and minimizes analytical effort and expense. When a time factor is being taken into consideration, grab samples should be collected in suitable sampling intervals, chosen according to the expected changes. When composition depends on location, collect grab samples from appropriate spots. Composite samples reflect the average characteristics during the sampling period and in most cases, a 24 hour period is standard. The volume of the samples should be constant (for example, 200 mls each time) in constant time intervals (for example, every hour), and mixed well at the end of the composite period.

Samples for Volatile Organic Compounds (VOCs), Oil and Grease, Total Recoverable Petroleum Hydrocarbons (TRPHs), and Microbiology testing should never be composited and should only be a grab sample!

Duplicates — Duplicate samples are collected for checking the preciseness of the sampling process.

Split samples — Split samples are taken for checking analytical performance. The sample is taken in one container, mixed thoroughly, and halved into another properly cleaned container. Preserve both samples as needed. Both halves are now samples that represent the same sampling point and are called split samples.

Nature of Sample Collection

Manual Sample Collection

In the case of the lack of automatic samplers, or in case of samples collected for immediate field tests, collect samples directly into the actual container. If the sample cannot be sampled directly in its container, an intermediate vessel should be used. It must be as clean as the sample container and must be made from the required material for that particular parameter. (Selection of the material of the sampling device according to the collected parameters is discussed later). The sample is collected by lowering one properly cleaned device on a rope, pole, or chain into the sample medium. In some cases, it is best to use a pump, either power or hand operated, to withdraw the sample. For most parameters, rinsing the sampling device three times is sufficient, except if the bottles are prepreserved, and are for analyses such as VOCs, Oil and Grease, TRPH, and Microbiology.

Sample Collection with Automatic Samplers

A wide variety of automatic samplers are commercially available. When sampling a large number of locations, the use of automatic samplers are more practical, help to reduce human errors, and are able to keep the samples cool to 4°C during the sampling period. The disadvantage of automatic sampling is the cost of the equipment.

The Material of the Sampling Device Should be Selected According to the Requested Parameters

For *inorganic parameters*, that do not need preservation, use plastic, glass, teflon, stainless steel, aluminum, or brass. For *nutrients*, use plastic, glass, teflon, stainless steel, aluminum, or brass. For *trace metals*, use plastic, stainless steel, or teflon. For *extractable organics*, use glass, aluminum, brass, stainless steel, or teflon. For *volatile organics*, use glass, stainless steel, or teflon. For *microbiological samples*, use a presterilized sample container.

Maintenance and Calibration of Sampling Equipment

To insure proper operation of the automatic samplers, the correct maintenance and calibration (as described in the manufacturer's guide) must be followed. A maintenance log should be used to record all of the activities, such as batteries and desiccant checks, any repair, etc. with the name and signature of the person who performed these activities. After returning from the field, the checked and cleaned sampling equipment should be properly stored.

1.2. General Sampling Rules

Sample collectors must understand and apply the following general rules in sampling:

1. Samples must be collected from the least to the most contaminated sampling locations within the site.

2. Disposable latex gloves should be worn when sampling and new, unused gloves must be used for each separate sampling point. For sampling hazardous materials, *rubber gloves* are recommended.
3. For compositing or mixing samples, use a bowl and spatula to thoroughly mix the sample. For trace organics and metal analysis, the material of these tools should be stainless steel, glass, or teflon. Samples should be mixed thoroughly and sectioned and the quantity of each subsample should be recorded.

The preferred order in sample collection is the following:

1. VOCs
2. Extractable organics, including Oil and Grease and TRPH
3. Total Metals
4. Dissolved Metals
5. Microbiological samples
6. Inorganic nonmetals

For aqueous matrices, sampling equipments and containers are rinsed with sample before the actual sample is taken! Exceptions are samples for VOCs, Oil and Grease, TRPH, Microbiological testing, and any samples collected in prepreserved containers.

Step-by-step, easy to follow, Standard Operation Procedures (SOP) for sampling should be available. Text may include all activities used to collect samples from the arrival on site through delivery to the laboratory. The title, revision date, sections, subsections, and page number(s) of the reference material, used in the preparation of the sampling SOP, should be incorporated.

1.3. Preparation of Sampling Equipments

Written, regulatory outlined step-by-step cleaning procedures, called "DECON" (for decontamination) should be performed. Equipments should be cleaned before sampling, and at the field between samples. At the end of the field trip, sample collection equipments must be labeled as "rinsed, ready to in-house cleaning". After sufficient cleaning in the laboratory, they should be labeled as "in-house cleaned, ready for field" with date and signature of the cleaner. Both house and field cleaning should be documented properly. Detergents specified for cleaning are **ALCONOX** (or equivalent) with <5% phosphate, or **LIQUINOX** (or equivalent) that is phosphate- and ammonia-free.

The solvent to be used in routine cleaning should be pesticide grade isopropanol. Analyte-free water is to be used as rinsing water, and for blanks preparation. The purity and reliability of the analyte-free water is shown by the results of the blank.

Outline for House Cleaning of Sampling Equipments

- Wash with hot soapy tap water and scrub with a brush.
- Rinse thoroughly with hot tap water.
- Rinse with 10 to 15% nitric acid (HNO_3). If nutrients are of interest, after the HNO_3 rinse, 10 to 15% hydrochloric acid (HCl) rinse is required or the HNO_3 rinse may be replaced with HCl rinse. Acid rinse should never be applied to stainless steel or any metallic equipment!
- Rinse thoroughly with deionized water.
- Rinse thoroughly with pesticide-grade isopropanol.
- Rinse thoroughly with analyte free water.
- Air-dry completely.
- Wrap in aluminum foil for storage and transportation.

Outline for Field Cleaning of Sampling Equipments

- Use the same procedure as in-house cleaning, with the exception of hot water.
- First wipe or scrub the equipment to remove particles with the appropriate soap solution, rinse with tap water, followed by deionized water, and finally air-dry.
- For heavily contaminated equipment, use Acetone or Acetone-Hexane-Acetone rinse before regular decon.
- The rinse with analyte-free water is recommended, but optional.
- When only inorganic parameters are of interest, equipment may be rinsed with analyte-free water and with the sample water.
- If proper cleaning of the equipment is impossible, it should be properly disposed until effective cleaning is possible.

Cleaning of Purging Equipments (Submersible Pumps and Non-Teflon Hoses)

Wipe or scrub to remove particles with appropriate soap solution, rinse with tap water, rinse with deionized water, and air dry as long as possible before purging next well. Care should be taken to completely clean the exterior of the pump, and the exterior and interior surfaces of tubing.

Decontamination of Teflon Tubing

Always clean in the laboratory and never in the field.

- Soak tubing in hot soapy water and use a brush to remove any particulate if necessary.
- Rinse tubing exterior and ends liberally with tap water.
- Rinse tubing surfaces and ends with 10 to 15% HNO_3.
- Rinse with tap water.
- Rinse with pesticide grade methanol or isopropanol.
- Rinse with analyte-free water.
- Place tubing on clean aluminum foil.
- With teflon inserts, connect all of the hose used on site. Using the field-use peristaltic pump, assemble the system used in the field, but use a larger size bottle that has the same cap size as the collection bottles. (A large size bottle such as the type containing solvents or acids is suitable.)
- Pump copious amounts of hot, soapy water through the connected tubing. Follow this with tap water.
- With the pump running, draw at least 1 liter of 1+1 HNO_3 through the tubing. Close valve, and stop the pump. Let the acid remain in the tubing for 15 to 20 minutes. Pump an additional 1 to 2 liters of acid through the tubing, followed by 1 to 2 liters of tap water.
- Pump 1 liter of pesticide grade methanol or isopropanol through the tubing. Let solvent remain in the tubing 15 to 20 minutes. Pump an additional 1 to 2 liters of solvent through the system.
- Finally, rinse with 2 to 3 liters of analyte-free water.
- Leave the teflon inserts between the pre-cut lengths and cap or connect the remaining end.
- After the interior has been sufficiently cleaned, the exterior needs a final rinse with analyte-free water.
- Wrap in aluminum foil and store in a clean, dry area. Label with the date of cleaning.

Documentation of this cleaning should be in a bound notebook.

1.4. Preparation of Sample Containers

The material that the sample container is composed of should be chosen so it will not react with the sample. It should be resistant to leakage and breakage and should have the proper volume necessary for the analyte(s) of interest. Plastic containers are the best for sampling inorganic parameters. The containers must have tight screw-type lids. Glass and teflon containers with teflon lined caps are suitable for organic analytes. However, there are some disadvantages. Glass is breakable and teflon is quite expensive. For purgeable organics, use 40 ml borosilicate glass vials with screw cap and teflon backed silicon septum. Sterile plastic cups, individually wrapped, or sterile whirl-pack plastic bags are commercially available for microbiological samples.

Sample containers may be cleaned by the sampling organization or purchased from commercial vendors as precleaned containers. All records for these containers (lot numbers, certification statements, date of receipt, etc.) and their uses must be documented.

Cleaning Procedures for Sample Containers

These regulated cleaning procedures must be strictly followed to eliminate sample contamination by the sample container.

Physical Properties and Mineral Analysis

Bottle type: Plastic or glass, minimum of half gallon capacity.

Soap: LIQUINOX or equivalent.

- Wash bottles and caps with hot soapy water, and rinse liberally with tap water until suds are no longer present.
- Rinse bottles and caps with laboratory pure water at least 3 to 5 times. Drain and store tightly capped until used.

Nutrients, Demands, and Radiological Analysis

Bottle type: Plastic or glass.

Soap: LIQUINOX or equivalent (phosphate- and ammonia-free).

- Wash bottles and caps in hot soapy water and rinse liberally with tap water until soap suds are no longer present.
- Rinse bottles and caps with 1+1 HCl, then follow by rinsing 3 to 5 times with laboratory pure water.
- Drain and store bottles tightly capped until use.

Metals

Bottle type: Plastic bottle with lid.

Soap: Should be metal free ACATIONOX or equivalent.

- Wash bottles and caps in hot soapy water, and rinse liberally with tap water, until soap suds are gone.
- Rinse bottles and caps with 1+1 HCl, followed by tap water rinse.
- Rinse bottles and caps with 1+1 HNO_3. Rinse three times with liberal amount of laboratory pure water.
- Drain and cap tightly until use.

Extractable Organics

Bottle type: 1 liter narrow necked glass bottle with Teflon lined caps. **Plastic bottles and plastic or rubber lined caps are not acceptable!**

Soap: ALCONOX or equivalent.