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# PURIFICATION OF LABORATORY CHEMICALS

FIFTH EDITION

# 实验室化学品的纯化 第5版

Wilfred L.F. Armarego • Christina L.L. Chai

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# PURIFICATION OF LABORATORY CHEMICALS

## Fifth Edition

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## Preface to the Fifth Edition

THE DEMAND for **Purification of Laboratory Chemicals** has not abated since the publication of the fourth edition as evidenced by the number of printings and the sales. The request by the Editor for a fifth edition offered an opportunity to increase the usefulness of this book for laboratory purposes. It is with deep regret that mention should be made that Dr Douglas D. Perrin had passed away soon after the fourth edition was published. His input in the first three editions was considerable and his presence has been greatly missed. A fresh, new and young outlook was required in order to increase the utility of this book and it is with great pleasure that Dr Christina L.L. Chai, a Reader in Chemistry and leader of a research group in organic and bio-organic chemistry, has agreed to coauthor this edition. The new features of the fifth edition have been detailed below.

Chapters 1 and 2 have been reorganised and updated in line with recent developments. A new chapter on the 'Future of Purification' has been added. It outlines developments in syntheses on solid supports, combinatorial chemistry as well as the use of ionic liquids for chemical reactions and reactions in fluorous media. These technologies are becoming increasingly useful and popular so much so that many future commercially available substances will most probably be prepared using these procedures. Consequently, a knowledge of their basic principles will be helpful in many purification methods of the future.

Chapters 4, 5 and 6 (3, 4 and 5 in the 4th edn) form the bulk of the book. The number of entries has been increased to include the purification of many recent commercially available reagents that have become more and more popular in the syntheses of organic, inorganic and bio-organic compounds. Several purification procedures for commonly used liquids, e.g. solvents, had been entered with excessive thoroughness, but in many cases the laboratory worker only requires a simple, rapid but effective purification procedure for immediate use. In such cases a **Rapid purification** procedure has been inserted at the end of the respective entry, and should be satisfactory for most purposes. With the increased use of solid phase synthesis, even for small molecules, and the use of reagents on solid support (e.g. on polystyrene) for reactions in liquid media, compounds on solid support have become increasingly commercially available. These have been inserted at the end of the respective entry and have been listed in the General Index together with the above rapid purification entries.

A large number of substances are ionisable in aqueous solutions and a knowledge of their ionisation constants, stated as pK (pKa) values, can be of importance not only in their purification but also in their reactivity. Literature values of the pK's have been inserted for ionisable substances, and where values could not be found they were estimated ($pK_{Est}$). The estimates are usually so close to the true values as not to affect the purification process or the reactivity seriously. The book will thus be a good compilation of pK values for ionisable substances.

Almost all the entries in Chapters 4, 5 and 6 have CAS (Chemical Abstract Service) Registry Numbers to identify them, and these have been entered for each substance. Unlike chemical names which may have more than one synonymous name, there is only one CAS Registry Number for each substance (with only a few exceptions, e.g. where a substance may have another number before purification, or before determination of absolute configuration). To simplify the method for locating the purification of a substance, a CAS Registry Number Index with the respective page numbers has been included after the General Index at the end of the book. This will also provide the reader with a rapid way to see if the purification of a particular

substance has been reported in the book. The brief General Index includes page references to procedures and equipment, page references to abbreviations of compounds, e.g. TRIS, as well as the names of substances for which a Registry Number was not found.

Website references for distributors of substances or/and of equipment have been included in the text. However, since these may be changed in the future we must rely on the suppliers to inform users of their change in website references.

We wish to thank readers who have provided advice, constructive criticism and new information for inclusion in this book. We should be grateful to our readers for any further comments, suggestions, amendments and criticisms which could, perhaps, be inserted in a second printing of this edition. In particular, we thank Professor Ken-chi Sugiura (Graduate School of Science, Tokyo Metropolitan University, Japan) who has provided us with information on the purification of several organic compounds from his own experiences, and Joe Papa BS MS (EXAXOL in Clearwater, Florida, USA) who has provided us not only with his experiences in the purification of many inorganic substances in this book, but also gave us his analytical results on the amounts of other metal impurities at various stages of purification of several salts. We thank them graciously for permission to include their reports in this work. We express our gratitude to Dr William B. Cowden for his generous advice on computer hardware and software over many years and for providing an Apple LaserWriter (16/600PS) which we used to produce the master copy of this book. We also extend our sincere thanks to Dr Bart Eschler for advice on computer hardware and software and for assistance in setting up the computers (iMac and eMac) used to produce this book.

We thank Dr Pauline M. Armarego for assistance in the painstaking task of entering data into respective files, for many hours of proofreading, correcting typographical errors and checking CAS Registry Numbers against their respective entries.

One of us (W.L.F.A) owes a debt of gratitude to Dr Desmond (Des) J. Brown of the Research School of Chemistry, ANU, for unfailing support and advice over several decades and for providing data that was difficult to acquire not only for this edition but also for the previous four editions of this book.

One of us (C.L.L.C) would specially like to thank her many research students (past and present) for their unwavering support, friendship and loyalty, which enabled her to achieve what she now has. She wishes also to thank her family for their love, and would particularly like to dedicate her contribution towards this book to the memory of her brother Andrew who had said that he should have been a scientist.

We thank Mrs Joan Smith, librarian of the Research School of Chemistry, ANU, for her generous help in many library matters which have made the tedious task of checking references more enduring.

W.L.F. Armarego & C.L.L. Chai
November 2002

## Preface to the First Edition

WE BELIEVE that a need exists for a book to help the chemist or biochemist who wishes to purify the reagents she or he uses. This need is emphasised by the previous lack of any satisfactory central source of references dealing with individual substances. Such a lack must undoubtedly have been a great deterrent to many busy research workers who have been left to decide whether to purify at all, to improvise possible methods, or to take a chance on finding, somewhere in the chemical literature, methods used by some previous investigators.

Although commercially available laboratory chemicals are usually satisfactory, as supplied, for most purposes in scientific and technological work, it is also true that for many applications further purification is essential.

With this thought in mind, the present volume sets out, firstly, to tabulate methods, taken from the literature, for purifying some thousands of individual commercially available chemicals. To help in applying this information, two chapters describe the more common processes currently used for purification in chemical laboratories and give fuller details of new methods which appear likely to find increasing application for the same purpose. Finally, for dealing with substances not separately listed, a chapter is included setting out the usual methods for purifying specific classes of compounds.

To keep this book to a convenient size, and bearing in mind that its most likely users will be laboratory-trained, we have omitted manipulative details with which they can be assumed to be familiar, and also detailed theoretical discussion. Both are readily available elsewhere, for example in Vogel's very useful book **Practical Organic Chemistry** (Longmans, London, 3rd ed., 1956), or Fieser's **Experiments in Organic Chemistry** (Heath, Boston, 3rd ed, 1957).

For the same reason, only limited mention is made of the kinds of impurities likely to be present, and of the tests for detecting them. In many cases, this information can be obtained readily from existing monographs.

By its nature, the present treatment is not exhaustive, nor do we claim that any of the methods taken from the literature are the best possible. Nevertheless, we feel that the information contained in this book is likely to be helpful to a wide range of laboratory workers, including physical and inorganic chemists, research students, biochemists, and biologists. We hope that it will also be of use, although perhaps to only a limited extent, to experienced organic chemists.

We are grateful to Professor A. Albert and Dr D.J. Brown for helpful comments on the manuscript.

D.D.P., W.L.F.A. & D.R.P.
1966

## Preface to the Second Edition

SINCE the publication of the first edition of this book there have been major advances in purification procedures. Sensitive methods have been developed for the detection and elimination of progressively lower levels of impurities. Increasingly stringent requirements for reagent purity have gone hand-in-hand with developments in semiconductor technology, in the preparation of special alloys and in the isolation of highly biologically active substances. The need to eliminate trace impurities at the micro- and nanogram levels has placed greater emphasis on ultrapurification technique. To meet these demands the range of purities of laboratory chemicals has become correspondingly extended. Purification of individual chemicals thus depends more and more critically on the answers to two questions - Purification from what, and to what permissible level of contamination. Where these questions can be specifically answered, suitable methods of purification can usually be devised.

Several periodicals devoted to ultrapurification and separations have been started. These include "Progress in Separation and Purification" Ed. (vol. 1) E.S. Perry, Wiley-Interscience, New York, vols. 1-4, 1968-1971, and **Separation and Purification Methods** Ed. E.S.Perry and C.J.van Oss, Marcel Dekker, New York, vol. 1- , 1973-. Nevertheless, there still remains a broad area in which a general improvement in the level of purity of many compounds can be achieved by applying more or less conventional procedures. The need for a convenient source of information on methods of purifying available laboratory chemicals was indicated by the continuing demand for copies of this book even though it had been out of print for several years.

We have sought to revise and update this volume, deleting sections that have become more familiar or less important, and incorporating more topical material. The number of compounds in Chapters 3 and 4 have been increased appreciably. Also, further details in purification and physical constants are given for many compounds that were listed in the first edition.

We take this opportunity to thank users of the first edition who pointed out errors and omissions, or otherwise suggested improvements or additional material that should be included. We are indebted to Mrs S.Schenk who emerged from retirement to type this manuscript.

D.D.P., W.L.F.A. & D.R.P.
1980

## Preface to the Third Edition

THE CONTINUING demand for this monograph and the publisher's request that we prepare a new edition, are an indication that **Purification of Laboratory Chemicals** fills a gap in many chemists' reference libraries and laboratory shelves. The present volume is an updated edition which contains significantly more detail than the previous editions, as well as an increase in the number of individual entries and a new chapter.

Additions have been made to Chapters 1 and 2 in order to include more recent developments in techniques (e.g. Schlenk-type, *cf* p. 10), and chromatographic methods and materials. Chapter 3 still remains the core of the book, and lists in alphabetical order relevant information on *ca* 4000 organic compounds. Chapter 4 gives a smaller listing of *ca* 750 inorganic and metal-organic substances, and makes a total increase of ca 13% of individual entries in these two chapters. Some additions have also been made to Chapter 5.

We are currently witnessing a major development in the use of physical methods for purifying large molecules and macromolecules, especially of biological origin. Considerable developments in molecular biology are apparent in techniques for the isolation and purification of key biochemicals and substances of high molecular weight. In many cases something approaching homogeneity has been achieved, as evidenced by electrophoresis, immunological and other independent criteria. We have consequently included a new section, Chapter 6, where we list upwards of 100 biological substances to illustrate their current methods of purification. In this chapter the details have been kept to a minimum, but the relevant references have been included.

The lists of individual entries in Chapters 3 and 4 range in length from single line entries to *ca* one page or more for solvents such as acetonitrile, benzene, ethanol and methanol. Some entries include information such as likely contaminants and storage conditions. More data referring to physical properties have been inserted for most entries [i.e. melting and boiling points, refractive indexes, densities, specific optical rotations (where applicable) and UV absorption data]. Inclusion of molecular weights should be useful when deciding on the quantities of reagents needed to carry out relevant synthetic reactions, or preparing analytical solutions. The Chemical Abstracts registry numbers have also been inserted for almost all entries, and should assist in the precise identification of the substances.

In the past ten years laboratory workers have become increasingly conscious of safety in the laboratory environment. We have therefore in three places in Chapter 1 (pp. 3 and 33, and bibliography p. 52) stressed more strongly the importance of safety in the laboratory. Also, where possible, in Chapters 3 and 4 we draw attention to the dangers involved with the manipulation of some hazardous substances.

The world wide facilities for retrieving chemical information provided by the Chemical Abstract Service (CAS on-line) have made it a relatively easy matter to obtain CAS registry numbers of substances, and most of the numbers in this monograph were obtained *via* CAS on-line. We should point out that two other available useful files are CSCHEM and CSCORP which provide, respectively, information on chemicals (and chemical products) and addresses and telephone numbers of the main branch offices of chemical suppliers.

The present edition has been produced on an IBM PC and a Laser Jet printer using the **Microsoft Word (4.0)** word-processing program with a set stylesheet. This has allowed the use of a variety of fonts and font sizes which has made the presentation more attractive than in the previous edition. Also, by altering the format and increasing slightly the sizes of the pages, the length of the monograph has been reduced from 568 to 391 pages. The reduction in the number of pages has been achieved in spite of the increase of *ca* 15% of total text.

We extend our gratitude to the readers whose suggestions have helped to improve the monograph, and to those who have told us of their experiences with some of the purifications stated in the previous editions, and in particular with the hazards that they have encountered. We are deeply indebted to Dr M.D. Fenn for the several hours that he has spent on the terminal to provide us with a large number of CAS registry numbers.

This monograph could not have been produced without the expert assistance of Mr David Clarke who has spent many hours to load the necessary fonts in the computer, and for advising one of the authors (W.L.F.A.) on how to use them together with the idiosyncrasies of Microsoft Word.

D.D.P. & W.L.F.A.
1988

## Preface to the Fourth Edition

THE AIMS of the first three editions, to provide purification procedures of commercially available chemicals and biochemicals from published literature data, are continued in this fourth edition. Since the third edition in 1988 the number of new chemicals and biochemicals which have been added to most chemical and biochemical catalogues have increased enormously. Accordingly there is a need to increase the number of entries with more recent useful reagents and chemical and biochemical intermediates. With this in mind, together with the need to reorganise and update general purification procedures, particularly in the area of biological macromolecules, as well as the time lapse since the previous publication, this fourth edition of **Purification of Laboratory Chemicals** has been produced. Chapter 1 has been reorganised with some updating, and by using a smaller font it was kept to a reasonable number of pages. Chapters 2 and 5 were similarly altered and have been combined into one chapter. Eight hundred and three hundred and fifty entries have been added to Chapters 3 (25% increase) and 4 (44% increase) respectively, and four hundred entries (310% increase) were added to Chapter 5 (Chapter 6 in the Third Edition), making a total of 5700 entries; all resulting in an increase from 391 to 529 pages, i.e. by *ca* 35%.

Many references to the original literature have been included remembering that some of the best references happened to be in the older literature. Every effort has been made to provide the best references but this may not have been achieved in all cases. Standard abbreviations, listed on page 1, have been used throughout this edition to optimise space, except where no space advantage was achieved, in which cases the complete words have been written down to improve the flow of the sentences.

With the increasing facilities for information exchange, chemical, biochemical and equipment suppliers are making their catalogue information available on the Internet , e.g. Aldrich-Fluka-Sigma catalogue information is available on the World Wide Web by using the address http://www.sigma.sial.com, and GIBCO BRL catalogue information from http://www.lifetech.com, as well as on CD-ROMS which are regularly updated. Facility for enquiring about, ordering and paying for items is available *via* the Internet. CAS on-line can be accessed on the Internet, and CAS data is available now on CD-ROM. Also biosafety bill boards can similarly be obtained by sending SUBSCRIBE SAFETY John Doe at the address "listserv@uvmvm.uvm.edu", SUBSCRIBE BIOSAFETY at the address "listserv@mitvma.mit.edu", and SUBSCRIBE RADSAF at the address "listserv@romulus.ehs.uiuc.edu"; and the Occupational, Health and Safety information (Australia) is available at the address "http://www.worksafe.gov.au/~wsa1". Sigma-Aldrich provide Material Safety data sheets on CD-ROMs.

It is with much sadness that Dr Douglas D. Perrin was unable to participate in the preparation of the present edition due to illness. His contributions towards the previous editions have been substantial, and his drive and tenacity have been greatly missed.

The Third Edition was prepared on an IBM-PC and the previous IBM files were converted into Macintosh files. These have now been reformatted on a Macintosh LC575 computer and all further data to complete the Fourth Edition were added to these files. The text was printed with a Hewlett-Packard 4MV -600dpi Laser Jet printer which gives a clearer resolution.

I thank my wife Dr Pauline M. Armarego, also an organic chemist, for the arduous and painstaking task of entering the new data into the respective files, and for the numerous hours of proofreading as well as the corrections of typographic errors in the files. I should be grateful to my readers for any comments, suggestions, amendments and criticisms which could, perhaps, be inserted in the second printing of this edition.

W.L.F. Armarego
30 June 1996

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# CHAPTER 1

# COMMON PHYSICAL TECHNIQUES USED IN PURIFICATION

## INTRODUCTION

Purity is a matter of degree. Other than adventitious contaminants such as dust, paper fibres, wax, cork, etc., that may have been incorporated into the sample during manufacture, all commercially available chemical substances are in some measure impure. Any amounts of unreacted starting material, intermediates, by-products, isomers and related compounds may be present depending on the synthetic or isolation procedures used for preparing the substances. Inorganic reagents may deteriorate because of defective packaging (glued liners affected by sulfuric acid, zinc extracted from white rubber stoppers by ammonia), corrosion or prolonged storage. Organic molecules may undergo changes on storage. In extreme cases the container may be incorrectly labelled or, where compositions are given, they may be misleading or inaccurate for the proposed use. Where any doubt exists it is usual to check for impurities by appropriate spot tests, or by recourse to tables of physical or spectral properties such as the extensive infrared and NMR libraries published by the Sigma Aldrich Chemical Co.
The important question, then, is not whether a substance is pure but whether a given sample is sufficiently pure for some intended purpose. That is, are the contaminants likely to interfere in the process or measurement that is to be studied. By suitable manipulation it is often possible to reduce levels of impurities to acceptable limits, but absolute purity is an ideal which, no matter how closely approached, can never be attained. A *negative* physical or chemical test indicates only that the amount of an impurity in a substance lies below a certain sensitivity level; no test can demonstrate that a specified impurity is entirely absent.

When setting out to purify a laboratory chemical, it is desirable that the starting material is of the best grade commercially available. Particularly among organic solvents there is a range of qualities varying from *laboratory chemical* to *spectroscopic* and *chromatographic* grades. Many of these are suitable for use as received. With many of the more common reagents it is possible to obtain from the current literature some indications of likely impurities, their probable concentrations and methods for detecting them. However, in many cases complete analyses are not given so that significant concentrations of unspecified impurities may be present.

## THE QUESTION OF PURITY

Solvents and substances that are specified as *pure* for a particular purpose may, in fact, be quite impure for other uses. Absolute ethanol may contain traces of benzene, which makes it unsuitable for ultraviolet spectroscopy, or plasticizers which make it unsuitable for use in solvent extraction.

Irrespective of the grade of material to be purified, it is essential that some criteria exist for assessing the degree of purity of the final product. The more common of these include:

1. Examination of physical properties such as:
   (a) Melting point, freezing point, boiling point, and the freezing curve (i.e. the variation, with time, in the freezing point of a substance that is being slowly and continuously frozen).
   (b) Density.
   (c) Refractive index at a specified temperature and wavelength. The sodium D line at 589.26 nm (weighted mean of $D_1$ and $D_2$ lines) is the usual standard of wavelength but results from other wavelengths can often be interpolated from a plot of refractive index versus $1/(\text{wavelength})^2$.

(d) Specific conductivity. (This can be used to detect, for example, water, salts, inorganic and organic acids and bases, in non-electrolytes).
(e) Optical rotation, optical rotatory dispersion and circular dichroism.

2. Empirical analysis, for C, H, N, ash, etc.

3. Chemical tests for particular types of impurities, e.g. for peroxides in aliphatic ethers (with acidified KI), or for water in solvents (quantitatively by the Karl Fischer method, see Fieser and Fieser, Reagents for Organic Synthesis J. Wiley & Sons, NY, Vol 1 pp. 353, **528**, *1967*, Library of Congress Catalog Card No 66-27894).

4. Physical tests for particular types of impurities:
   (a) Emission and atomic absorption spectroscopy for detecting organic impurities and determining metal ions.
   (b) Chromatography, including paper, thin layer, liquid (high, medium and normal pressure) and vapour phase.
   (c) Electron spin resonance for detecting free radicals.
   (d) X-ray spectroscopy.
   (e) Mass spectroscopy.
   (f) Fluorimetry.

5. Examination of spectroscopic properties
   (a) Nuclear Magnetic Resonance ($^{1}H$, $^{13}C$, $^{31}P$, $^{19}F$ NMR etc)
   (b) Infrared spectroscopy (IR)
   (c) Ultraviolet spectroscopy (UV)
   (d) Mass spectroscopy [electron ionisation (EI), electron ionisation (CI), electrospray ionisation (ESI), fast atom bombardment (FAB), matrix-associated laser desorption ionisation (MALDI), etc]

6. Electrochemical methods (see Chapter 6 for macromolecules).

7. Nuclear methods which include a variety of radioactive elements as in organic reagents, complexes or salts.

A substance is usually taken to be of an acceptable purity when the measured property is unchanged by further treatment (especially if it agrees with a recorded value). In general, at least two different methods, such as recrystallisation and distillation, should be used in order to ensure maximum purity. Crystallisation may be repeated (from the same solvent or better from different solvents) until the substance has a constant melting point or absorption spectrum, and until it distils repeatedly within a narrow, specified temperature range.

With liquids, the refractive index at a specified temperature and wavelength is a sensitive test of purity. Note however that this is sensitive to dissolved gases such as $O_2$, $N_2$ or $CO_2$. Under favourable conditions, freezing curve studies are sensitive to impurity levels of as little as 0.001 moles per cent. Analogous fusion curves or heat capacity measurements can be up to ten times as sensitive as this. With these exceptions, most of the above methods are rather insensitive, especially if the impurities and the substances in which they occur are chemically similar. In some cases, even an impurity comprising many parts per million of a sample may escape detection.

The common methods of purification, discussed below, comprise distillation (including fractional distillation, distillation under reduced pressure, sublimation and steam distillation), crystallisation, extraction, chromatographic and other methods. In some cases, volatile and other impurities can be removed simply by heating. Impurities can also sometimes be eliminated by the formation of derivatives from which the purified material is regenerated (see Chapter 2).

## SOURCES OF IMPURITIES

Some of the more obvious sources of contamination of solvents arise from storage in metal drums and plastic containers, and from contact with grease and screw caps. Many solvents contain water. Others have traces of acidic materials such as hydrochloric acid in chloroform. In both cases this leads to corrosion of the drum and contamination of the solvent by traces of metal ions, especially $Fe^{3+}$. Grease, for example on stopcocks of separating funnels and other apparatus, e.g. greased ground joints, is also likely to contaminate solvents during extractions and chemical manipulation.

A much more general source of contamination that has not received the consideration it merits comes from the use of plastics for tubing and containers. Plasticisers can readily be extracted by organic solvents from PVC and other plastics, so that most solvents, irrespective of their grade (including spectrograde and ultrapure) have been reported to contain 0.1 to 5ppm of plasticiser [de Zeeuw, Jonkman and van Mansvelt *Anal Biochem* **67** 339 *1975*]. Where large quantities of solvent are used for extraction (particularly of small amounts of compounds), followed by evaporation, this can introduce significant amounts of impurity, even exceeding the weight of the genuine extract and giving rise to spurious peaks in gas chromatography (for example of fatty acid methyl esters [Pascaud, *Anal Biochem* **18** 570 *1967*]. Likely contaminants are di(2-ethylhexyl)phthalate and dibutyl phthalate, but upwards of 20 different phthalate esters are listed as plasticisers as well as adipates, azelates, phosphates, epoxides, polyesters and various heterocyclic compounds. These plasticisers would enter the solvent during passage through plastic tubing or from storage in containers or from plastic coatings used in cap liners for bottles. Such contamination could arise at any point in the manufacture or distribution of a solvent. The problem with cap liners is avoidable by using corks wrapped in aluminium foil, although even in this case care should be taken because aluminium foil can dissolve in some liquids e.g. benzylamine and propionic acid.

Solutions in contact with polyvinyl chloride can become contaminated with trace amounts of lead, titanium, tin, zinc, iron, magnesium or cadmium from additives used in the manufacture and moulding of PVC.

*N*-Phenyl-2-naphthylamine is a contaminant of solvents and biological materials that have been in contact with black rubber or neoprene (in which it is used as an antioxidant). Although it was only an artefact of the separation procedure it has been isolated as an apparent component of vitamin K preparations, extracts of plant lipids, algae, livers, butter, eye tissue and kidney tissue [Brown *Chem Br* **3** 524 *1967*].

Most of the above impurities can be removed by prior distillation of the solvent, but care should be taken to avoid plastic or black rubber as much as possible.

## PRACTICES TO AVOID IMPURITIES

### Cleaning practices

Laboratory glassware and Teflon equipment can be cleaned satisfactorily for most purposes by careful immersion into a solution of sodium dichromate in concentrated sulfuric acid, followed by draining, and rinsing copiously with distilled water. This is an exothermic reaction and should be carried out **very** cautiously in an efficient fume cupboard. [To prepare the chromic acid bath, dissolve 5 g of sodium dichromate (CARE: cancer suspect agent) in 5 mL of water.The dichromate solution is then cooled and stirred while 100 mL of concentrated sulfuric acid is added slowly. Store in a glass bottle.] Where traces of chromium (adsorbed on the glass) must be avoided, a 1:1 mixture of concentrated sulfuric and nitric acid is a useful alternative. (*Use in a fumehood to remove vapour and with adequate face protection.*) Acid washing is also suitable for polyethylene ware but prolonged contact (some weeks) leads to severe deterioration of the plastic. Alternatively an alcoholic solution of sodium hydroxide (alkaline base bath) can be used. This strongly corrosive solution (CAUTION: Alkali causes serious burns) can be made by dissolving 120g of NaOH in 120 mL water, followed by dilution to 1 L with 95% ethanol. This solution is conveniently stored in suitable alkali resistant containers (e.g. Nalgene heavy duty rectangular tanks) with lids. Glassware can be soaked overnight in the base bath and rinsed thoroughly after soaking. For much glassware, washing with hot detergent solution, using tap water, followed by rinsing with distilled water and acetone, and heating to 200-300° overnight; is adequate. (Volumetric apparatus should not be heated: after washing it is rinsed with acetone, then hexane, and air-dried. Prior to use, equipment can be rinsed with acetone, then with petroleum ether or hexane, to remove the last traces of contaminants.) Teflon equipment should be soaked, first in acetone, then in petroleum ether or hexane for ten minutes prior to use.

For trace metal analyses, prolonged soaking of equipment in 1M nitric acid may be needed to remove adsorbed metal ions.

Soxhlet thimbles and filter papers may contain traces of lipid-like materials. For manipulations with highly pure materials, as in trace-pesticide analysis, thimbles and filter papers should be thoroughly extracted with hexane before use.

Trace impurities in silica gel for TLC can be removed by heating at 300° for 16h or by Soxhlet extraction for 3h with distilled chloroform, followed by 4h extraction with distilled hexane.

### Silylation of glassware and plasticware

Silylation of apparatus makes it repellant to water and hydrophilic materials. It minimises loss of solute by adsorption onto the walls of the container. The glassware is placed in a desiccator containing dichloromethyl silane (1mL) in a small beaker and evacuated for 5min. The vacuum is turned off and air is introduced into the desiccator which allows the silylating agent to coat the glassware uniformly. The desiccator is then evacuated, closed and set aside for 2h. The glassware is removed from the desiccator and baked at 180° for 2h before use.