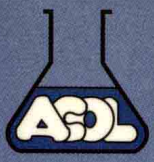
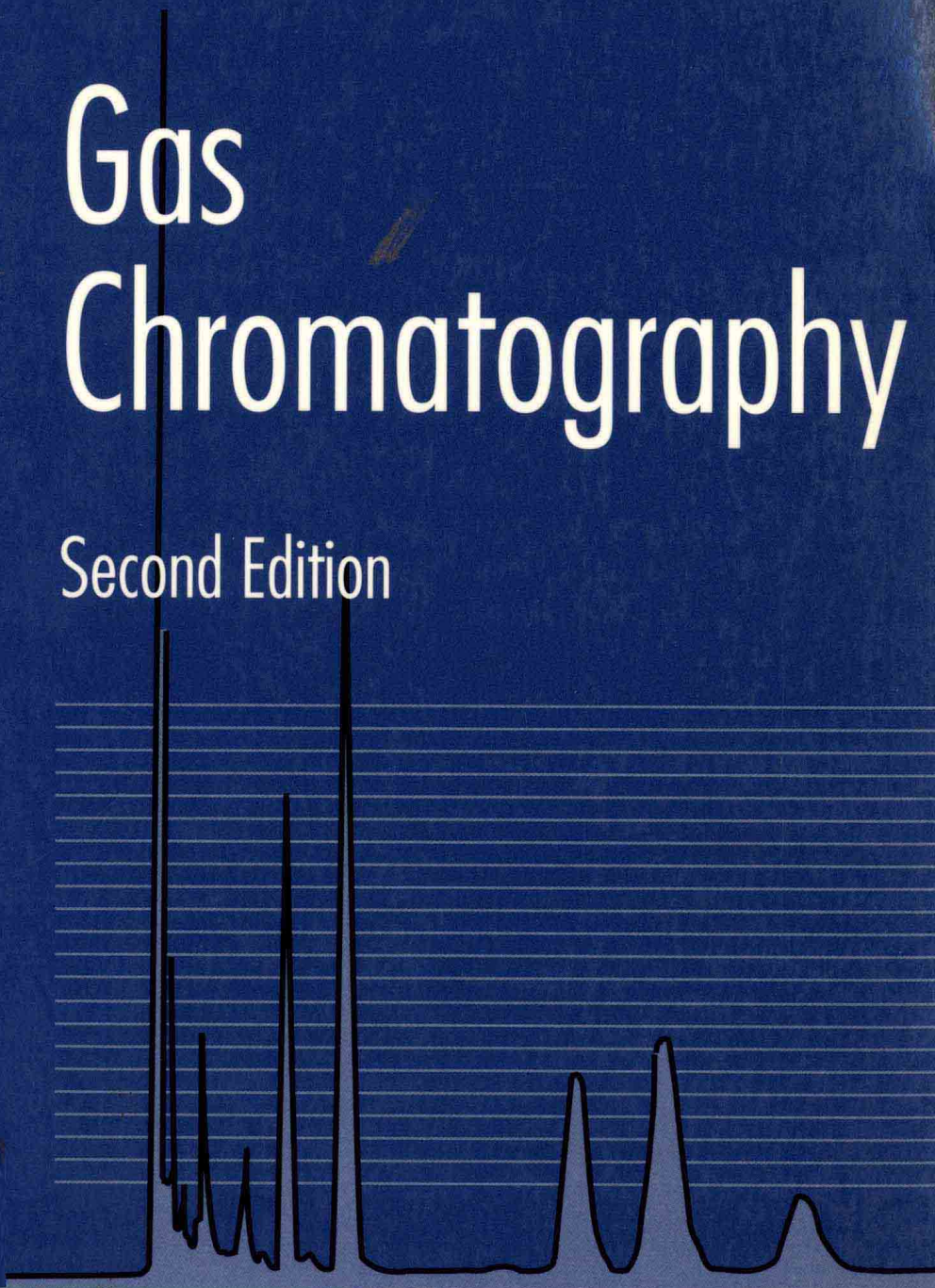


ANALYTICAL CHEMISTRY BY OPEN LEARNING

Gas Chromatography

Second Edition



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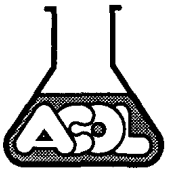

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- Find the most suitable place to study where you can work without disturbance.

- If you have a tutor supervising your study discuss with him, or her, the date by which you should have completed this text.

- Some people study perfectly well in irregular bursts, however most students find that setting aside a certain number of hours each day is the most satisfactory method. It is for you to decide which pattern of study suits you best.

- If you decide to study for several hours at once, take short breaks of five or ten minutes every half hour or so. You will find that this method maintains a higher overall level of concentration.

Before you begin a detailed reading of the book, familiarise yourself with the general layout of the material. Have a look at the course contents list at the front of the book and flip through the pages to get a general impression of the way the subject is dealt with. You will find that there is space on the pages to make comments alongside

the text as you study — your own notes for highlighting points that you feel are particularly important. Indicate in the margin the points you would like to discuss further with a tutor or fellow student. When you come to revise, these personal study notes will be very useful.

Π When you find a paragraph in the text marked with a symbol such as is shown here, this is where you get involved. At this point you are directed to do things: draw graphs, answer questions, perform calculations, etc. Do make an attempt at these activities. If necessary cover the succeeding response with a piece of paper until you are ready to read on. This is an opportunity for you to learn by participating in the subject and although the text continues by discussing your response, there is no better way to learn than by working things out for yourself.

We have introduced self-assessment questions (SAQs) at appropriate places in the text. These SAQs provide for you a way of finding out if you understand what you have just been studying. There is space on the page for your answer and for any comments you want to add after reading the author's response. You will find the author's response to each SAQ at the end of the book. Compare what you have written with the response provided and read the discussion and advice.

At intervals in the book you will find Lists of Learning Objectives. These will give you a check-list of tasks you should then be able to achieve at these various stages of the Unit..

You can revise the Unit, perhaps for a formal examination, by re-reading the Objectives, and by working through some of the SAQs. This should quickly alert you to areas of the text that need further study.

At the end of this book you will find, for reference, lists of commonly used scientific symbols and values, units of measurement and also a periodic table.

Study Guide

This Unit is intended to provide you with a working knowledge of gas chromatography. It will not turn you into a fully experienced gas chromatographer — only months and years of practice can do that, for gas chromatography is still something of an art. Many of us have learned through standing in front of a gas chromatograph and making endless mistakes. It is very frustrating, but it is probably the best way to learn to recognise the symptoms of the things which can go wrong with gas chromatography. There are many settings on a gas chromatograph which can be wrongly chosen, and many physical malfunctions that have nothing to do with the chemistry of the process but affect the outcome of an analysis dramatically. They have to be experienced, or at least described to you before you learn to recognise the symptoms and diagnose the faults. This Unit will try to provide you with the equivalent of that experience by adopting the standpoint of a practising gas chromatographer.

Even so, while emphasising the practical side of the subject, the theoretical side will not be ignored. It is only by understanding the processes occurring in your instrument that you will get the best out of it. The purpose of considering the theory, though, is quite clearly to improve your practical performance.

It will be assumed that you have an understanding of chemistry equivalent to that of a student who has passed HNC or HTC in chemistry (BTEC), and a knowledge of physics up to at least GCE (OL). You may also have some basic understanding of chromatography.

You may find that another author's views on gas chromatography will clarify, for you, some aspects of this text. A number of recent and not-so-recent texts and publications are listed in the Bibliography, some of which are included for their historical interest.

Inevitably, in a distance learning package you will not get anything like enough 'hands-on' practical experience, but at your regional centre you may have the opportunity to use gas chromatographs and computer controlled simulations which will help you along the road to becoming an experienced gas chromatographer.

Supporting Practical Work

1. GENERAL CONSIDERATIONS

Gas chromatographs are one of the workhorses of many laboratories so it is quite likely that you will be able to gain practical experience at your place of work.

In general, gas chromatographers are a friendly bunch who will be only too glad to show you the equipment they use and to suggest experiments you may try out on the instruments available. Remember of course that you will have to slot in with the priorities of the laboratory and do not neglect your own specific duties in your enthusiasm for your new interest.

Take note carefully of the instructions given to you and be prepared to discuss various aspects of this text as you progress with the chosen exercises. These discussions will help to reinforce your understanding of the text and its relationship to the outcome of your practical experiments.

It will probably be of more value to you if you obtain access to an older instrument in the first instance since you can then become thoroughly familiar with the hardware, changing columns and the controls rather than just addressing a computer which controls the system. Gas chromatographs are very robust and provided you handle syringes with care as instructed you are unlikely to do any damage. Having mastered the basics, it is then essential to gain experience using both packed and capillary columns. If, in addition, you can spend some time running high resolution gas chromatography (HRGC)–mass spectrometry (MS) or HRGC–infrared (IR) spectroscopy then so much the better.

Finally, always endeavour to produce chromatograms which look

good. Gas chromatography is an art and usually if the chromatogram looks good it is good in terms of separation and sensitivity.

2. AIMS

(*a*) To provide a basic experience of using gas chromatographic equipment.

(*b*) To illustrate the effect on separation in gas chromatography of the various operating parameters using both packed and capillary columns.

(*c*) To illustrate the feasibility of qualitative and quantitative analysis.

(*d*) To illustrate how gas chromatography can be combined with spectroscopic methods to provide identification of eluted components.

3. SUGGESTED EXPERIMENTS

(*a*) The examination of the effect of stationary phase, column length, temperature, carrier gas flow-rate, sample size, etc., upon the separation of benzene, cyclohexane and ethanol.

(*b*) Comparison of the resolving power of packed and capillary columns on a mixture of long-chain fatty acid esters.

(*c*) The determination of the concentration of ethanol in a dilute aqueous sample in the concentration range relevant to drink–drive legislation (approximately 100 mg/100 ml) using propan-1-ol as internal standard.

(*d*) Optimise conditions for the analysis of spearmint oil by capillary column gas chromatography.

(*e*) Compare the responses obtained for a sample mixture containing toluene, *p*-chlorophenol, bromobenzene, dibromobenzene and n-butanol using flame ionisation, electron capture and mass spectrometry detection.

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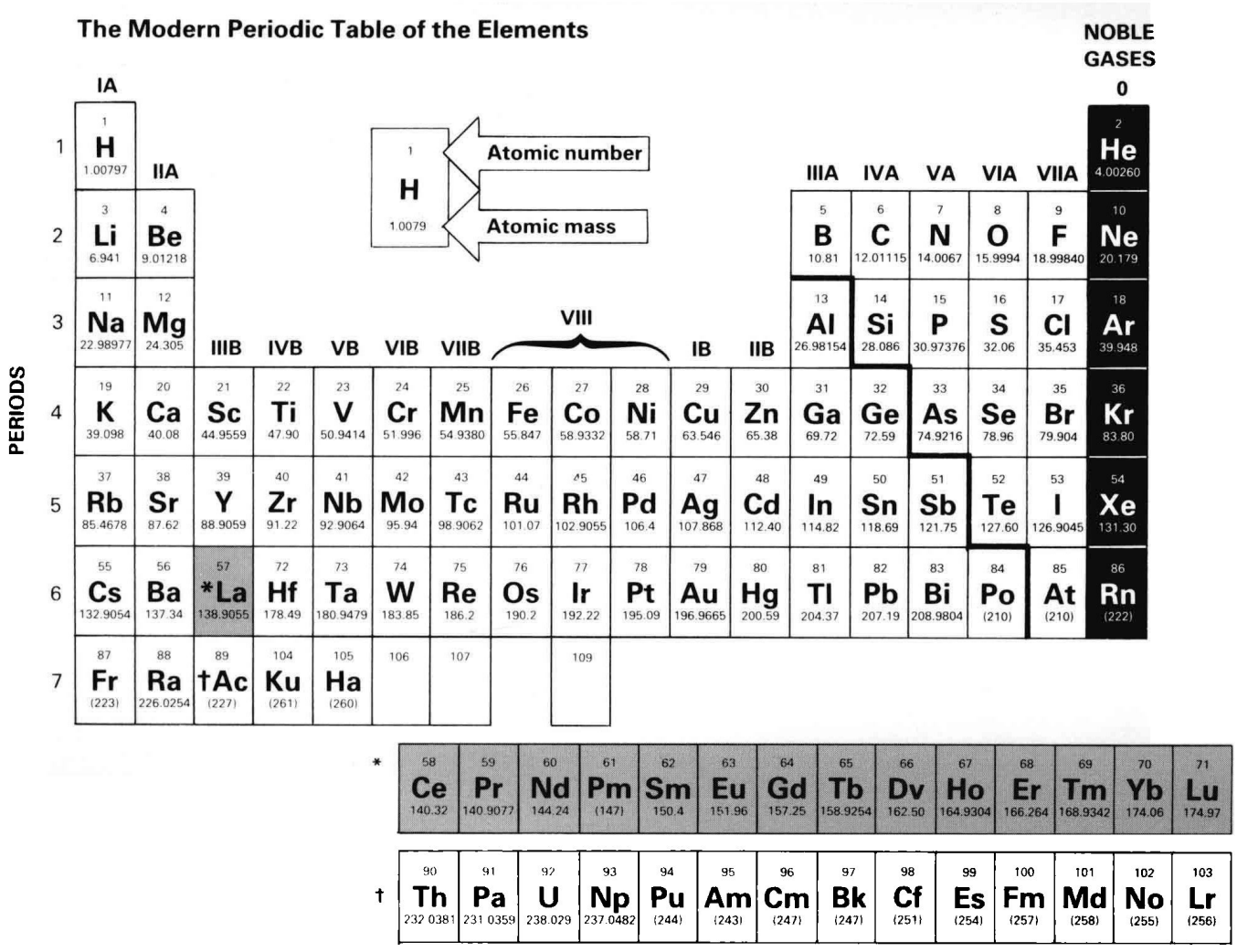
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