Thermal Methods

Analytical Chemistry by Open Learning

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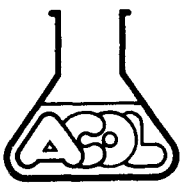

Analytical Chemistry by Open Learning

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Analytical Chemistry

This series of texts is a result of an initiative by the Committee of Heads of Polytechnic Chemistry Departments in the United Kingdom. A project team based at Thames Polytechnic using funds available from the Manpower Services Commission 'Open Tech' Project have organised and managed the development of the material suitable for use by 'Distance Learners'. The contents of the various units have been identified, planned and written almost exclusively by groups of polytechnic staff, who are both expert in the subject area and are currently teaching in analytical chemistry.

The texts are for those interested in the basics of analytical chemistry and instrumental techniques who wish to study in a more flexible way than traditional institute attendance or to augment such attendance. A series of these units may be used by those undertaking courses leading to BTEC (levels IV and V), Royal Society of Chemistry (Certificates of Applied Chemistry) or other qualifications. The level is thus that of Senior Technician.

It is emphasised however that whilst the theoretical aspects of analytical chemistry can be studied in this way there is no substitute for the laboratory to learn the associated practical skills. In the U.K. there are nominated Polytechnics, Colleges and other Institutions who offer tutorial and practical support to achieve the practical objectives identified within each text. It is expected that many institutions worldwide will also provide such support.

The project will continue at Thames Polytechnic to support these 'Open Learning Texts', to continually refresh and update the material and to extend its coverage.

Further information about nominated support centres, the material or open learning techniques may be obtained from the project office at Thames Polytechnic, ACOL, Wellington St., Woolwich, London, SE18 6PF.

How to Use an Open Learning Text

Open learning texts are designed as a convenient and flexible way of studying for people who, for a variety of reasons cannot use conventional education courses. You will learn from this text the principles of one subject in Analytical Chemistry, but only by putting this knowledge into practice, under professional supervision, will you gain a full understanding of the analytical techniques described.

To achieve the full benefit from an open learning text you need to plan your place and time of study.

- 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 text, 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.

∏ 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 (SAQ) 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 text. Compare what you have written with the response provided and read the discussion and advice.

At intervals in the text you will find a Summary and List of Objectives. The Summary will emphasise the important points covered by the material you have just read and the Objectives will give you a checklist of tasks you should then be able to achieve.

You can revise the Unit, perhaps for a formal examination, by re-reading the Summary and 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 the 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 give you a reasonable grounding in the major techniques of thermal analysis. It cannot, of course, turn you into an expert practitioner overnight—only long experience can do that. However, it can consolidate any interest you might have in the field already—or perhaps lead you into pastures new.

You will soon realise (by the end of Part 1 in fact) that thermal methods are extremely versatile as far as their range of application is concerned. Almost every type of material can be usefully investigated by this fascinating area of analytical science. Of course, the corresponding equipment is becoming more sophisticated all the time, as a glance at the manufacturers' brochures will show. Therefore we shall aim in this Unit to understand the basic principles of the methods, and the manner in which they are affected by experimental conditions and so on. Only in this way will you be able to apply the techniques we discuss, to new situations with a maximum chance of success.

Now then, what academic background do you need to cope with this Unit? Ideally you should have studied Chemistry to A level, in particular an appreciation of the effect of heat on materials, and an understanding of the stoichiometric basis of chemical decomposition will be necessary. (The latter wil enable you to tackle the numerical assignments). The text of this Unit contains a number of self-assessment questions of various types, as well as in-text questions for you to tackle. But *do* resist the temptation to look at the response before you make your own attempt at the question!

Although this Unit is designed to be self-contained, you may wish to consult the more specialised texts on thermal analysis for further information on various topics. Details of these are given in the Bibliography. We shall occasionally refer to other methods of analysis like gas liquid chromatography, infrared spectroscopy or mass spectrometry. If you are unfamiliar with these—don't worry. References are given for further information if you need it.

Last, but certainly not least, *practical work.* A recommended list of associated practical work in thermal analysis is given, but obviously the more 'hands-on' experience you can get the better. To extend your opportunity to deal with real thermal data, and to bridge the gap between theory and practical, the Unit concludes with a 'Data Pack' in Part 6.

Well, so much for the introductions. Now to the Unit itself. I hope you enjoy working through it.

Supporting Practical Work

The following practical course would support Parts 2 and 3 (dealing with TG) and Parts 4 and 5 (dealing with DSC/DTA), and Part 6 (exercises in the use of thermoanalytical techniques based on HAC cement). Part 1 of the Unit is introductory and illustrative, and has no direct practical back-up. Overall, about 10-15 hours of practical work would be necessary to cover the practical objectives set out below.

By the end of the practical course the student should be able to:

1. Set up, calibrate and operate a thermobalance (for TG) calorimeter (for DSC) or analyser (for DTA).

2. Select optimum operating conditions for obtaining thermal data by TG/DSC/DTA.

3. Prepare a variety of samples, in suitable forms, for thermal analysis.

4. Use TG/DTG for the quantitative analysis of compounds, or mixtures, eg carbonates/oxalates.

5. Use DSC/DTA for the qualitative comparison of materials, eg polymeric materials.

6. Use DSC for making quantitative thermodynamic measurements, eg enthalpies of fusion.

7. Use thermoanalytical techniques to investigate the degree of conversion of HAC cement.

Bibliography

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3. W W Wendlandt, *Thermal Methods of Analysis*, 2nd Edn., Wiley-Interscience, 1974.

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8. *Journal of Thermal Analysis* (published bimonthly) Wiley Heyden.

9. *Thermal Analysis Abstracts* (published bimonthly) Wiley Heyden.

10. *Analytical Chemistry*—ongoing reviews of thermal applications, biannually, eg:

    W W Wendlandt, *Anal. Chem.* 1982, 54, 97R-105R
    ibid 1984, 56, 250R-261R.

References 1–3 are books immediately useful for this Unit.

References 4–5 are *general* textbooks on analytical chemistry, containing useful chapters on thermal analysis.

References 6–7 are *advanced texts* containing specialised information.

References 8–10 are to specialist *journals* dealing with thermal analysis.

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