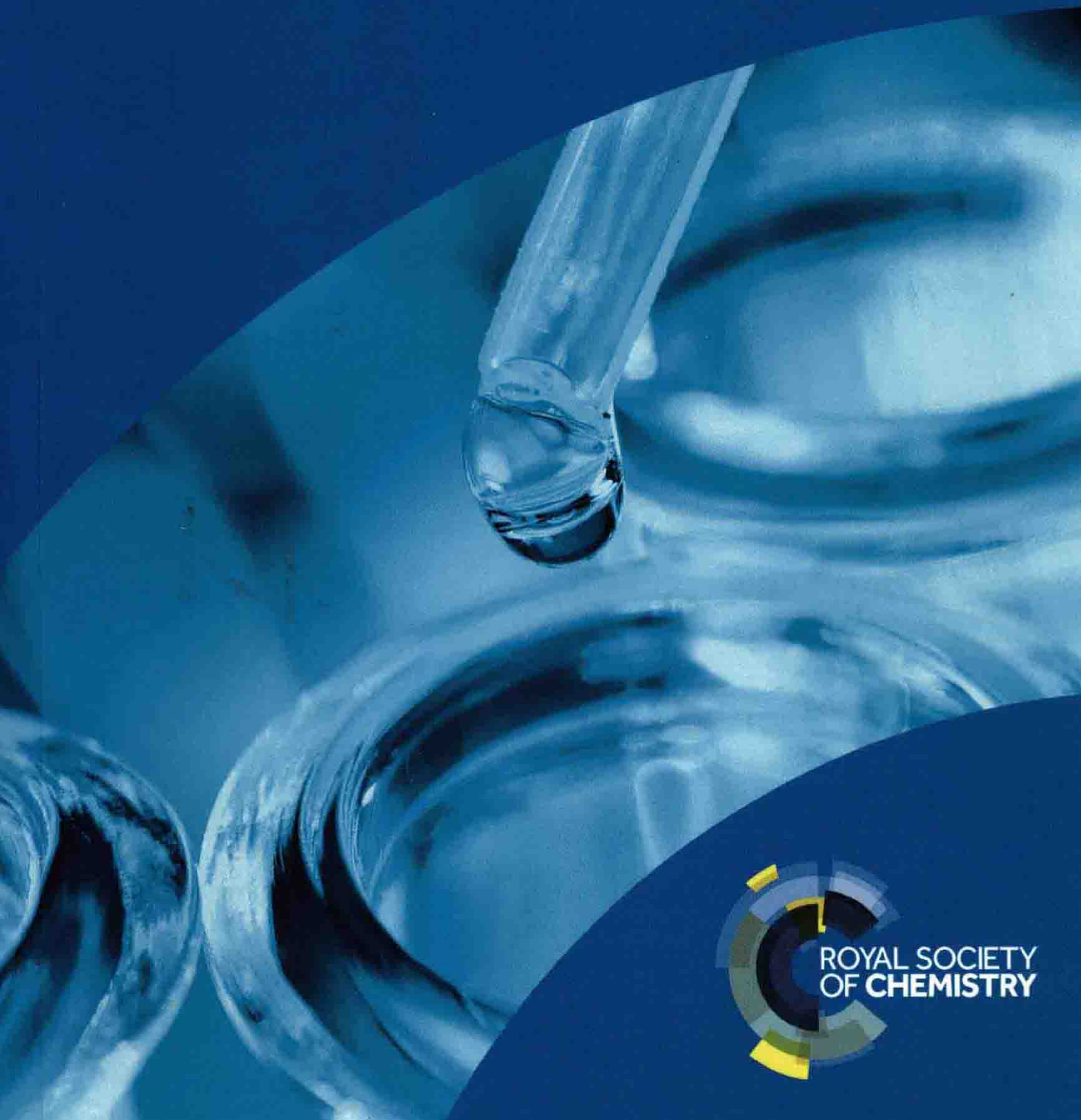
Practical Volumetric Analysis

Peter A C McPherson



ROYAL SOCIETY OF CHEMISTRY

Practical Volumetric Analysis

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Practical Volumetric Analysis

Preface

Volumetric analysis, or titrimetry, has been an essential tool of the chemist since the mid-1600s. The success and longevity of volumetric analysis is no doubt primarily due to its relative simplicity and also the fact that manual titrations require relatively inexpensive equipment. Despite this simplicity, the skills required for the technique are far from trivial—it demands a sound theoretical knowledge of the underlying chemistry, as well as a high degree of manual dexterity. For these reasons, volumetric analysis is still included in virtually all course specifications, ranging from GCSE to undergraduate-level chemistry. Of course volumetric analysis is not restricted to educational settings. Titrations are still performed in industry as a means of monitoring the purity of feedstocks and products, and although many of these are now performed by automated titration systems, some are still performed manually.

Despite the inclusion of volumetric analysis in virtually all school, college and university courses in chemistry, there have been few dedicated texts published in this area. This is perhaps due to its inclusion in many of the fine analytical chemistry texts on the market, as well as the general chemistry textbooks aimed at school/college-level students. However, the ever increasing demands on teachers' time means that we can ill afford hours spent searching online or in libraries for details of a particular titration. Furthermore, we find that the standard repertoire of standardising solutions and determining how much iron is in ferric ammonium sulphate no longer satisfies our students. They want to see how volumetric analysis can be applied

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to their world—a world of food, cosmetics, medicines and fashion. Whilst some of these are more amenable to volumetric analysis than others, if we are to inspire a future generation of analytical chemists, we must become innovative, and mix the traditional with the contemporary in both our teaching and in our practical labs. This is one of the aims of this text.

Each chapter begins with a review of essential underlying theory and is followed by a series of well-tested practicals. The theory may be in much greater depth than is required for a particular course of study, but it is expected that the student/teacher is sufficiently familiar with the requirements of their course to be able to select what is needed. The practicals can be taken and used as stand-alone laboratory work, or as part of a series. These structured practicals are followed by a selection of open-ended investigations which may be particularly useful for those students pursing vocational science courses, such as the Extended Diplomas in Applied Science, as well as Higher National Certificate/Diploma. A 'compendium' of routine acid–base titrations is also provided at the end of Chapter 5 which provides additional practical work for students following A-level/IB/ Leaving Certificate or Higher Grade Chemistry.

There are a number of people who have helped bring this book into being. My family and friends, who reminded me that I ought to be at my computer and not in front of the TV; Peter Boyle for helping with artwork and fetching cups of Earl Grey to my office; my colleagues, Bernard McKeaveney and Caroline Kelly, who provided a teacher's perspective; the legions of students who have passed through my lab over the years, only to have burettes forced upon them at a moment's notice to test out another titration; and finally, the staff at RSC Publishing who kept me on the straight and narrow and provided support whenever it was needed. Of course, all errors are my own, and I could welcome feedback from readers, especially those who have used the practical work and developed useful modifications.

PACMcP
Belfast

Units, Symbols & Terminology Used

Chemical names and formulae: as far as is practically possible, the IUPAC conventions for the naming of chemical compounds have been used. Inorganic compounds are usually named according to the Stock notation—the oxidation number given in Roman numerals after the name of the element, *e.g.* ferrous becomes iron(II).

Quantities: throughout the book, the concentration of solutions is expressed in moles per cubic decimetre unless otherwise stated. At the risk of becoming *persona non grata* with some readers, I have opted to abbreviate the units 'mol dm^{-3}' to M, firstly because this is what is used in the 'real world' where our students will one day work, and secondly because it is less cumbersome. Similarly, I have opted to use millilitres (mL) to express volumes of liquids and solutions, rather than the SI unit cm^3. Those students following courses with an examination component should be encouraged to use the units stated in their course specification. When a quantity is described as 'an aliquot' or 'exactly', a pipette or burette should be used to deliver that quantity; otherwise a measuring (graduated) cylinder is sufficient.

LABORATORY HEALTH AND SAFETY

The practical activities given in this book have been developed to use the lowest concentration of reagents possible. However, an essential

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part of training in chemistry is to learn how to safely handle chemicals. For schools and colleges in the UK, CLEAPSS (www.cleapss.org.uk) provides invaluable information on chemical safety in the form of their Hazcards. ***It is the responsibility of all those who use the practical information in this book to conduct their own risk assessment for each activity.***

General Laboratory Safety

- Always perform your own risk assessment for a practical activity—don't rely on other people's advice, especially if you have a medical condition such as asthma. Make sure you are familiar with hazard warning symbols.
- Always wear personal protective equipment (PPE) which normally includes a buttoned-up laboratory coat and safety goggles. Ordinary glasses will not provide sufficient protection for the eyes and contact lenses should not be worn during practical sessions. Open-toed sandals or very high heels should not be worn in the laboratory.
- Always label all glassware that contains solutions with the name of the reagent and its concentration. Do not remove more of the solution from the storage bottles than is required.
- When heating solutions with an open flame (Bunsen burner) make sure all long hair is tied back and that no flammable liquids are in the laboratory. Adjust the Bunsen to give a luminous flame when not in use.
- Never force pipettes into a pipette filler. The pipette should be held a few centimetres from the upper-most end and gently inserted into the filler.

Hazard Warning Symbols

Under the CLP regulations (Classification, Labelling and Packaging of Substances and Mixtures) introduced in January 2009, a new set of hazard warning symbols were introduced to replace the existing symbols. It is likely that both will be in use for some time as many older stocks of chemicals may still be used in teaching laboratories.

New CLP Symbol	Description/Meaning	Old CHIP Symbol
	Irritant or harmful (but less than acute toxicity category 4)	
	Acute toxicity (categories 1–3)	
	Oxidising (solids, liquids and gases)	
	Corrosive	
	Flammable (solids, liquids and gases) Self-reactive substances Organic peroxides	
	Long-term health effects (*e.g.* germ cell mutagenicity)	–
	Explosive	
	Hazardous to aquatic life	
	Gas under pressure	–

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