



Experiments in Physical Chemistry

*Second
revised and enlarged
Edition*

J.M. Wilson,
University of Technology,
Loughborough

R.J. Newcombe,
College of Technology,
Grimsby

A.R. Denaro,
College of Technology,
Liverpool

R.M.W. Rickett,
Sir John Cass College,
London

Pergamon Press

**EXPERIMENTS
IN
PHYSICAL CHEMISTRY**

This page intentionally left blank

EXPERIMENTS IN PHYSICAL CHEMISTRY

Second Revised and Enlarged Edition

J. M. WILSON,
M.Sc., F.R.I.C.

Senior Lecturer in Physical Chemistry, University of Technology, Loughborough

R. J. NEWCOMBE,
M.Sc., F.R.I.C., A.Inst.P.

Head of Department of Science and Mathematics, College of Technology, Grimsby

A. R. DENARO,
M.Sc., Ph.D., F.R.I.C.

Principal Lecturer in Physical Chemistry, College of Technology, Liverpool

R. M. W. RICKETT,
Ph.D., F.R.I.C.

Vice-Principal, Sir John Cass College, London



THE QUEEN'S AWARD
TO INDUSTRY 1966

PERGAMON PRESS

OXFORD · LONDON · EDINBURGH · NEW YORK
TORONTO · SYDNEY · PARIS · BRAUNSCHWEIG

Pergamon Press Ltd., Headington Hill Hall, Oxford
4 & 5 Fitzroy Square, London W.1
Pergamon Press (Scotland) Ltd., 2 & 3 Teviot Place, Edinburgh 1
Pergamon Press Inc., 44-01 21st Street, Long Island City, New York 11101
Pergamon of Canada Ltd., 207 Queen's Quay West, Toronto 1
Pergamon Press (Aust.) Pty. Ltd., 19a Boundary Street,
Rushcutters Bay, N.S.W. 2011, Australia
Pergamon Press S.A.R.L., 24 rue des Écoles, Paris 5°
Vieweg & Sohn GmbH, Burgplatz 1, Braunschweig

Copyright © 1962 and 1968
Pergamon Press Ltd.

First edition 1962
Second Revised and enlarged edition 1968

Library of Congress Catalog Card No. 68-18536

PRINTED IN GREAT BRITAIN BY J. W. ARROWSMITH LTD., BRISTOL

08 012541 7

Contents

PREFACE	ix
LIST OF SYMBOLS	xi

PART I

Experiments

Physical Properties and Molecular Structure

1. Molecular Weight using van der Waals' Equation	1
2. The Density of a Liquid as a Function of Temperature	3
3. Molar Refraction	6
4. Viscosity as a Function of Temperature	8

Thermodynamics

5. The Ratio of the Heat Capacities of a Gas (Clément and Desormes Method)	10
6. The Ratio of the Heat Capacities of a Gas (Lummer and Pringsheim Method)	13
7. Molecular Weight by Ebullioscopy (Landsberger's Method)	16
8. Molecular Weight by Ebullioscopy (Cottrell's Method)	19
9. Molecular Weight by Cryoscopy	21
10. Molecular Weight by Rast's Method	24
11. Heat of Neutralization by Calorimetry	25
12. Heat of Transition by Calorimetry	28
13. Heat of Vaporization by Calorimetry	29
14. The Vapour Pressure of a Liquid as a Function of Temperature	31
15. Heat of Solution from Solubility	33
16. Heat of Combustion by Bomb Calorimetry	35
17. An Equilibrium Constant by the Distribution Method	39

Phase Equilibria

18. Distillation of an Azeotropic Mixture with a Minimum Boiling Point	41
19. Molecular Weight of a Liquid by Steam Distillation	44
20. Phase Diagram for a Binary System	46
21. The Variation of Miscibility with Temperature	47
22. A Study of the Ternary System: Benzene-Acetic Acid-Water	49
23. The Thermal Analysis of a Hydrate by the Differential Couple Method	52
24. A Transition Temperature by a Solubility Method	55

Light and Spectra

25. Verification of the Lambert-Beer Law	57
26. Composition of a Complex Ion in Solution	59

Chemical Kinetics

27. A Velocity Constant by a Titration Method	61
28. Variation of a Velocity Constant with Catalyst Concentration by a Polarimetric Method	64

29.	A Velocity Constant by a Gas Evolution Method	67
30.	A Velocity Constant by a Conductimetric Method	70
31.	The Effect of Change of Temperature on the Rate of a Reaction	72
32.	Determination of the Order of a Reaction	75

Surface Chemistry and Colloids

33.	Surface Tension using a Traube Stalagmometer	77
34.	Interfacial Tension (Micrometer Syringe Method)	80
35.	Variation of the Surface Tension of a Liquid with Temperature	82
36.	Adsorption Isotherm	85

Conductance and Transference

37.	Variation of Conductance with Concentration (Strong and Weak Electrolytes)	88
38.	Conductimetric Titration of an Acid Mixture	91
39.	Solubility by a Conductimetric Method	93
40.	Transport Numbers (Hittorf's Method)	95

Ionic Equilibria

41.	Dissociation Constant of a Weak Acid (Approximate e.m.f. Method)	98
42.	Dissociation Constant of a Weak Acid (Conductimetric Method)	102
43.	Hydrolysis Constant by a Conductimetric Method	104

Electrode Potential and Electrode Processes

44.	The Standard Electrode Potentials of Zinc and Copper	106
45.	Concentration Cells	110
46.	Solubility Product from e.m.f. Measurements	112
47.	The Quinhydrone Reference Electrode	114
48.	pH Titration Curve	116
49.	Study of Potentiometric and Indicator End Points	119
50.	Potentiometric Titration—Verification of the Nernst Equation	121
51.	Thermodynamics of Cells	123

PART II

Experiments

Physical Properties and Molecular Structure

52.	The Radius of a Molecule from Viscosity Measurements	129
53.	Dipole Moment of a Polar Molecule	131
54.	The Additivity of Group Moments in Aromatic Compounds	135

Thermodynamics

55.	The Joule-Thomson Coefficient	137
56.	The Partition Coefficient in Gas Chromatography	142
57.	The Raoult Law Factor in Gas Chromatography	146
58.	Activity of a Non-electrolyte by Cryoscopy	149
59.	Activity of an Electrolyte by Cryoscopy	152
60.	Activity Coefficient by an e.m.f. Method	160
61.	The Partial Molal Volumes of a Binary Solution	164

Phase Equilibria

62.	A Ternary Phase Diagram for a System of Two Solids and a Liquid	167
-----	---	-----

Light and Spectra

63. Emission Spectra Study of Atomic Hydrogen	171
64. The Ultra-violet Absorption Spectra of Geometrical Isomers	174
65. Force Constants from Vibrational Frequencies	177
66. Infra-red Spectra of Carbonyl Compounds	179

Chemical Kinetics

67. Variation of Velocity Constant with Catalyst Concentration by a Dilatometer Method	181
68. A Velocity Constant by an Amperometric Method	184
69. A Velocity Constant by a Potentiometric Method	187
70. A Velocity Constant by a Polarimetric Method	189
71. A Velocity Constant for an Anionotropic Rearrangement (Spectrophotometric Method)	193

Surface Chemistry and Colloids

72. Gas Adsorption (McBain-Bakr Balance)	195
73. Surface Tension-Concentration Relationship for Solutions (Gibbs Equation)	198
74. Molecular Weight of a Polymer from Viscosity Measurements	200
75. Electrophoresis	202

Conductance and Transference

76. Variation of Conductance with Concentration	205
77. Transport Numbers (Moving Boundary Method)	207
78. Transport Numbers (e.m.f. Method)	210

Ionic Equilibria

79. The Absorption Curve of an Indicator as a Function of pH	213
80. Dissociation Constant of an Acid (Accurate e.m.f. Method)	216
81. Dissociation Constant of an Acid (Spectrophotometric Method)	221
82. Dissociation Constant of a Weak Acid (Accurate Conductimetric Method)	224

Electrode Potential and Electrode Processes

83. Dead-stop End Point Titration Technique	227
84. Potentiometric Titration-Solubility of Silver Halides	229
85. The Polarographic Method of Analysis	231
86. The Polarographic Method of Analysis—Wave Separation	234
87. Polarographic Study of Acetaldehyde	236

Radiochemistry

Safety Precautions	237
88. Separation of Radioelements by Ion Exchange	239
89. Distribution Constant by a Radioactive Tracer Technique	242

PART III

*Investigations**Physical Properties and Molecular Structure*

90. A Chromatographic Study of the Effects of Substitution on Acetophenone	249
91. A Chromatographic Study of the Relationship between Heats of Solution and Molecular Structure	253

92.	Steric Effects and Resonance	255
93.	Dipole Moments of Polar Compounds (Guggenheim's Method)	257
94.	Diffusion of Solvent Molecules through High Polymers	260
95.	Interpretation of X-ray Powder Photographs	264
96.	Delocalization Energies of Π Molecular Orbital Systems	269
<i>Thermodynamics</i>		
97.	Thermodynamic Equilibrium Constant of a Molecular Charge Transfer Complex	276
98.	Thermodynamic Functions for Acid-Base Equilibria	280
<i>Phase Equilibria</i>		
99.	A Comparison of the Efficiency of Laboratory Fractionating Columns	282
<i>Light and Spectra</i>		
100.	The Angle of Twist around an Essential C—C Single Bond for a Series of Substituted Carbonyl Compounds	286
101.	Relative Strengths of Hydrogen Bonds by a Spectrophotometric Method	291
102.	The Evaluation of the Bond Angle, Force Constants and Heat Capacity of Sulphur Dioxide from its Vibrational Spectrum	294
103.	The Internuclear Distance of Hydrogen Chloride from its Vibration-Rotation Spectrum	299
104.	The Dissociation Energy of Iodine from its Absorption Spectrum	303
105.	The Interpretation of Nuclear Magnetic Resonance Spectra	307
<i>Chemical Kinetics</i>		
106.	Explosion Limits of the Hydrogen-Oxygen Reaction	325
107.	Mechanism of the Reaction between Hydrogen Iodide and Hydrogen Peroxide	329
108.	The Brønsted Primary Salt Effect	332
109.	The Variation of Rate Constant with Catalyst Concentration by a Polarimetric Method	335
<i>Surface Chemistry and Colloids</i>		
110.	The Measurement of Surface Area by the B.E.T. Method	337
111.	Critical Micelle Concentration	343
<i>Ionic Equilibria</i>		
112.	The Protonation of Aldehydes and Ketones in Sulphuric Acid Media	345
<i>Electrode Potential and Electrode Processes</i>		
113.	The Anodic Behaviour of Metals	349
114.	The Polarograph: A Study of the Variables when Interpreting Polarograms	351
115.	Differential Potentiometric Titrations	353
APPENDIX I Statistical Treatment of Experimental Data		356
APPENDIX II List of Textbooks		377
APPENDIX III List of Instruments and Manufacturers		379
LOGARITHMS OF NUMBERS		384
ANTILOGARITHMS		386
INDEX		389

Preface

Experiment is the interpreter of nature. Experiments never deceive. It is our judgement which sometimes deceives itself because it expects results which experiment refuses.

LEONARDO DA VINCI

THIS book is designed for students reading chemistry for the degree of B.Sc. Hons. of a British University or of the Council for National Academic Awards and also for the Graduate membership examination of the Royal Institute of Chemistry. It would also be suitable for students in the Junior and Senior Years of an American University who are majoring in Chemistry.

In each experiment there is a discussion of the theory associated with the objective involved. This theoretical discussion does not claim to be exhaustive but we hope that it is adequate to illustrate the point of the experiment and that it will provide a link between the theory and practice of physical chemistry. In every case the student should consult other textbooks, especially the theoretical books, in order to supplement the information given.

A course of practical physical chemistry which consists of about a hundred experiments cannot claim to be entirely original. Some of the standard experiments which are to be found in other books will also be found here. In this respect we are indebted to the authors of other textbooks and by way of acknowledgement those books to which we have had the greatest recourse are listed in Appendix II.

The book is divided into three parts. It is difficult to make rigorous divisions but we have been guided by the following principles. Part I consists of those experiments which, in general, have a simple theoretical background. Part II consists of experiments which are associated with more advanced theory or more recently developed techniques, or which require a greater degree of experimental skill. Part III contains experiments which are in the nature of investigations. These investigations may be regarded as minor research projects which are suitable for final-year students. The instructions given in these cases are intended to serve only as a framework and the investigations may be elaborated as time permits.

In those experiments which require some degree of instrumentation it will be found that particular instruments have been listed. We do not suggest that these instruments are the only ones suitable for a

particular experiment but we mention them because our students have found them to be satisfactory for the task assigned.

In short, this book has been written to facilitate experimental work in the physical chemistry laboratory at every stage of a student's career. It is hoped that by following this course the student will gain confidence in his ability to perform a physical chemistry experiment and to appreciate the value of the experimental approach.

We should like to express our thanks to Professor K. S. W. Sing (formerly Head of the Department of Chemistry, Liverpool), and to Professor R. F. Phillips (Head of the Department of Chemistry, Loughborough University of Technology) in whose laboratories the greater part of the experimental work was done. We are indebted to a number of our colleagues mentioned in Part III and the Appendixes for the contributions given with their names. We are also indebted to other colleagues for contributions to some of the experiments or investigations: Dr. J. Cast (Exps. 65 and 66), Dr. G. G. Jayson (Exps. 88 and 89), Dr. A. L. Smith (Exps. 6 and 51), Dr. M. W. T. Pratt (Exp. 32), Dr. G. G. J. Boswell (Exp. 63), Mr. N. Gore (Inv. 101) and Mr. P. Tickle (Inv. 112). We thank Dr. O. E. Ford, Dr. R. Stock and other colleagues for useful discussion, and our students who tested many of the new and modified experiments. In conclusion our gratitude is due to our wives for their forbearance and assistance during the course of the preparation of this work.

J. M. W.	R. J. N.
A. R. D.	R. M. W. R.

List of Symbols

EXCEPT in a few cases, the symbols used are those recommended in the Report by the Symbols Committee of the Royal Society, representing the Royal Society, the Chemical Society, the Faraday Society, and the Physical Society, issued in 1951, and amended by that Committee in 1959.

<i>A</i>	ampère.
<i>A</i>	absorbance.
Å	ångström unit.
<i>a</i>	activity, area, van der Waals' constant.
<i>b</i>	van der Waals' constant.
°C	degree Centigrade.
<i>C</i>	capacitance.
	with subscript: molar heat capacity.
<i>c</i>	concentration.
	with subscript: specific heat capacity.
<i>D</i>	debye.
<i>d</i>	density.
<i>∂</i>	partial differential.
<i>E</i>	energy, electromotive force of voltaic cell, potential of an electrode.
<i>e</i>	base of natural logarithm.
<i>e</i>	proton charge, electron charge.
<i>F</i>	Faraday.
<i>f</i>	activity coefficient.
<i>G</i>	Gibbs function.
<i>g</i>	gram.
<i>g</i>	acceleration of gravity, osmotic coefficient, gaseous state.
<i>H</i>	enthalpy.
<i>h</i>	Planck's constant, height.
<i>I</i>	ionic strength, intensity of light.
<i>K</i>	Kelvin, chemical equilibrium constant, molal b.p. elevation constant, molal f.p. depression constant.
<i>k</i>	Boltzmann's constant, velocity constant of chemical reaction, constant.
<i>l</i>	length, liquid state.
<i>l</i>	litre.
<i>M</i>	molar concentration.
<i>M</i>	molecular weight.
<i>m</i>	mass, molality.
<i>N</i>	normal concentration.
<i>N</i>	Avogadro's number.
<i>n</i>	number of moles, transport number, refractive index.
<i>P</i>	pressure, polarization.
<i>p</i>	pressure.
<i>R</i>	gas constant, electrical resistance, refraction.

r	radius.
S	entropy.
s	solubility, solid state.
T	temperature on Kelvin scale, transmittance.
t	time, temperature not on Kelvin scale.
U	internal energy.
V	volt.
V	molar volume.
v	volume.
w	weight.
x	mole fraction.
z	valency of an ion, number of electrons in an electrode reaction.
α	degree of dissociation, angle of optical rotation.
Γ	surface concentration.
γ	ratio of heat capacities, surface tension, activity coefficient, Raoult law factor.
Δ	increment.
ϵ	electric permittivity, molar absorptivity.
η	viscosity.
Λ	equivalent conductance.
λ	wavelength, equivalent ionic conductance at infinite dilution.
μ	micron, chemical potential, dipole moment, Joule–Thomson coefficient.
ν	frequency.
π	ratio of circumference to diameter.
K	conductance.
κ	specific conductance, quantity proportional to the square root of ionic strength and having the dimensions of reciprocal length.

ln, log logarithms to bases e and 10, respectively.

PART I

This page intentionally left blank

EXPERIMENT 1

Molecular Weight using van der Waals' Equation

Discussion

For one mole of gas, van der Waals' equation of state may be written

$$(P + a/V^2)(V - b) = RT$$

where a and b are constants. Hence the molar volume V is given by the cubic equation

$$PV - Pb + a/V - ab/V^2 = RT$$

Substituting P/RT for $1/V$ in the third term and neglecting the small term ab/V^2 the equation can be written

$$PV = RT - P\left(\frac{a}{RT} - b\right) \quad (1)$$

If the constants a and b are known the volume of one mole of gas at the temperature and pressure of the experiment can be calculated.

The weight, w grams, of a definite volume of nitrogen or carbon dioxide, say v litres, may be determined by experiment. As

$$M/w = V/v \quad (2)$$

the molecular weight M of the gas can be calculated.

Apparatus and Chemicals

Glass globe, wooden box, wide mouth bottle, rubber stopper, three stop cocks, silica gel tower, T-shaped glass tap, mercury manometer, vacuum pump, cylinder of nitrogen or of carbon dioxide, and lute.

Method

The glass globe is evacuated, weighed and then filled with boiled distilled water. The globe and contents are immersed in a water bath and the system is allowed to come to thermal equilibrium. During this period the tap of the globe should be left open. The weight of the globe full of water is determined. Knowing the density of the water at the temperature of the experiment, the volume of the globe can be calculated.

The globe is emptied, cleaned with chromic acid mixture, rinsed with water, and dried using alcohol and ether. The key of the tap is adequately greased. The globe is evacuated and weighed.

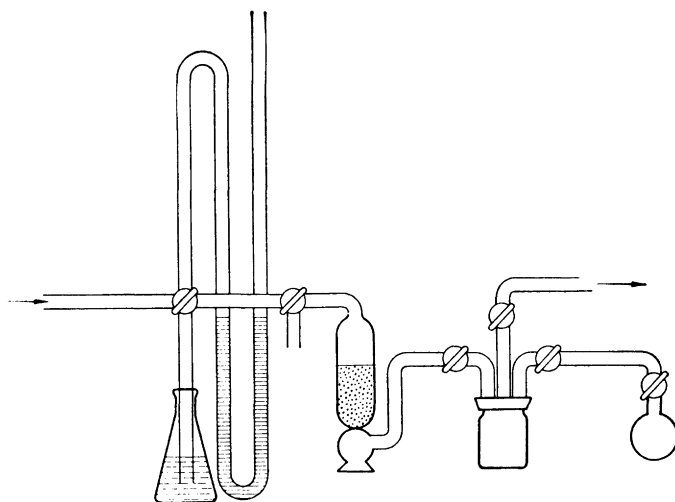


FIG. 1. Apparatus for filling gas globe.

The globe is filled with nitrogen or carbon dioxide to a pressure slightly greater than atmospheric. Whilst the globe is being filled it is kept in the wooden box. When thermal equilibrium is attained the three-way tap is opened and the pressure adjusted to that of the atmosphere, which is read from the barometer. The globe containing the gas is weighed.

The values of the van der Waals' constants a and b for nitrogen and carbon dioxide are

	a	b
nitrogen	$1.390 \text{ l.}^2 \text{ atm mole}^{-2}$	$0.03913 \text{ l. mole}^{-1}$
carbon dioxide	$3.592 \text{ l.}^2 \text{ atm mole}^{-2}$	$0.04267 \text{ l. mole}^{-1}$

These values are to be used when the pressure is in atmospheres and the volume is in litres. The gas constant R is $0.08206 \text{ l. atm/mole per } ^\circ\text{C}$. Using these values, the volume of one mole of gas at the temperature and pressure of the experiment can be calculated from equation (1).

The molecular weight of the gas can be calculated from equation (2).

EXPERIMENT 2

The Density of a Liquid as a Function of Temperature

Discussion

The density of a liquid is the mass of unit volume of the liquid. The generally accepted unit of volume is the millilitre which is defined as the volume occupied by 1 g of water at the temperature of maximum density (4°C). The density of a liquid at $t^\circ\text{C}$ is expressed relative to that of water at 4°C and is represented by the symbol

$$d_{4^\circ(\text{H}_2\text{O})}^{t^\circ(\text{liq.})}$$

The density of a liquid at a particular temperature t is the product of the relative density of the liquid

$$d_{t^\circ(\text{H}_2\text{O})}^{t^\circ(\text{liq.})}$$

(the ratio of the weight of a given volume of the liquid to the weight of the same volume of water at the same temperature) and the density of water at that temperature, i.e.

$$\begin{aligned} d_{4^\circ(\text{H}_2\text{O})}^{t^\circ(\text{liq.})} &= d_{t^\circ(\text{H}_2\text{O})}^{t^\circ(\text{liq.})} \times d_{4^\circ(\text{H}_2\text{O})}^{t^\circ(\text{H}_2\text{O})} \\ &= \frac{w'}{w} \times d_{4^\circ(\text{H}_2\text{O})}^{t^\circ(\text{H}_2\text{O})} \end{aligned}$$

where w is the apparent weight of water and w' the apparent weight of liquid at the temperature t . In order to arrive at an accurate result all weighings must be corrected for the buoyancy of air, i.e.

$$d_{4^\circ(\text{H}_2\text{O})}^{t^\circ(\text{liq.})} = \frac{w'}{w} \times d_{4^\circ(\text{H}_2\text{O})}^{t^\circ(\text{H}_2\text{O})} - \frac{0.0012(w' - w)}{w}$$

where the factor 0.0012 is the mean density of air.

The volume V_t of m grams of a liquid at $t^\circ\text{C}$ is related to the volume V_0 of the same mass at 0°C by the equation

$$V_t = V_0(1 + \alpha t)$$

As

$$V_t/m = (V_0/m)(1 + \alpha t)$$

then

$$1/d_t = (1/d_0)(1 + \alpha t)$$

A plot of $(1/d_t)$ against t will have a slope of (α/d_0) and an intercept, when $t = 0$, of $(1/d_0)$. Thus, α , the coefficient of expansion, may be determined.

The internal pressure P_i of a liquid is a measure of molecular attractive forces. It is defined by

$$P_i = (\partial U / \partial V)_T$$

where U is the internal energy. It can be shown that $P_i = T(\alpha/\beta)$ where β is the coefficient of compressibility.

Apparatus and Chemicals

Two density bottles or two pyknometers, pyknometer support, pyknometer caps, cyclohexane, benzene and a thermostat at 25°C.

Method

The pyknometer and caps are cleaned and dried thoroughly and then weighed. The pyknometer is filled with distilled water by attaching a rubber tube to one end and sucking gently whilst the other end of the pyknometer is immersed in the water.

The pyknometer is suspended in a thermostat in such a way that only the arms of the pyknometer are above the surface of the thermostat liquid.

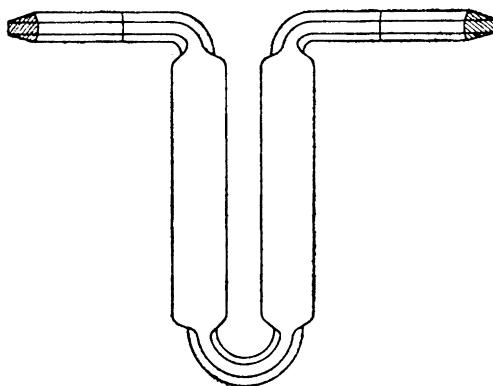


FIG. 1. A pyknometer.

After allowing 15–20 min for thermal equilibrium to be established, the amount of water in the pyknometer is adjusted so that the pyknometer is filled from the tip of the jet of one limb up to the mark on the other limb. Water may be added by placing a glass rod carrying a drop of water in contact with the jet of the limb which is already full and water may be withdrawn by placing a filter paper in contact with the same jet. A short time should be allowed for the system to regain thermal equilibrium.

The caps are placed on the pyknometer which is then removed from the thermostat and the outside carefully dried (care is taken not to expel water from the pyknometer by the heat of the hand). The pyknometer and contents are weighed after allowing a short time for them to take the temperature of the balance case.

The pyknometer and caps are emptied and dried and the above procedure repeated with the apparatus filled with the liquid under investigation.

If density bottles are used instead of pyknometers, the stoppers of the bottles should only be inserted when the contents have reached the required temperature. Density bottles are easier to use than pyknometers and for most purposes they are sufficiently accurate.

Values of the density of water at various temperatures are obtained from tables and the densities of benzene and cyclohexane are determined at four different temperatures. A graph of $(1/d_t)$ against t is plotted and the coefficient of expansion, α , is calculated. The internal pressure of each liquid is also calculated given that β has the values $1.08 \times 10^4 \text{ atm}^{-1}$ for cyclohexane and $0.94 \times 10^4 \text{ atm}^{-1}$ for benzene at 20°C .

EXPERIMENT 3

Molar Refraction

Discussion

The specific refraction of a pure liquid is given by the formula

$$R_s = \frac{n^2 - 1}{n^2 + 2} \cdot \frac{1}{d}$$

where n is the refractive index and d the density. The molar refraction R_m is given by the formula

$$R_m = R_s \cdot M$$

where M is the molecular weight.

Molar refraction is an additive property, therefore, if it is determined for different members of an homologous series the contribution of the $\text{—CH}_2\text{—}$ group may be obtained.

Apparatus and Chemicals

Abbé refractometer, semi-micropipette, cotton wool, ethyl acetate, propyl acetate and butyl acetate.

Method

The mirror of the refractometer may be illuminated with the light from a pearl lamp. Measurements may be made at room temperature. (Note this temperature for each determination.) No water should be circulated through the prism for this experiment.

A few drops of the liquid are placed on the ground-glass surface of the lower prism of the refractometer using the semi-micropipette. (Caution: do not scratch the surface, and when subsequently cleaning the prism, use a piece of cotton wool.) The prism box is closed and clamped. Volatile liquids should be introduced through the groove of the prism box. The telescope is focused upon the cross hairs in the field by turning the eyepiece. The edge of the dark band is brought to the intersection of the cross hairs and the compensator adjusted until the coloured fringe disappears and a sharp dark-white contrast is obtained. The instrument is carefully adjusted until the intersection of the cross hairs and the dark edge of the band coincide. The refractive index is read on the scale with the aid of the reading telescope.