AN INTRODUCTION

TO

LABORATORY TECHNIQUE

BY

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SECOND EDITION

MACMILLAN AND CO., LIMITED
ST. MARTIN'S STREET, LONDON
1952

FOREWORD

This book is one which should commend itself to a wide field of teachers of Science, for it contains an up-to-date résumé of the processes and technique required in laboratories. The references to the standard works and recent papers will enable the reader to pursue further those subjects which he finds of especial interest and use. In particular, the book should be of great service to laboratory assistants who have the care of apparatus, and are expected to be familiar with, and skilled in, the many tasks which are encountered in demonstration and experimental routine.

The writer of the book had many years' experience as a laboratory technical assistant and that experience he now places at the disposal of readers, and I commend the book, which contains much useful information.

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PREFACE

THE period of transition from student days and learning to that time when the imparting of knowledge has commenced is one of difficulty. The teacher faces a new world with fresh responsibilities, which are by no means diminished when the science master is confronted with the care of laboratory equipment, which is made more difficult by the lack of satisfactory assistance, for not many schools can afford the expense of a full-time laboratory assistant. The detailed knowledge necessary for this work is simply picked up in a very haphazard manner, frequently at the cost of many mistakes. In the presentation of this " introduction " the aim has been to impart the various techniques employed in the many processes and methods used in laboratory maintenance.

At the most, however, it cannot be claimed to be more than the title suggests, " an introduction "; the full intricacies and range of such work could not be brought within such a small compass as this book. Accordingly a selection, which in the writer's opinion would be the most useful to science teachers, has been made from the many kinds of work included under laboratory arts, and presented, it is hoped, in such a manner, that those with little practical training will be able readily to grasp the technique employed.

A criticism might be made of the form of presentation of the following pages, of the seeming repetition employed, or the many alternative methods of technique. It is believed that written in this particular form—the practical blended with the theoretical, and with alternative methods—it will make a more successful appeal to the would-be-efficient technician and, at the same time, allow for the preferences of different workers. Furthermore, it must be remembered that the facilities and equipment of laboratories vary greatly, while, too, the skill of the worker will be of importance.

These variable quantities then must be the deciding factor in the choice of methods. If we take for example the production of mirrored surfaces, two methods are available, the chemical and the electrical. The latter, however, can only be carried out by means of expensive equipment, beyond the reach of some institutions and otherwise unessential; in such a case the chemical method can be used with every satisfaction; but where the results required justify an expenditure upon elaborate apparatus then the alternative methods are substituted. The results, of course, will in the first place be sufficient for most practical purposes, but will be inferior to those of the latter process.

Since, too, the skill of the technician will also be a variable factor, methods which yield reasonable results with the use of average care and ability have been given. Furthermore, the " snags ", as they are generally known, have been so far as possible removed, or a way of overcoming them suggested. No mere bookwork, however, can completely fulfil the purpose for which this volume is designed; this can only be realised in its highest degree by patient and constant practice. This is necessary if satisfactory results are to be obtained, and the beginner should not be discouraged at the first failure, but should repeat the process until the technique has been thoroughly mastered. Constant attention to details, practice, and determination to succeed, can alone ensure success in this as in other realms.

Many of the processes described are entirely new, some are old and familiar, while others are the results of the labours of workers in different fields of science. In a work of this description it has been unavoidable that frequent help has been given by others in the form of printed communications and in other ways, but it is hoped that sufficient acknowledgment has been accorded in the text.

Although primarily written at the request of those engaged in the teaching of Physics, it is hoped that workers in other branches of science will find the book of service, and that all who read it may gain a better understanding of laboratory methods and technique.

The author is specially indebted to Mr. V. H. L. Searle, M.Sc.,

for the unwearied assistance given in reading over the original MS. and in making numerous and valuable suggestions. Also to Mr. C. Hellary and Mr. A. D. Harford, Dr. Cosins, and the following firms, for practical assistance and for the loan of blocks: Westinghouse Brake Co., Becker & Co., Bakelite Ltd., B.T.H., Rugby; Buck & Hickman, London; Canning & Co., Birmingham; Crompton Parkinson, Erinoid Ltd., the General Electric Co. Ltd., Johnson and Phillips, London; the *Journal of Scientific Instruments*, Newton & Co., Newnes & Co., and Phillip Harris of Birmingham.

April 1937

PREFACE TO SECOND EDITION

The issue of a second edition has afforded an opportunity of bringing the work in line with recent developments in materials and methods. Mention has been made of new insulating materials such as "Distrene" and "Perspex", which however have a usefulness in many other realms of laboratory work. The epidiascope and the film projector have rapidly gained popularity in school work; whilst new methods of slide production have extended the usefulness of optical projection. Occasion has also been taken to extend the glass-working section, giving more details of electrodes and their construction, while some mention has been made of commercial cements. The notes on the treatment for burns have been modernised.

The author wishes once again to thank Mr. V. H. L. Searle, M.Sc., for his continued interest and to Miss J. P. Ffooks for preparing the typescript; also the following firms for information and the loan of blocks: Ross Ltd., London, I.C.I. Ltd., and British Resin Products.

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1949

CONTENTS

CHAPTER I

THE CARE OF LABORATORY EQUIPMENT

SCIENTIFIC instruments and laboratory equipment are costly and often difficult to obtain, and for this reason alone care and attention should be given to keep them in good condition. Nothing reflects adversely upon the character and ability of the science teacher or laboratory technician so much as the discovery of instruments ill-used and ill-kept, and a laboratory which is generally in a poor condition. Equipment and laboratory may be maintained in presentable and efficient condition by a methodical allocation of time and attention.

Metals of various kinds such as brass, steel, copper, etc., have to be employed in instrument construction, some being more readily affected by moisture and vapours than others. Acids and chemicals, too, have a destructive effect on the wooden and metal parts of apparatus and, together with constant usage, make such deterioration almost inevitable. To counteract this tendency it is obviously essential that constant attention should be given to the full equipment of the laboratory. In recent years great advances have been made in the employment of new materials for the construction of apparatus, partially as a result of mass production and the elimination of unnecessary costs; synthetic resins are replacing wood, and polished steel has been superseded by stainless steel. There are still, however, many parts which need constant attention to prevent deterioration from the action of moisture and chemical fumes. Instrument makers endeavour as far as possible to eliminate this action by covering the exposed surfaces with a layer of lacquer, enamel or electro-plating. In the case of rubbing surfaces, such as the sliding saddle and bed of a microscope and similar parts of other instruments, this is of little use, since such a lacquer surface is readily removed by abrasion between the two metals.

Undoubtedly, prevention is better than cure, and adequate protection against these destructive agents should be made when instruments are new rather than have to counteract their ill-effects at a later period when damage has been done. Especially is this so with steel portions of apparatus, for while it is possible to eradicate the film of iron oxide which has formed on the surface, it must however be carefully done to avoid defacement. Similarly, exposed parts made of polished brass without any protective lacquer coating should be safeguarded.

Metal-protecting wax. Protection may be readily accomplished by covering the surface with a very thin film of best "White Vaseline". Alternatively the following protecting wax may be used. One part of caoutchouc is dissolved in sixteen parts of turpentine, and is then mixed with eight parts of boiled oil, obtainable from any paint store, the whole being most conveniently mixed in a vessel placed on a water bath, and applied to the steelwork, while warm and molten, by means of a good quality paint brush. This coating may be easily removed on any occasion by means of a cloth soaked in turpentine.

Cleaning of bright steel and iron. The first intimation of corrosive action is given by the readily noticeable appearance of small specks of rust upon the iron or steel. The removal of such foreign particles may be accomplished by using a special instrument soap applied to the affected parts.

Special instrument soap. Add to a solution of potassium cyanide in water an amount of precipitated chalk until a cream-like paste is obtained; to this, add fine shavings of white Castille soap, mixing the whole together until thoroughly incorporated. The article to be cleansed should be first immersed, if at all possible, in a solution of one part cyanide of potassium in four parts of water and kept immersed until the dust and dirt have disappeared.

It should then be polished with the instrument soap and thoroughly dried by means of a soft dry cloth. An alternative method is to lay the instrument or its infected parts overnight in a saturated solution of chloride of tin, when the rust spots will disappear through reduction. Upon removal from the solution, the instrument should be rinsed with distilled water, placed in a hot soda-soap solution and then dried. This should be followed by cleaning with absolute alcohol (industrial spirit) and, if it is desired to polish the metal, polishing chalk should then be applied.

Unfortunately, however, it is not always possible to immerse instruments or their parts in such a solution because of their bulk, or because the employment of other materials in their construction renders such a procedure impracticable. A modified method such as that described below is worthy of trial: Best olive oil is poured on the affected parts of the instrument, and allowed to remain there some days. The rust spots should then be rubbed with very fine emery or tripoli powder by means of a cloth, without wiping off the oil, as far as possible, or always bringing the oil back again on the affected area. After this operation the powder and oil should be removed with a cloth, the surface again being treated with emery soaked in vinegar and rubbed well into the metal, which is finally treated with fine plumbago used in conjunction with a chamois cloth.

In the case of iron upon which the oxide has been allowed to accumulate for a considerable time, the following cleaning paste will be found invaluable. Equal parts of tripoli powder and flowers of sulphur are mixed with olive oil to form a thick paste. This is applied with a cloth to the encrusted parts, and briskly rubbed in and then wiped off. When, however, the iron has rusted only recently, the spots should be rubbed with a cork charged with oil; this will result not only in the removal of the film of iron oxide, but will also produce a very fine polished metal surface.

The treatment of nickel. Films of oxide deposited on nickel will have to be treated by another method, as follows. The article should be first well greased and after a few days vigorously rubbed with a cloth charged with ammonia; if the spots still persist, a few drops of hydrochloric acid should be added to the previously used chemical and applied to the metal in the same manner and then immediately wiped dry. The article should then be rinsed in water, dried and polished with tripoli powder.

Groggins and Scholl* have described the use of o-dichlorobenzene as a suitable cleaner for this metal and for copper and silver. It was found particularly useful in the case of metals heavily

* *Ind. Eng, Chem.* **19**, 1029, 1927.

coated with grease or where tarnishing had taken place very badly. In their experiments copper was used and the results show that this cleaner was far superior to many of the ordinary liquids and pastes employed specially for this metal. The following is the formula used. A paste is prepared by adding one part of precipitated chalk to five parts of the liquid and stored in a well-corked bottle; it will be found that by the use of these proportions the fluid is sufficiently mobile to be poured readily from the bottle. Since this liquid has its own particular odour, small quantities of aromatic oils incorporated in the mixture during the preparation process will render it more agreeable to the worker. Its advantages are that it contains no acid or alkali; it does not scratch or corrode metal, nor does it vaporise quickly.

Cleaning of lacquer surfaces. Metals protected by some form of lacquered coating require but little cleaning; a periodic rubbing of the surface with a polishing cloth should be found sufficient.

Metal-cleaning paste. If no such protecting surface has been applied, constant cleaning of the metal is imperative if an ill-kept appearance is to be avoided. A suitable paste may be made by mixing together 1 oz. of oxalic acid, 6 oz. of rotten stone and ½ oz. gum arabic previously well powdered and with 1 oz. of sweet oil added. To make it into a workable paste, sufficient water is added to this mixture to give a creamy consistency, and it is then applied to the metal with a cloth and rubbed dry with a flannel or polishing cloth. Alternatively the following paste may be employed. Mix 2 parts of oxalic acid, 7 parts tripoli powder with 20 parts of water and use as in the former case.

When metal surfaces are used in such a manner that no rubbing of frictional action takes place, it is advisable to apply a suitable coating of lacquer where this is not already provided. Although the technique of lacquering is somewhat difficult to the inexperienced, the time used in mastering the method will be well spent; details of this process will be found under the section Lacquering, on page 17.

The Cleaning of electroplated articles. To clean metals which have a deposit of another metal on them such as silver-plated articles, *e.g.* the scales of instruments, parts of microscopes and spectrometers, care must be exercised in the selection and application of a suitable cleaning powder, since the deposited layer is extremely thin and easily removed. Although many excellent polishes are marketed, none should be employed unless it is certain that there is no gritty matter, which would be bound to cause damage in application. These foreign particles are found in the absorbent material used to make the cleaner.

When it is possible to remove the portion of the apparatus so plated for cleaning purposes, the paste described below will be found most suitable. Mix together 2 parts cream of tartar, 2 parts of chalk, and 1 part alum. The alum should be well powdered and thoroughly mixed with the other ingredients and placed in a wide-mouthed bottle with a tightly-fitting cork. To use, wet sufficient powder and apply with a very soft linen cloth—this is absolutely essential since scratches are readily produced on such surfaces—to the plated material, using as little pressure as possible during the operation. Rinse in hot suds, wash in clean water and thoroughly dry in sawdust. It frequently occurs, however, that the removal of such parts is impossible, in which case the use of another cleaning powder has much to commend it. Mix together equal parts of magnesium carbonate, chalk and rouge, apply with a damp cloth and polish with a dry flannel.

Balance pans. Balance pans, no matter how carefully used, in time get dirty; unfortunately, however, no matter what method of cleaning is adopted, a very thin film of metal is removed during the process, necessitating the readjustment of the balance.

If the pans are merely tarnished by oxidation the very smallest amount of non-gritty metal polish should be placed on a cloth and rubbed over the surface. When the pans are covered with grease they should be dipped in a very weak solution of ammonia, washed with water, dried, and then polished with metal polish. Even if great care is taken, chemicals accidentally get spilt and react with the metal; in such a case it will often be necessary to dip the pans in a very dilute solution of nitric acid. These processes destroy the plating, but such pans can be re-electroplated at the cost of a few pence, and the balance again adjusted for use.

Plating by chemical paste. Since the deposited metal, as already pointed out, is extremely thin, the surface is quickly removed by constant usage, thus rendering the scales and verniers difficult to read and reducing the value of the instrument. Such parts of the apparatus can have their surfaces restored by re-plating, either at a plating works or by plating on a small scale—a description of the method will be found in Chapter V. When such treatment is thought to be unnecessary a very simple means may be adopted by employing a special form of silvering powder which is rubbed into the metal with a cloth. A word of caution is necessary, however, in the use of this mixture, on account of its very poisonous nature; it should not be allowed to come into contact with the hands. Potassium cyanide, 12 parts, is mixed with 6 parts of silver nitrate and 30 parts of calcium carbonate are added. The whole is placed in a bottle—preferably coloured and labelled " poison "—which is kept well corked. It must be applied to the desired spot with hard rubbing; the bright surface thus produced is afterwards rinsed in water, dried and polished in the usual manner.

Silver cyanide method. Prepare two solutions as follows:

(*a*)	Silver nitrate - -	1 part
	Distilled water - -	50 parts
(*b*)	Potassium cyanide -	3½ parts
	Distilled water - -	50 parts

Mix the two solutions together and heat to 82° C. in an enamelled vessel. Then place the articles to be coated in the solution until a uniform coating has been obtained.

Bad tarnish stains on silver-plated scales, etc., caused by exposure to an atmosphere very heavily heated through gas burning, may be removed by rubbing with a cloth coated with the following preparation:

Potassium carbonate - -	12 parts
Castille soap - - -	¼ part
Sodium chloride - -	12 parts
Distilled water - -	48 parts

Optical instruments. The use of optical instruments in the laboratory calls for special attention and care, due to the fact that the component parts of such instruments are of very delicate construction and are not so well protected as other instruments which are largely mounted in cases. This, of course, results in the more rapid contamination of exposed surfaces and, wherever possible, these instruments should be placed in convenient cupboards when not in use in the laboratory. When this is impossible, *e.g.* when an experiment has not been completed, a useful form of protection may be provided by covering the instrument with a square of black satin cloth of such a size that no part of the instrument is left exposed.

Not only is it necessary to afford adequate protection and care when in use, but the same attention must be given during the cleaning of such instruments, and especially is this applicable to the lenses, mirrors and prisms of such apparatus.

It will be realised that unless the glass parts are scrupulously clean the definition of the lens system will be seriously impaired by the presence of finger marks and the accumulation of grease and dirt on the surface of the glass. Lenses should be removed from their cells or containers, cleaned and then replaced.

The removal of a lens. This is accomplished in the following manner. Remove the screws which hold the ring in position, or alternatively unscrew the holder and lift out from the body of the tube. Place the fingers so as to grasp the lens on the opposite sides; reverse the cell, and with the thumbs gently press the objective squarely out of the cell on to a block a little less in diameter than that of the lens, which has had a cushion of soft material laid upon it. Before separating any component lenses they should be carefully marked on their edges, if this has not already been done, by means of a hard lead pencil, so that when they have been separated they may be replaced in the same way and position in the cell.

The cleaning of lenses. The essential requirement of the cleaning process is the restoration of the original clean and polished surface without damaging the face of the glass in any way. Throughout the entire operation extreme care must be used because of the relative hardness of dust particles, which are apt to accumulate under the polishing cloth. Experiments indicate that particles of dust, having attached themselves to the cleaning material, penetrate the surface of the glass and make microscopic scratches in the surface after pressure has been applied to the cleaning pad; so that in the selection of the latter, it must be scrupulously clean and frequently renewed. To avoid these difficulties the following method has been successfully developed for glass surfaces in very bad condition, removing grease, dirt, etc., with ease and imparting a highly finished polish to the glass.

A paste of benzene and magnesium oxide is made of such consistency that, when the mass is pressed together between the fingers, a drop of benzene will exude. By means of a small wad of cotton wool the paste should be applied to the surface of the glass, rubbing it well, but at the same time gently. After drying, any remaining paste should be removed by means of a soft camel-hair brush.

Immersion type lenses. Lens objectives of the immersion type should be wiped clean by means of a fine piece of cambric moistened only with xylol; the use of spirits of wine must be absolutely avoided, as in some instances it readily dissolves the cement holding the lens parts *in situ*, as also do certain forms of cedar oil.

Optical glass may also be cleaned by washing the material in hot water and soap, using pure Castille soap or sodium oleate, and a swab of cotton wool. The soap should be washed off with alcohol and the glass rinsed with distilled water. If the water wets the surface uniformly, the cleaning is satisfactory; if not, repeat the operation. The glass should be finally dried with a clean chamois leather, care being taken that the fingers do not touch the surfaces.

Diffraction gratings. The majority of gratings employed today are celluloid—cast replicas of the original ruled Rowland grating. Dr. J. Fox* has pointed out that the easiest method of cleaning this type of grating is to take a collodion cast from it. A quantity of best quality gun-cotton is dissolved in amyl acetate and the solution is poured into water, the precipitated gun-cotton being filtered off and thoroughly dried. It is again dissolved in amyl acetate and the resulting collodion is then poured over the grating, care being taken that all dust is excluded. After being allowed to dry slowly the film is carefully removed from the grating, leaving behind a clean grating face.

Cleaning of mirrors. As with lenses, mirrors have to be handled

* Baly's *Spectroscopy*, vol. 1, p. 182.

very carefully in the cleaning process. The method of procedure is as follows. Rub the mirror very carefully with a ball of soft paper slightly damped with methylated spirit; then with a clean cloth on which a little whiting has been sprinkled; finally polish with a clean paper pad or chamois cloth. Oil spots on glass may be removed in the same manner.

Spectrometer slits. Brass is usually employed in the construction of the slits and framework of this part of the spectrometer, its only disadvantage being that it readily corrodes, and for this reason and the fact that it is somewhat soft for this purpose, platinoid is often substituted in the more expensive and accurate form of instrument. Since the jaws are of such delicate construction, care must be taken in their adjustment, avoiding the tendency to bring the edges too close together and thus damaging the vital part. Frequent tests should be made for the parallelism of slits wherever they are employed, as follows. The slit is removed from the instrument and examined by looking through it at some source of light, at the same time closing the aperture by rotating the micrometer screw until the light just disappears. If the slit is properly adjusted the aperture will disappear entirely along its whole length, but if one end closes first leaving a wedge-shaped opening, the jaws are out of alignment and may be corrected by the adjustment of the jaws themselves in their sliding holders. Upon examination it may be found that while the jaws are parallel, dust particles are projecting at the edge. The rapid removal of these may be accomplished by sharpening to a point the end of a piece of lancewood, obtainable from any watchmaker, and lightly moving this end of the wood along the edge of the contaminated slit, using as little pressure as possible.

Replacement of cross wires in microscopes and telescopes. Since there is a frequent need for the replacement of cross wires in optical instruments, a description of the different methods employed in their mounting may not be out of place. In the case of low-power eyepieces, or where it is not essential to read to very accurate limits, cross wires of single threads of silk are very satisfactory.

The cross-wire ring having been removed from its holder, the cement used to attach the old wires is removed—preferably with

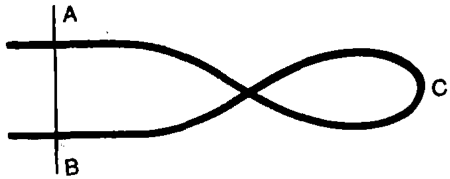

Fig. 1

the aid of a solvent—when there will be found scribed upon the ring two lines at right angles. These are indispensable for fixing the fibres at right angles and, if obliterated, should be scribed on the metal ring with the aid of a needle and rule. In mounting the fibre in position across the scribed lines the difficulty to avoid is that of the sagging of the wires after they have been fixed. A piece of brass wire, bent to the shape shown in Fig. 1—the distance between AB being about twice the diameter of the cross-wire ring—will greatly assist towards overcoming this difficulty. A fibre of unspun silk is now split into a single thread with the aid of a fine needle, and one of appropriate length and size selected and fixed by means of cement across the brass frame at the point A and, when stretched sufficiently, the other end is fastened at B.

The fibre should then be brought into position across the scribed lines on the ring, allowing the end C of the brass frame to rest on the table, the other supporting the fibre as it rests on the ring. Then with a pin the cement is applied (shellac varnish, but preferably beeswax) and when set the superfluous fibre is cut away with a sharp pair of scissors. The second wire is cemented in exactly the same manner, the scribed lines ensuring that the lines are at right angles.

An alternative method of performing the same operation dispenses with the brass frame and is shown in Fig. 2. The fibre having been selected as described, one end is fastened to the table-top by means of wax, such as soft red or beeswax, and the fibre brought into position over the scribed lines. A slight pull is then put on the fibre at the point X, in the opposite direction to the anchored end, and at the same time it is brought into contact with the ring; the amount of pull will of course be determined through the experience of the worker. The fibre is then cemented

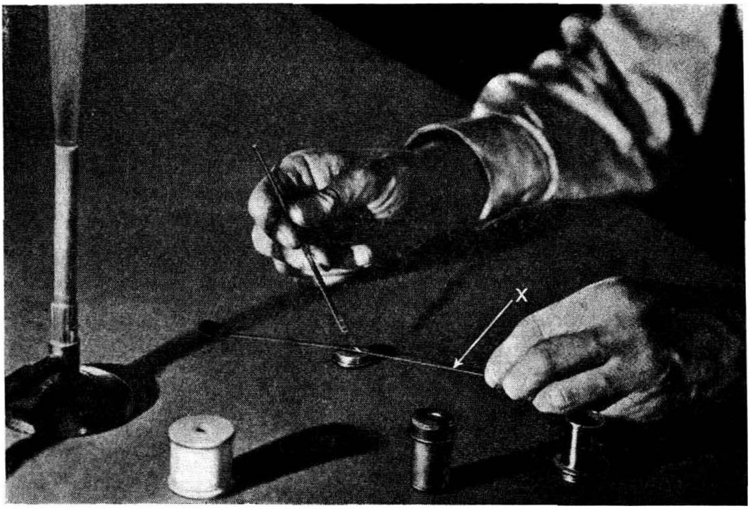

FIG. 2

into position as before. In the hands of a skilled worker excellent results and a great saving of time may be obtained by this method.

For accurate work these fibres are far too coarse and the best material to employ is a silk cocoon or a spider's web, although glues have been developed with success for this purpose. A spider can usually be persuaded to spin a thread by tilting him off a card. This thread is then used, with the assistance of the wire holder as already described above, in the mounting process.

Drummond* has described the use of glue for the production of cross wires, with the advantage that the technique involved is extremely simple. A little " Seccotine " is placed at the required spot on the cross-wire ring, and then a fibre is drawn out with the tip of a needle, allowed to harden slightly before being lowered into position, and fixed at the free end with another spot of glue.

This operation is again repeated for the other wire; the quality of the result, which depends on the rate and extent of drawing and the temperature, is determined largely by the experience of the operator. Drummond obtained fibres of various diameters by the use of different types of glue, viz.: Silkworm cocoon gave

* *Jour. Sc. Instr.*, **10**, 259, 1933.