COMMERCIAL METHODS OF ANALYSIS

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and

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WITH PHOTOMICROGRAPHS BY
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PREFACE TO SECOND EDITION

The favorable reception accorded the first edition of *Commercial Methods of Analysis* has led us to prepare this revised edition. In the intervening twenty years, there have been introduced many methods of carrying out special determinations often characterized as the "sophisticated" methods because of their complexity. Typical examples are infrared analysis and gas chromatography. They are so specialized that each would require a book for any comprehensive treatment. Therefore, it has been the judgment of the authors not to try to introduce them.

So changes are surprisingly few. Analysis of minerals has not changed over these past twenty years and it is improbable that it will change over the next twenty. The same statement applies to many, many other determinations. The refractometer shown looks different in this edition from the model of twenty years ago but it operates in the same way. Our thanks go to the manufacturers and distributors for many illustrations of new apparatus and equipment.

Even the comments in the preface to the first edition are equally cogent today, so we suggest that you review it.

FOSTER DEE SNELL,
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NEW YORK, N.Y.,
March, 1964.

PREFACE TO FIRST EDITION

Any new book in the field of analytical chemistry should present new material, and preferably a new point of view, in order to justify its existence. Many meritorious and deservedly well-known textbooks have been published on theoretical analytical chemistry. This book has another purpose—to show the method of approach to analysis of the innumerable complex commercial products existing on the market today. Many of these are colloids, some are emulsions that may contain half a dozen ingredients besides water and oil, a majority contain organic as well as inorganic ingredients, and as unknown samples most of them would present problems extremely difficult of solution by the inexperienced chemist.

In order to make the book of more universal use, standard methods, such as those given in the "Methods of Analysis of the American Society for Testing Materials" and the "Methods of Analysis of the Association of Official Agricultural Chemists," have in many cases been introduced, though often not in the exact words of these methods. In line with the general scheme of the book, simplifications have been adopted in some parts, and, what is more important, explanations of steps, the reason for which is not sufficiently clear, have been given. Standard methods are not given in full, only such determinations as seem necessary to the general analyst. If a sample must be analyzed in detail, strictly in conformance with these official methods, the original methods should be referred to. Other, usually considerably shorter, methods are sometimes introduced.

Particular care has been taken to set forth in logical sequence the preliminary steps that necessarily precede the actual analysis of samples of unknown composition. Both in this and in subsequent procedure, an endeavor is made to clarify the meaning of each step and often to give the reason for it. This is to develop the inexperienced analyst into an experienced one by logical methods, to show the importance of what may at first seem to be unimportant details of procedure, details which, if omitted, will in many cases lead to incorrect results. Methods are as simple and direct as possible, compatible with accuracy. Where data

are available, limits of accuracy are discussed as to what is desirable and what is attainable for a particular type of sample, or sometimes for a particular application. The practical point of view of the commercial laboratory has been emphasized by methods that aim at general economy of time and materials without sacrificing reliability of results.

A feature of the book is an attempt to set forth succinctly, frequently by stating the reactions involved, the methods of calculating the results of determinations in order to show reason for the formula given.

As a matter of organization, after such routine and widely used determinations as those for nitrogen, sulfur, and halogen compounds, only commercial types of samples are considered. Condensation has been accomplished by profuse cross reference to methods in other parts of the book, in nearly every case to specific portions rather than to general paragraphs or chapters.

Special methods have been included that have been found to be saving of both time and labor. Many modern methods of analysis and methods for analyzing of substances of recent importance, such as synthetic plastics and synthetic elastomers, have been included, even when only qualitative information is available. Emphasis is also laid on the growing importance of the determination of minute quantities.

The useful new branch of qualitative analysis by spot tests is condensed to one chapter (Chap. 5). The Precision Scientific Company of Chicago has designed a micro kit (Fig. 69) for use with this book. It contains only the reagents required in Chap. 5.

It is hoped that this volume will prove a useful text to the student who has already become familiar with the tools of quantitative analysis and the routine methods, and who desires further training in this field by a study of commercial products. It may also serve as a manual to the industrial analyst whose work is not confined to simple control operations.

Many of the methods and ideas presented here have been developed and used over a period of two decades in commercial laboratory operations. As with most things new in the field of chemistry, much time, effort, and research have gone into their making.

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NEW YORK, N.Y.,
July, 1944.

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COMMERCIAL METHODS OF ANALYSIS

CHAPTER 1

GENERAL

If a chemist were given a commercial material of a specific type and asked to determine its composition, he might well be in a quandary as to how to approach the task unless he had much prior experience with that specific type. A sample is rarely analyzed without some general preliminary information as to its probable composition; but, equally definitely, such preliminary information is rarely sufficiently comprehensive to indicate all the materials present.

The ultimate purpose to be served by the analysis is important. Lack of this essential preliminary information may lead to much lost time in doing unnecessary work. How accurate need the results be? Time should not be spent in the use of highly accurate methods when a briefer and less accurate procedure will give all the information needed. Conversely, and more commonly, work that is not of adequate accuracy may vitiate all the results obtained.

Is it essential to find out everything possible about the sample, to do a complete analysis? In many instances, proper advance information will indicate that only part of the analysis will ever be used. How much of the sample is available? Considerable advance planning must often be done to get the necessary results from a small sample. Can two or three or more determinations be made on the same portion of the sample? This must often be known in advance to permit such use. What size of sample is desirable for each determination? Naturally this depends on the amount of the substance being determined, which should be known within some general limits before proceeding. What methods are to be used for each determination? These and many other questions must be answered by the analyst before he weighs out the first part of the sample.

Purpose of the Analysis. Analyses are not made merely to be decorated with the signature of the analyst and filed. Each has one or more purposes. If the purpose is to present a comparison with a competitor's product, the approach is not the same as it is if the analysis is to serve as the basis of determining whether the product is of good quality, and that in turn differs from an analysis for the purpose of permitting manufacture of a similar product. It is necessary to find out as precisely as possible the purpose of the analysis.

It is well at the beginning to stress what will always be tacitly assumed throughout, that in commercial practice economy of operation is paramount. The ability to do a thing well and efficiently is ever desirable and one of the marks of a well-trained and practical chemist; more especially is this so in applied chemistry. Business, to be profitable, must be run on sound economic principles. If the making of chemical analyses is a minor or major part of that business, they, as much as any other part of the business, must be performed with a view to a favorable balance sheet in terms of time and materials.

Accuracy. To the worker who takes pride in his work—and one who does not is not worth his salt—raising the question of accuracy may seem almost sacrilegious. Reflection, however, will indicate that results with accuracy to parts per million are only occasionally needed. In determining the amount of abrasive in a tooth paste, for example, accuracy within 1 per cent will often be sufficient; a tube of that brand from another batch may vary by more than that. In determining the amount of fatty acid in a fatty oil, an error of 0.1 per cent may make the difference between the product's being classifiable as an edible oil or as semicrude for further refining. An error of a few parts per million in the arsenic content of a dyestuff may mean the difference between a quality suitable for certification and an industrial dyestuff at a fraction of the price per pound.

In a check against specifications, will the person receiving a report be grateful for a result reading to 20.86 per cent when the requirement is that an ingredient be between 20 and 22 per cent? Clearly this will take longer than to obtain a result of 20.8 or 20.9 per cent, if only in longer time for weighing or longer to get the end point in titration, and longer to calculate. So, the accuracy—and the method—should be suited to the requirements.

It is equally true, and perhaps this is the more common fault, that lack of sufficient accuracy nullifies work not only on that

determination but on others. A short method that for other purposes may be excellent may become but waste of time for the sample on hand.

Poor, slipshod work will always give inaccurate results, and it may, though it should not, be necessary to warn against this. On no account should such work be tolerated, whether results are being expressed as one part in ten or one part in ten million. There are times when, to obtain any results at all, in the nature of the case a higher degree of accuracy is obtained than is necessary. When this is unavoidable, it should occasion no worry.

In general, it may be said that the determination of the amounts of an element in parts per million involves special methods that are usually far more lengthy than less accurate procedures.

Size of Sample. This is governed by several factors. If only a small original sample is available, more thought and ingenuity must be used. It is remarkable how much information can be extracted from very small amounts of a sample by careful forethought. Much real satisfaction is experienced by the worker who is able to get all the information and accuracy required from what would normally be called an inadequate size of sample by judiciously arranging the order of the determinations. A certain sense of what is best and fitting becomes part of the analyst's intuition, an intuition that, however, is based on logical chemical reasoning based in turn on sound chemical knowledge. Analysis is the application of known chemical facts in a logical sequential manner.

Even if the sample is sufficient, preliminary thought as to how much to use will be amply repaid. Large samples that give bulky precipitates and are usually unwieldy all through the analysis should be avoided. It is well to aim at obtaining a precipitate weighing less than 1 gram, preferably about half of that; a colored solution that can easily be matched; or a titration value of, conveniently, between 20 and 40 ml. Such figures often cannot be obtained, and the above must be looked upon not as hard and fast rules but rather as indicative of what may be termed optimum conditions. Experience is the best teacher, and even a short period can enable one to become quite expert at judging what is required.

At the same time, too small a sample should be avoided if possible. The experimental error becomes proportionately greater; and, theoretically at any rate, a limit may be reached at which

this error is equal to the actual determination. In these cases, and also when any blank that may be made is nearly as great as the determination, the degree of accuracy is seriously impaired. Experimental error varies both with the worker and with the method. In the first case, the experimental error will, with a reasonably good worker, not be of great consequence. In the second case, a large experimental error may be inherent in the method used. If this is so, it is essential to become aware of the fact before employing such a method.

Choice of Method. The many methods presented in a textbook as available for a certain determination may well be a source of worry. What method is best under the circumstances is a question that can be answered only by careful attention to these methods. First of all, if possible, find out what other substances are in the sample. Then it may be found necessary to eliminate one or more of the given methods as this or that other substance interferes or may have to be removed before the estimation can be made.

If the approximate amount of the required element is known, this may also limit the methods that can be employed. Choice may also be made advantageously with a view to further work that may be made on the same portion of sample. Ease of manipulation and availability of apparatus may be determining factors, and the method chosen may be decided by the required accuracy of the result.

In any event, the methods given should be read carefully with particular attention to the effect of interfering substances. The whole determination may become invalid if the worker fails to pay sufficient attention to any such particulars. This source of error is very difficult to trace if it is supposed that a given method has been properly followed; and not only is an inaccurate result derived for the element under consideration, but the whole analysis may be wrongly interpreted.

That the method selected should be thoroughly read, understood, and followed might seem a most obvious fact. Nevertheless, it is emphasized here because experience has shown that many errors and incomprehensible results are traceable to careless reading and interpretation. For the most part, there is a sound theoretical reason for each step given, and no deviation should be made without an equally sound reason. Unfortunately, little explanation is, as a general rule, given as to why certain procedures are to be followed. Even this failure may sometimes be turned to good account in that

the worker is enabled to find out the reason for himself from theoretical considerations. With beginners, however, this is frequently not possible, and even experienced chemists may have real difficulty in this respect. To sum up, for this matter is of real importance, read the method carefully, find out as well as possible the reason for each step, and follow it faithfully. Let it be said that most so-called short cuts turn out to be otherwise. This does not preclude the possibility that an experienced person can sometimes improve the method and modify it to suit his particular requirements. But that experienced person will not take the short cut unless his experience shows it to be satisfactory, and that means that at some time he blazed the trail.

Calculation of Results. Another matter that seems self-evident should be mentioned. Good work needs equally good mathematical interpretation. A reliable analyst is necessarily a reliable arithmetician. One little error in calculation has wasted many a day's work and many a firm's money. In working out results, four-place logarithms are usually the preferred tool. A 20-inch slide rule possesses nearly the same accuracy and is not unduly expensive considering the time it saves. It is acceptable for most commercial calculations. But even if calculated on the slide rule, all figures used in the computation should be set down so that they can be checked. Results should ordinarily be checked by an associate. For this purpose a 10-inch slide rule has adequate accuracy and saves time. Here too, the greater ease of reading the 20-inch rule justifies the higher cost. All work sheets should be considered a part of the analysis and filed with the original data. A worker may have to check back on his results tomorrow or next week or next year.

Results are usually reported as per cent and calculated to the first or second place of decimals. To go beyond the second place in dealing with whole percentages is a waste of effort and quite out of keeping with the accuracy of usual methods. When the result is entirely in a fraction of a percentage, normal accuracy to one or two parts per thousand dictates that results be expressed as three significant figures. Often the details of the method will indicate a lower accuracy, and two significant places are all that are justified.

Consistency and order should be aimed at in calculating and presenting results. Unless there is sound reason for doing otherwise, such as that one substance is present in a comparatively large quantity and another in a very small amount, all results of a particu-

lar analysis should be given calculated to the same decimal place.

As a final check on himself, a worker should make sure that the correct conversion factor for the weight of precipitate to its derivative or the appropriate normality factor of acid, alkali, or other titrating medium, the proper weight of sample, and the proper aliquot of that sample have been employed.

CHAPTER 2

TOOLS OF THE ANALYST

Although the analyst is a professional man, he has his tools just as do men in other professions, for example, the surgeon, and an intimate knowledge of their virtues and limitations is essential. Only some of the more important typical tools can be discussed. Numerous illustrations are used to save space even though the item shown may be of a well-known type.

Beakers and Flasks. The majority of operations that involve heating are carried out in beakers or flasks. The glass of which they are made may be divided into borosilicate and all other kinds.

Pyrex is a brand of borosilicate glass with a sufficiently low coefficient of thermal expansion, 0.0_532 per degree centigrade, so that it can be and is made of substantial thickness. Breakage due to heat strain is so rare as to be properly attributable to an overlooked crack or other defect. It is reasonably but not entirely resistant to alkalies. It is glass, however, and will crack or shatter on violent impact with hard surfaces.

A special low-alkali type of Pyrex is rarely referred to because of its limited availability. A newer type, Vycor, is used as crucibles and otherwise as a substitute for fused silica. Its coefficient of expansion is about one-fourth that of Pyrex, for all practical purposes negligible. Because of cost, it is not apt to be much used as beakers or flasks.

Some laboratories stock only Pyrex-ware beakers and flasks; in others a type of soft soda glass is also used. With a coefficient of thermal expansion about three times that of Pyrex, soda glass is best used for operations in which heating is not performed and attack on the glass is of no great consequence. When soda glass is used as beakers and flasks, it should be rigorously kept separate from Pyrex; failing that, the chemist should look at the brand mark before heating the contents. Failure to do one or the other may result in lost time and patience.

Availability of a generous supply of glassware represents an economy in laboratory operation. The majority of operations in

beakers are carried out in 250- or 400-ml. low form sizes with a spout (Fig. 1). This is also known as a Griffin beaker. Similarly the 250-ml. conical flask with wide or narrow mouth (Figs. 2 and 3) possesses a wide utility. This is also known as an Erlenmeyer flask. For titrations, such a flask possesses definite elements of superiority to beakers. Thus the titrating medium is run in from the buret with one hand by the experienced operator while the flask is swirled with the other hand. Prompt mixing without danger of splashing gives real economy of time.

Such apparently minor points as these are illustrative of many that must receive consideration for economical operation. It will be found that the employment of methods that are as simple as permissible for the purpose will in the aggregate make for both speed

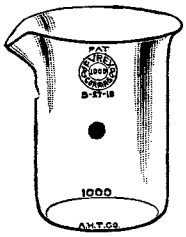

FIG. 1.—Low form beaker with spout.

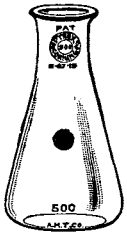

FIG. 2.—Widemouthed conical flask.

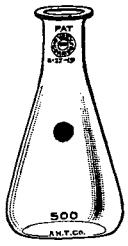

FIG. 3.—Conical flask with narrow mouth.

and skill in performance. Good manipulative ability coupled with the choice of equipment best suited to the job in hand is a big step forward to success as an analyst. It is to attain this end that many points that may appear obvious but that, as experience shows, are often neglected are mentioned from time to time.

Volumetric Glassware. This is normally of soft glass. Little advantage is gained by use of the initially more expensive Pyrex.

Two main classes of volumetric ware are on the market. A certificate of accuracy within certain close limits, issued by the National Bureau of Standards, is supplied with one class. The other class is stated to conform to wider but still reasonably close limits of accuracy. The former costs appreciably more than the latter and may, especially in the case of burets and pipets, be worth the extra expenditure. It is good practice if time and facilities permit to purchase the latter type and calibrate it. For the majority of purposes no further degree of accuracy than that already present is required.

A word as to keeping volumetric apparatus clean is in order. The tips of pipets and burets are liable to become clogged, particularly if solutions immiscible with water are allowed to dry in them. Much labor is needed to clean such equipment, and often during the process the piece may be damaged. It is time well spent to clean such glassware just after it is used. A very fine wire, judiciously

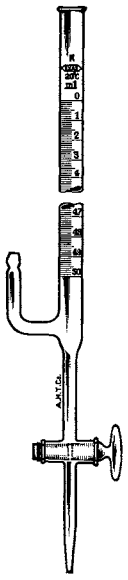

FIG. 4.—Standard buret with side arm for refilling from reservoir.

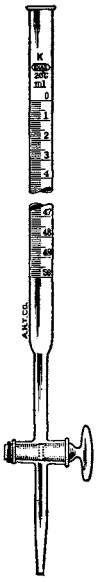

FIG. 5.—Standard buret.

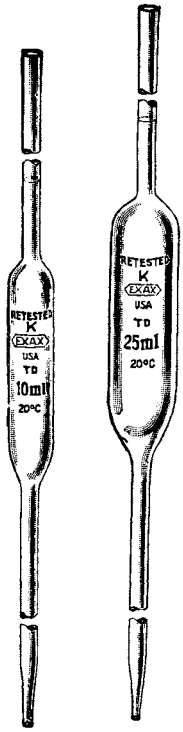

FIG. 6.—Pipets.

applied, helps in cleaning out clogged tips. Stopcock grease or the petroleum jelly commonly used for the purpose of lubrication should not be applied too generously.

Normally soap and water, preferably with the addition of a little trisodium phosphate, sodium metasilicate, or soda ash, will serve to clean glassware. When any substantial number of chemists are working in the same laboratory, it is good economy to accumulate soiled glassware and have the junior of the group, if no labora-

tory boy is available, do a general cleaning at regular intervals, not less frequently than daily.

Chromic acid or sodium bichromate in concentrated sulfuric acid, often simply called "cleaning solution," is a very efficient cleaner, particularly if it is hot, for removal of organic contamination. Burets (Figs. 4 and 5) and pipets (Fig. 6) have a tendency to become greasy in continuous use and should therefore be cleaned at reasonable intervals. If this is not done, the correct volume of liquid will not be delivered, because of surface tension effects. Hot

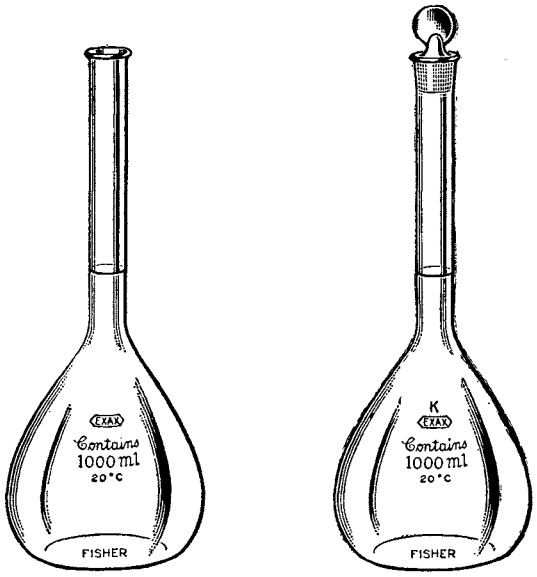

FIG. 7.—Volumetric flasks.

concentrated sodium hydroxide solution is effective in many cases for greasy glass or when material is burned on. It should not be used on graduated or calibrated glassware (Fig. 7), for it will attack glass to some extent. Care must be exercised in use of either of these cleaning solutions, because they are very corrosive to the skin.

Glass Joints. Laboratories use relatively few pieces of equipment with ground-glass joints, other than burets and separatory funnels. The most common is a stopcock. However, nearly every laboratory has some. By agreement among manufacturers, such joints of more complex apparatus are of standard size and taper so that units are interchangeable and replaceable. For such types of equipment as are so provided, it is good economy to get the replace-

able type. Glassware will get broken, and the purchase of a complete replacement unit is thus avoided.

Watch glasses in large numbers are an essential item of laboratory equipment. They are of ordinary soda glass or of Pyrex. Necessarily large and small sizes are used to fit the varying sizes of beakers, but the major use is to cover 150- , 250- and 400-ml. sizes. When used to prevent contamination by dust and dirt, they may well fit closely, with steam escape provided at the spout of the beaker. But often a beaker is covered during evaporations, to prevent contamination and to avoid loss of material by spattering. Then close fitting is a defect. Special glasses for this use are ridged to hold them from contact with the lip of the beaker other than at a few points (Fig. 8).

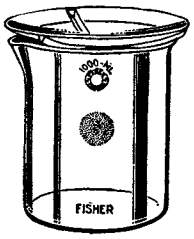

FIG. 8.—Ridged watch glass in use.

Equal effectiveness but less convenience is provided by supporting the watch glass on a glass triangle laid on the top of the beaker or by placing three to four small glass hooks around the edge of the beaker to support the watch glass. In such cases a somewhat oversized watch glass is preferable, for it furnishes better protection from adventitious contamination by dust. Depending on the use, the watch glass may require rinsing into the beaker, or even cleaning with a policeman if spattering of a precipitate has occurred.

Stirring Rods and Ebullators. Stirring rods are a necessary adjunct of the chemical laboratory, even if they are used too often in titrating. One of the most common practices is to round off each end in a Bunsen flame. Experience, and this is backed by theory, indicates the desirability of leaving one end rough as an aid to even boiling. Bubbles tend to form on these sharp edges. A very useful ebullator may be made by drawing out a narrow tube to a near capillary size and sealing about 2 to 3 mm. of it to the end of a thin rod. When it is placed in a liquid and the liquid is heated, just below the boiling point a fine stream of bubbles appears to issue from the capillary and continues to issue throughout the boiling. This is a great aid in the prevention of bumping. The ebullator should be withdrawn while the liquid is still boiling. If it is allowed to cool in the liquid, the vapor in the capillary condenses and it becomes filled with liquid. Thus before the ebullator can be useful again, the liquid must be removed. Practically, a new one must be made.

Separatory Funnels. These are of three distinct shapes, a pear shape or conical, a tubular and a globe type (Figs. 9, 10, and 11). The pear shape is preferable when a small amount of lower layer is to be drawn off; otherwise there is little choice. Many standard methods specify one shape when another can be used without introduction of error. In general a short broad stem is preferable to a long thin one because the short stem usually drains promptly and dripping of the liquid during handling is avoided. There is seldom occasion to use sizes other than 250 and 500 ml.

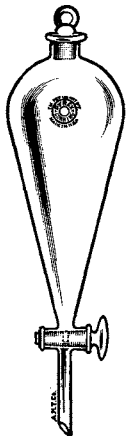

FIG. 9.—Pear-shaped separatory funnel.

FIG. 10.—Tubular separatory funnel.

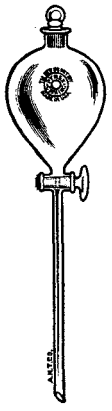

FIG. 11.—Globe-type separatory funnel.

Wash Bottles. The wash bottle is an essential unit in analytical chemistry. Figure 12 shows four types. The rubber tubing connecting the jet to the inner tube adds great flexibility in washing out beakers. The inner tube is bent at the end so as to enable the last portion of liquid to be blown out. Though a wicker collar is a great convenience for use with hot-water wash bottles, strong tightly wound cord is a satisfactory alternative. For solvents, such as ether, that attack rubber, an all-glass wash bottle is indicated. A number of fancy wash bottles with little gadgets attached are available. Polyethylene squeeze bottles are very useful for unheated solutions. In a busy laboratory, at least one wash bottle is kept on a hot plate to supply hot water. It is also a great convenience to have a small wash bottle containing alcohol and another containing ether, each labeled to save time in identification.

Jones Reductor. This unit permits reduction of a solution by retaining it in a column of amalgamated zinc for the required time. The item is shown in Fig. 13. To fill the unit, place a few glass

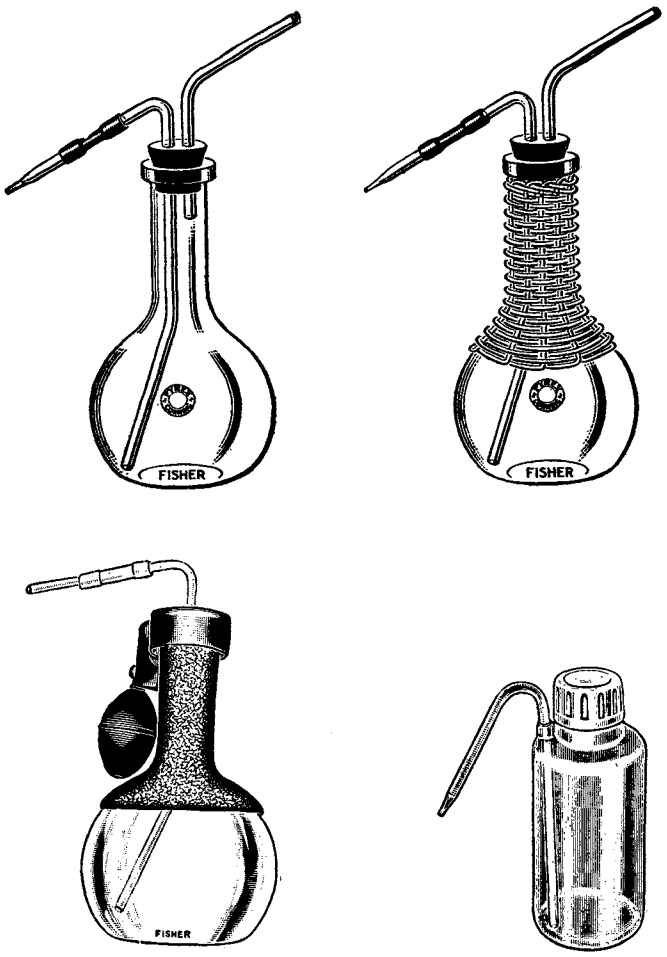

FIG. 12.—Wash bottles. That at the lower right is polyethylene.

beads in the bottom of a tube having a stopcock at its lower end. The tube should be about 30 cm. long and have an inside diameter of about 18 mm. On the beads place a layer of glass wool and then a thin layer of asbestos prepared for use in Gooch crucibles. Amal-

gamate 20- to 30-mesh zinc by first cleaning it with hydrochloric acid and adding mercuric chloride to the mixture with stirring until the zinc is well covered with mercury. This is evidenced by no hydrogen being evolved. While capable in this condition of reducing iron, titanium, or molybdenum as effectively as if it were not amalgamated, the zinc will scarcely be acted upon by hydrochloric or sulfuric acids. Fill the tube with this amalgamated zinc, and on the top place a pad of glass wool. Attach the tube to a filter flask so as to apply suction. Thoroughly wash with water before use. The titration of the reduced solution will usually be carried out in this suction flask.

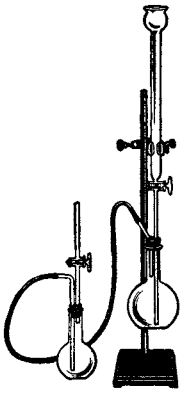

FIG. 13.—Jones reductor.

Policeman. The policeman is a necessary tool to loosen precipitates from the bottom and sides of beakers and flasks. It is of particular use in the case of gelatinous precipitates such as silica and aluminum hydroxide. In its elementary form a policeman is a short, tight-fitting piece of rubber tubing on a stirring rod. This form, as compared with the commercial policeman, has the disadvantage that additional crevices are provided and may lead to loss of material; it also contacts the surface with less efficiency. Figure 14 shows four commercial types

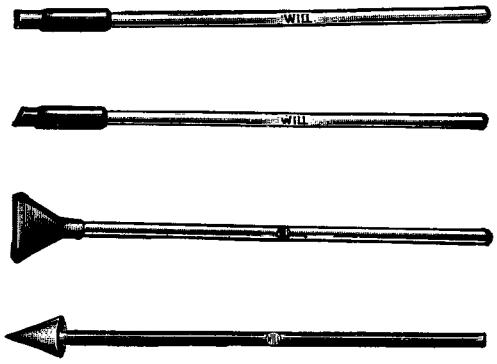

FIG. 14.—Policemen.

of which the long, flat-ended type is the most generally useful. When used, make sure that any precipitate adhering to the policeman is washed off with a strong stream of water from a wash bottle.

Chemicals. Pure chemicals are another tool of the chemist. If possible, use reagents from the original containers in which the

chemicals are received, thus avoiding possible contamination or error in transfer to service bottles. Avoid service bottles with cork stoppers. The majority of manufacturers of analytical-grade chemicals now supply them in 1-pound bottles with plastic screw-on tops or ground-glass stoppers. In a few instances, purchase in larger containers and transfer to service bottles is justified by the saving in cost. But always bear in mind that a single error may be dangerous, will surely be expensive in terms of wasted time, and may be expensive in many other ways.

After use, immediately replace the cap on a bottle to minimize contamination, dehydration, or absorption of moisture. And replace the bottle in its place on the shelf to save time not only for others who may want to use it but also for yourself the next time you want it. Alphabetical arrangement on stock shelves is usual and logical.

For analytical work chemically pure (C.P.) grades of chemicals are generally used. Some are sold as conforming to the published standards of the American Chemical Society. C.P. is a standard that is set by the individual manufacturer. Tolerances as to impurities are shown on the bottle labels and must be consulted and taken into account when traces are to be determined. In such cases it may be necessary to run blank determinations on even the most highly purified chemicals.

Some familiarity with other grades of chemicals is desirable. U.S.P. grades are as specified in the United States Pharmacopoeia and N.F. as in the National Formulary. They are either equivalent to C.P. or lower in grade, too often the latter. The cost of such grades of chemicals is often less than for C.P. They should be used with caution and only when it is known that results will not be vitiated by their use.

Technical-grade chemicals usually cost appreciably less than the purer chemicals. They are normally not suitable in analytical procedures. However they often are used as an adjunct to analytical procedures. In a desiccator, technical calcium chloride is used; for freezing baths, technical sodium chloride is suitable; technical grades of solvents such as chloroform, ether, and carbon tetrachloride are satisfactory, provided that they leave no residue.

Every chemical necessitates precautions depending on the material. No analytical chemical should come in contact with the hands. And above all bear in mind that pure is a relative term. Depending on the grade, a specified impurity may be expressible

in the first decimal place of percentage or in the third or the fifth. It may not be detectable. But all analytical reagents contain some impurities. Chemically pure is a convenient expression that should mean best quality but cannot mean exactly what it says.

Alcohol. This is one specific chemical that merits individual consideration. Although educational institutions obtain it without tax at under one dollar a gallon, all others must pay the tax, which is approximately $20 per gallon of 95% alcohol. A United States permit for purchase and storage is required; in addition many states also require a permit. Practically all analytical procedures can be as satisfactorily conducted with specially denatured alcohol 3A, usually called SD3A, which is tax-free. This contains 5 gallons of synthetic methanol to 100 gallons of ethyl alcohol. Its purchase and storage requires another type of permit, but a bond is not required unless purchases exceed 100 gallons per year.

This is not the place to go into great detail. Each chemical laboratory must maintain familiarity with Federal and state laws, submit at least one report a month, and comply with a host of regulations. There is no way of evading the burden. Completely denatured grades of alcohol can be purchased without license or regulations but are not suitable for analytical purposes.

Distilled Water. Although one is inclined to think of water as just water, it is either a tool or a chemical according to the particular use and in part according to the point of view. Ample supplies of good quality water and of distilled water are essential to any laboratory. If the local water supply is high in dissolved solids, over 300 parts per million (ppm.), cleaned glassware should be rinsed with distilled water before it is set aside to dry. Very few laboratories are small enough to be able to supply their full requirements for distilled water by purchase. Units for producing distilled water in the laboratory are available of as low capacity as ½ gallon per hour. The difference in original cost is small enough so that a still of ample capacity with some margin for expansion should be provided. This is particularly true because the best quality of distillate is obtained by a considerable degree of incompleteness of condensation.

There is no definite choice between the three trade types of still, Barnstead (Fig. 15), Precision (Fig. 16), and Stokes (Fig. 17). All are available in designs heated by gas, electricity, or steam. The latter is suitable only for a laboratory in an industrial plant that has steam the year round.

Many laboratories use *deionized water*, which for most analytical purposes is as good as distilled water. Laboratory scale apparatus for producing this is available, including disposable units in which the ion-exchange resin is not regenerated.

Gas Burners. Various types of burners derived from the Bunsen burner are standard equipment in every laboratory. In the most elementary form, gas is regulated at the stopcock and air at the burner. Such designs find little use in the analytical laboratory. The next step in development provides both gas and air adjustment

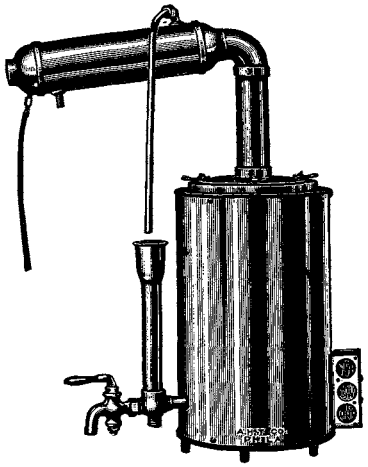

FIG. 15.—Barnstead still.

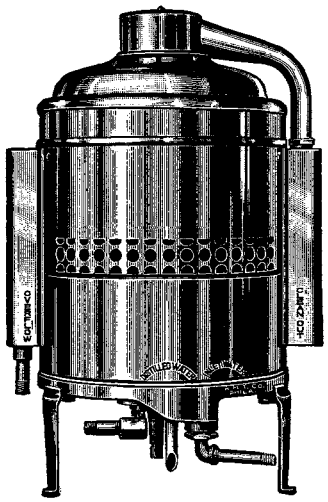

FIG. 16.—Precision still.

on the burner. These burners, which are assigned different names by different manufacturers, are about as simple a design as is useful to the analyst (Fig. 18). They usually do not have a grid over the top. When intense local heat is desired, the Meker burner is used (Fig. 19). This has a fine-mesh grid over the top, and in effect each opening in the grid acts as a small burner. Proper regulation of the relative gas and air supply is necessary with all these types to avoid flashing back, the burning of gas inside the tube. The Meker burner is often incorrectly called a blast burner. It fulfills most of the functions formerly performed by a blast burner. Correctly the latter is a special design of burner fed with both air and gas under pressure, thus getting more concentrated combustion and higher temperature (Fig. 20). For the maximum temperature, such as for working Pyrex or hard potash glass, oxygen under pressure is supplied. In the true blast burner, control of the form of the flame is

also provided, making possible, for example, a thin hot flame for use in some types of glassworking. Although minor glasswork is done in the laboratory, major work is usually supplied by professionals, because it is more accurate and, everything considered, more economical.

A useful form of burner not in such common use is the luminous flame, or Argand burner (Fig. 21). This type is most useful when low-temperature ashing is required. With careful use, complete ashing may be done efficiently with an Argand burner far below red heat. More evenly distributed heat is

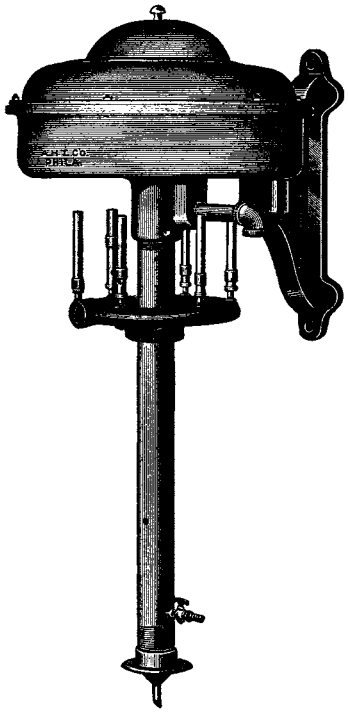

FIG. 17.—Stokes still.

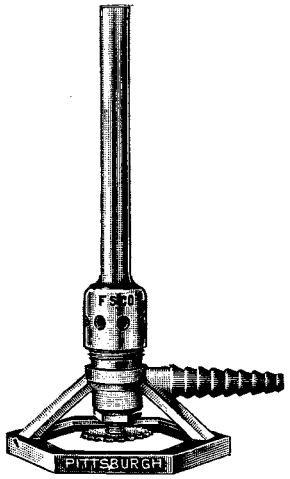

FIG. 18.—Bunsen burner with gas and air control.

obtained, and combustions of many samples may be performed without fusion, which might otherwise cause trouble.

In all these comments, it is assumed that usual city gas supplies are available such as carbureted water gas, coke-oven gas, or natural gas diluted with inert gas. These all have about the same heating value, around 500 B.t.u. per cubic foot. In many regions undiluted natural gas or gas from pentane in steel bottles is used. Adjustment to provide more air relative to the gas is required. Special burners for such gases are available, but the usual types are adjustable to fit their use.

When work is being concentrated in a limited area of workbench, the number of gas outlets is often less than the number of burners required. This is easily provided for by connecting two or more burners to a single outlet with a T or Y in the tubing. They are then individually controlled by the gas control on the burner. Safety dictates that all be turned off at the gas cock when the last person leaves at night and that each burner on a line be inspected when the cock is open, to avoid escaping gas.

Triangles. In elementary chemistry, triangles in many cases consist of iron wire with unglazed porcelain protectors on the three sides. For continuous use these are uneconomical. Also the

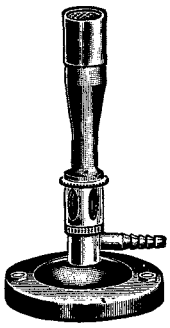

FIG. 19.—Meker burner.

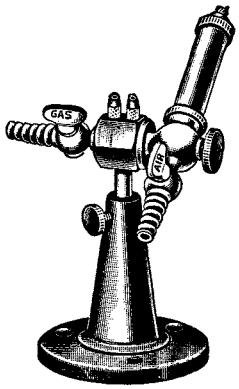

FIG. 20.—Blast burner.

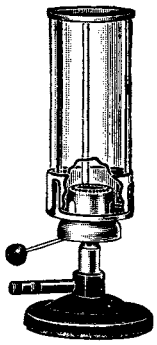

FIG. 21.—Argand burner.

porous porcelain absorbs anything spilled on it and thereafter will leave a mark and often a weighable deposit on porcelain or platinumware. For utility, triangles are preferably made of Nichrome wire. Very resistant to oxidization, this wire is comparatively thin, but strong, and allows the gas flame to reach practically every part of the crucible or dish.

Wire Gauzes. These similarly are often of iron, with or without a round asbestos central area. But wire gauzes, like triangles, are preferably made of Nichrome wire. A cost of six to eight times that of plain iron gauze is offset by a life exceeding that ratio many times. When distribution of heat is required, a piece of asbestos paper on such a gauze is both inexpensive and effective.

Filter Paper. The filter paper is a necessary adjunct in filtration. Both time and expense are economized by selection of the grade best suited to the purpose. The best known are the What-

man grades, of English manufacture, which will be used for illustration. Three general broad qualities are purchasable: qualitative, semiquantitative, and quantitative grades.

The *qualitative* grade is hydrochloric acid–washed and is used for work when the ash weight is unimportant. No. 1 is for filtering precipitates of average fineness. No. 2 is thicker and therefore not so rapid-filtering. Both are recommended for all-round qualitative use. No. 3 is thicker still and is used for very fine precipitates and for Büchner funnels. It is fairly resistant to alkalies. No. 4 is soft, open, and rapid-filtering and is recommended for gelatinous precipitates. No. 5 is tough, hard, and of close texture and may be used with a vacuum with little fear of breakage. All these grades may be used when the precipitate is to be saved but redissolved, when it is to be titrated, but not when it is to be ignited and weighed. Folded filters, which fit a funnel in a series of ridges, permit more rapid filtration but are relatively expensive. They are rarely applied in quantitative work because of the difficulty of washing the precipitate on them.

The *semiquantitative* grade is also hydrochloric acid–washed and is useful for quantitative work when the lowest weight of ash is not important. No. 30 is retentive and rapid-filtering and is used in routine determinations. No. 31 is rapid-filtering and is recommended for gelatinous precipitates. No. 32 is retentive, of close texture, and may be used for the filtration of fine precipitates. The ash of an 11-cm. circle in each case weighs just over 0.0003 gram.

Quantitative filter paper is of practically pure cellulose fiber. The pulp has been washed with hydrochloric and hydrofluoric acid until it is substantially free from silica and other inorganic sources of ash. Such papers are always properly stored in the box in which they come, which carries on the back a statement of the ash content per unit. They should be used in exact quantitative work when the ash weight is important and should usually give a weight of ash in the fourth decimal place, frequently in the fifth. Correction for the ash introduced by the paper is therefore rarely applied, the cases being those of unavoidable ignition of amounts of precipitate of the order of less than 10 mg.

Whatman No. 40 is probably the most used general paper. It will retain well-digested barium sulfate precipitates. For gelatinous precipitates such as iron and aluminum hydroxide or silicic acid, a more open texture is provided in No. 41. It must be handled carefully tc avoid breaking and is entirely unsuited to fine precipi-

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