CUSTOMS LABORATORY GUIDE

(September 2002)

WORLD CUSTOMS ORGANIZATION

THE CUSTOMS LABORATORY GUIDE

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1. Introduction

1.1. General

The Customs Laboratory Guide is primarily meant to be a practical handbook for the establishment or improvement of Customs laboratories in developing countries. The Guide includes the "best practices" covering a variety of laboratory operations. It is recognized that it would not be possible or necessary for any laboratory, to implement all of these provisions. Thus, the Guide can only present ideas and recommendations to the reader who is expected to apply these to his or her own laboratory facility. According to the need of his or her Administration to the extend possible and appropriate.

A large part of the successful planning of a Customs laboratory depends on how well the planner understands the needs of the laboratory. There is no fixed formula for designing a Customs laboratory.

Customs laboratories differ from other laboratories principally in the fact that they are called upon to analyse many different kinds of goods for Customs tariff, trade statistical, drug enforcement and other purposes.

Customs laboratories must therefore be able to establish an efficient system whereby samples of goods for analysis are channelled to the laboratory, prompt and accurate analyses of such samples are performed, and the results of the analyses are expeditiously conveyed to the Customs officer concerned for appropriate action.

The Guide's model layouts seek to provide the information necessary to plan all aspects of the design of a Customs laboratory. The various organizational, design and operational considerations are discussed in detail from the viewpoint of the establishment of a standard Customs laboratory.

Taking into account the different needs of countries and the resources available for Customs laboratories, model layouts of basic and advanced Customs laboratories are also dealt with in relation to the model layout for the standard laboratory.

It is, of course, impossible to write a Customs laboratory guide that would be suitable for all circumstances or cover all eventualities. It is hoped that the information set forth in the Guide will help Customs administrations to participate actively in the design or improvement of their own facilities.

The mark of a good laboratory design is that efficiency, safety and economy complement each other. In essence, it is necessary to create an environment that is conducive to an administration's way of operation, without compromising safety and efficiency, or adversely affecting performance.

Finally, the user of this Guide should always keep in mind that the information and principles presented are advisory and represent recommendations on how a Customs laboratory could be organized, arranged, etc., not how it must be.

1.2. Basic, standard and advanced Customs laboratories

It is not possible to define precisely what is meant by basic, standard and advanced Customs laboratories; all Customs laboratories differ to some extent from country to country in general terms. In the Customs Laboratory Guide, basic, standard or advanced Customs laboratories are assumed to have the following characteristics respectively :

A basic Customs laboratory is a non-instrumental laboratory, or a laboratory which has only a few basic instruments, with a few staff to perform only specific analyses required by the country for the classification of goods in the Harmonized System in a cost-effective manner.

From the practical standpoint, it is not always desirable to establish a standard-sized Customs laboratory. For example, a country where the total number of goods traded is low, may wish, for financial reasons, to establish a small-sized Customs laboratory for the analysis of key samples, specific samples of goods subject to rapid clearance, or suspicious samples collected at Customs offices, rather than to set up a standard-sized Customs laboratory.

A standard Customs laboratory is a basic-instrumental laboratory with sufficient staff and equipment to perform most of the analyses required by that country at least for the classification of goods in the Harmonized System.

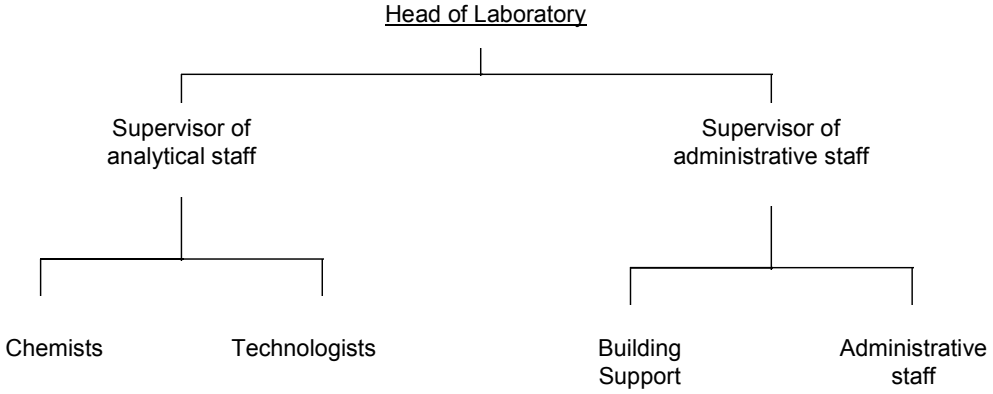
An "advanced Customs laboratory" is characterized by its competency to carry out a **diversity** of quantitative and qualitative analyses (especially the successful characterization of diverse, unknown commodities). This requires most, if not all, of the advanced instrumental technologies (GC, IRS, MS, HPLC, $^{13}C/^{1}H$ NMR, ICP, SEM, XRD, etc.) and a staff experienced in the interpretation of data relative to a wide range of industrial commodities.

The model layout of a Customs laboratory described below seeks to provide the information necessary to plan all aspects of the design of such a laboratory. The various organizational, design, administrative, safety, anti-pollution, sampling, operational and quality-assurance considerations are discussed in detail.

2. Organization

2.1. Organizational structure

Although there are obviously differences among countries as regards the type of Customs laboratory which may be needed and the resources available for this purpose, the organizational structure of a typical standard Customs laboratory should be as follows :



2.2. Head of the laboratory

The duties of the Head of the laboratory are numerous. For this reason it is not possible to do more than to draw attention to certain aspects of the position.

The main mission of the Customs laboratory is to analyse a large number of samples of all kinds of goods and to provide any necessary technical advice, as required for Customs tariff, trade statistical, drug enforcement and other reasons, as quickly as possible and with great accuracy. Consequently it is desirable for the Head of the laboratory to be a graduate chemist with experience and training in Customs procedures and particularly in the analysis of goods for tariff classification and drug enforcement purposes.

The laboratory Head is responsible for the efficient and effective performance of the Customs laboratory through the recruitment, hiring, training, development and motivation of subordinates, the development of resource and work plans, the establishment of responsibilities, priorities and objectives and the monitoring of work performance.

The Head of the laboratory is also responsible for the preparation and the administration of the laboratory budget. In this connection he must be able to bring the needs of the laboratory to the attention of higher authorities for appropriate action to allow as high a level of technical efficiency and expertise as possible to be maintained and developed.

The laboratory Head may have to give evidence in court, provide guidance to staff on court procedures, or otherwise provide information used in court. He should, consequently, have some understanding of court procedures.

Usually the duties of the Head of the laboratory, in his or her absence, are handled by a senior chemist or the head of the analytical staff.

2.3. Analytical staff

The basic function of the analytical staff is to analyse the samples received by the laboratory and to issue a report therein providing the requested information. Analytical staff may offer advice required for tariff or statistical classification, drug enforcement or other requirements which are within the competence of the laboratory.

Depending on the size of the analytical staff, it may be necessary to divide the group into two or more units. It is common to subdivide on the basis of principal types of commodities, i.e. textiles, foods, polymers, etc. The division of the analytical staff into commodity specific groups allows for specialization, which improves the overall capabilities of the laboratory.

Whether, and to what extent, the laboratory undertakes the role of performing work other than purely analytical work will be a matter of organizational policy. The policy will depend on a number of factors including the sophistication of the laboratory and the availability of alternative facilities. The analytical staff may also be required to appear in court as fact or expert witnesses.

The analytical staff of a standard laboratory should consist of highly qualified chemists, preferably university graduates, and trained technicians. The chemists should have extensive experience and training in the analysis of goods for purposes of tariff classification and drug enforcement, and a fairly good knowledge of Customs procedures. This includes a thorough knowledge of the Harmonized System which is necessary to ascertain which analyses should be undertaken.

It is not economical and not even wise from the point of view of efficiency of the laboratory to involve only graduate chemists in analytical work. Specially trained laboratory technicians who have received a two to three year practical training course in laboratory analysis can be particularly useful in carrying out a number of routine or even highly complex analyses under the supervision of a chemist. As is the case with graduate chemists, laboratory technicians also need specialised on-the-job training in tariff classification, drug enforcement and other Customs matters in addition to technical training to remain current.

There is no fixed or accepted ratio for the number of laboratory technicians needed in a laboratory per chemist but a team of one chemist and three or four laboratory technicians have been demonstrated in practice to be appropriate where the work is less repetitive and methods frequently have to be developed or modified, and where data interpretation (such as in the qualitative identification of unknown or poorly defined products) is common, then a higher ratio of chemists.

2.4. Supervisory staff

In laboratories where the number of analysts exceeds six to eight, a supervisor is needed. In a laboratory of this size a supervisor cannot be expected to do analytical work; his or her time is usually taken up with the management of the analytical staff. In smaller laboratories, it is desirable for the supervisor to spend a portion of his or her time on a variety of non-routine analyses to retain familiarity with instruments and to keep abreast of state-of-the art techniques.

The duties of a supervisor are numerous. He or she assists the Head of the laboratory in over-all management, work planning, receiving and assigning samples for analysis, work review, ensuring the availability of the necessary supplies and equipment, etc. The supervisor should also recommend training for the analytical staff.

2.5. Administrative staff

The administrative staff include all administrative assistance such as secretarial, typing and filing, record-keeping (logging and tracing samples, financial record-keeping), maintenance of laboratory stores, management assistance, cleaner service and library services (if the laboratory library is of a size to need a librarian). Administrative staff are very important to the effective operation of a laboratory.

In addition to clerical support staff, the laboratory may also require building support staff (described below).

It is uneconomical to have inadequate administrative staff because then their work must be performed by chemists or other staff who should be involved only in analytical work. Support staff should usually number 15 to 20% of the number of total analytical staff.

2.6. Building support staff

Part of the work performed in any laboratory, regardless of the size, is not directly connected with the actual analytical or administrative duties. Some of this support work includes maintenance of the heating, cooling, water, and electrical systems; removal of garbage; upkeep to the exterior of the building and land or parking around the building; cleaning of the laboratory; heavy lifting and moving; etc. These duties can be performed by support staff without special educational qualifications. Personnel doing these duties should be made aware of laboratory hazards and instructed in safety procedures immediately on starting the job and on a regular basis they should be informed of all the risks associated with chemicals or instruments they may come in contact with.

3. Design

3.1. General considerations

When a new laboratory is being built, close co-operation is required between the architect who designs it and guides its construction and the chemists who are familiar with the technical needs of the laboratory. It is important to employ architects that are familiar with laboratory operations and design in corporation with chemists of the laboratory. Standard construction techniques are not always the best for laboratories. The users must be involved starting from the design stages and there must be open lines of communication between the architects, engineers, contractors, tradesmen and users throughout the design and construction process.

However, it is not often that chemists have the chance to take part in the planning of a completely new laboratory building. More often one has to make do with an old and, sometimes, inadequate facility. In both cases, whether designing a new laboratory or renovating an old one, it is important to take into account the possibility of the need of future expansion or improvement of the laboratory, however unlikely that may seem at the time. The installation of new scientific instruments and equipment will require additional space.

Wherever possible, the placement of controls, the location of safety equipment and room layouts should be standardized.

3.2. Basic structure

In planning a Customs laboratory, whether for an old facility or a new one, the rooms for the different laboratory activities must be located and designed in a manner well adapted to the purpose. For this reason it is necessary to know the goods which are to be analysed in the laboratory and the volume and frequency of submissions to the laboratory of the principal types of commodities. With the help of this information it is possible to calculate the number of analyses to be carried out, the level of analytical examinations involved and to determine the specific instruments and equipment needed.

After the number and quality of analyses required has been evaluated it is possible to determine the time needed for analyses. Based on this information the size of the staff and surface area of the laboratory premises can be estimated.

It is normal to allow about 10 m^2 of laboratory space and 3 m of bench surface per analyst (minimum value from which to start). In addition to actual laboratory premises, space is also needed for offices (including a meeting room), storage and major pieces of analytical equipment. These generally make up roughly 40% of the total area of the laboratory.

In planning a Customs laboratory, for flexibility in meeting changing needs, it is best to allow some areas of the laboratory building to remain "open-plan" to the extent possible, including the area used as offices. However, there are certain basic concepts for the layout of the laboratory which should be taken into consideration when planning a Customs laboratory (e.g., segregation of the office area from the laboratory area is highly desirable).

These are :

(a) Separation of the instrumental analysis area and the chemical analysis area in order to avoid contamination of expensive scientific instruments by various chemical reagents and to ensure their long operating life.

(b) Ensuring that the analysis area for flammable or hazardous goods is separated from the office area, but close to it. If the Customs laboratory is large enough, the floors for offices (including meeting room and library) and for the operational laboratory zone should be separated (e.g., the ground floor for administrative offices and the first floor for the operational laboratory zone). The area must be constructed with the necessary ventilation, precautions against electrical (including static electricity) sparks or other ignition sources.

(c) Allocation of a quiet but convenient location for a weighing or balance room. A separate balance room is recommended to avoid unnecessary draughts and vibrations caused by ventilation systems, laboratory workers, machines, etc.

(d) A special room is desirable for an infra-red spectrophotometer (with air-conditioning in areas of high temperature and humidity, and with a filter system for dust).

(e) A separate outside storage room is recommended for flammable or hazardous chemical reagents and laboratory waste materials in a large laboratory.

(f) A separate outside storage room is recommended for high-pressure gases (e.g., hydrogen and nitrogen).

(g) The location of fume cupboards and exhaust fans should be carefully considered to ensure effective ventilation throughout the laboratory. The air intake for the heating and cooling system should be located upwind of the fume cupboard exhausts and a good distance from them. The fume cupboards should also have high stacks on them to allow the fumes to dissipate thoroughly.

(h) The sample preparation area should be separated from laboratory rooms where analysts are working on trace analyses or using sensitive instruments.

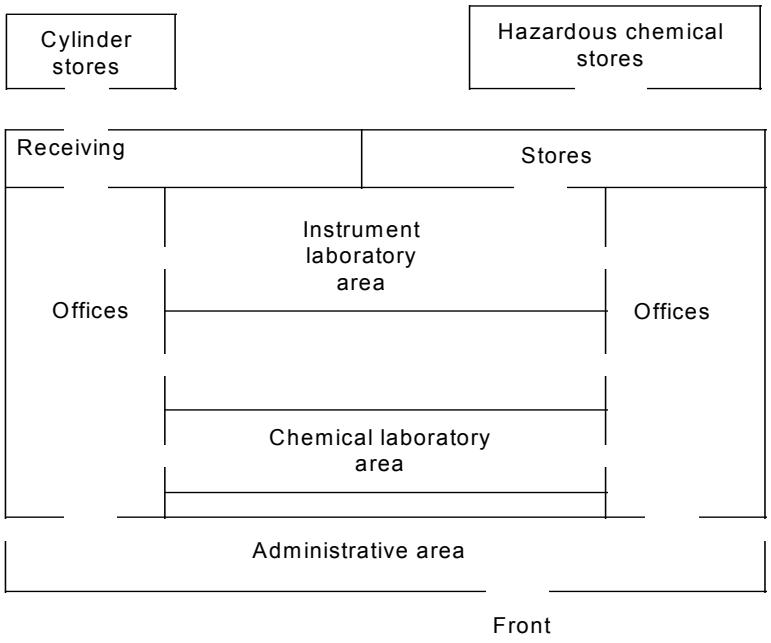


Fig. 1. Basic structure of a Customs laboratory

Taking these basic concepts into account a Customs laboratory layout that offers flexibility, safety and good work flow is shown in Fig 1.

The administration section is located at the front of the building. This office area has up to four enclosed offices and a conference room with open-area work areas for the rest of the computer and clerical staff. If the staff is large enough, a cafeteria or coffee room should be provided in this area.

Separated from the administration area by a fire wall are the chemical and physical instrumental laboratories with closed and open offices for supervisors, chemists and technologists running along the outer walls and separated from the laboratories. At the rear of the building, there is a sample and equipment receiving area, and stores areas for new and old samples, chemicals and supplies. Separated from the main building should be two fireproof and locked storage facilities. One should contain flammable solvents and hazardous chemicals used for analyses, as well as old hazardous samples which are awaiting disposal. The other building should contain compressed gases, both full and empty cylinders.

3.3. Space utilization

The work space in a laboratory must be planned and designed to accommodate laboratory activities and to ensure proper work flow. The most common equipment in laboratory facilities consists of benches. An important consideration in this connection is that the benches be designed and equipped for the intended purpose and that their placement be carefully considered. The placement of furnishings can significantly affect operational efficiency and safety.

Bench setups for chemical laboratory rooms and for analytical instrumentation (Figure 2) are quite different.

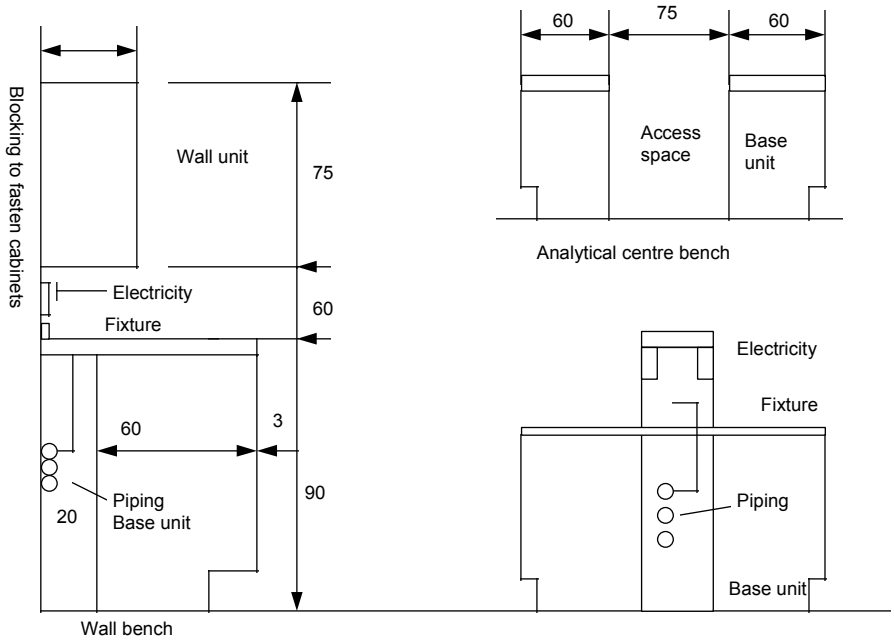


Fig. 2. Traditional casework system - wall supported. (Measures in centimetre)

3.3.1. A Room for chemical analysis

The benches in a room for chemical analysis can be placed in two different ways as shown in Figure 3 and Figure 4 (often referred to as island benches and peninsular benches, respectively).

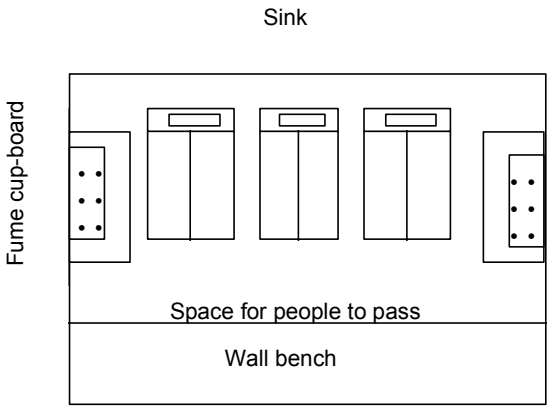
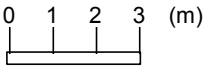


Fig. 3.



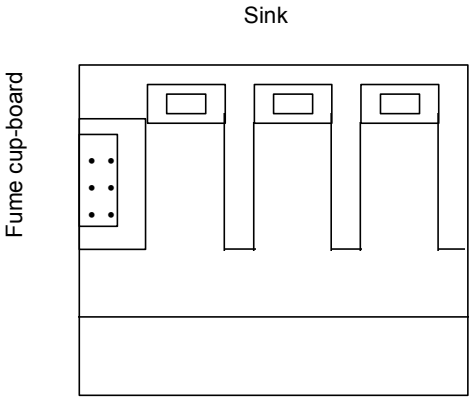


Fig. 4.

Both of these placings have their advantages. The standard width used for a single bench is 60 cm, which is about the same as the width of the space required by a standing analyst.

By comparing peninsular benches and island benches it can readily be seen that the maximum bench area is obtained with the peninsular bench placing, which makes this placing preferable if floor space is limited. With the island bench placing, the working surface is somewhat more accessible and allows more flexibility for the placement of windows and doors.

One of the other benefits of the peninsular bench placing is that the analyst working between the benches has access, in effect, to three working surfaces at once. This is, of course, very convenient if there are several different analyses in progress at the same time. It should be stressed that the space between benches should be enough to allow two analysts to work back to back (at least 1.5 m).

It could also be argued that the peninsular bench placing is safer since the traffic patterns in the laboratory tend to be more restricted and predictable. In the final analysis the choice of bench placing depends very much on the size and shape of the room available.

For health and safety reasons, analysts should not have desks in rooms where chemical work is performed. However, the desks or offices should be close to analytical work stations.

Another important aspect to remember is that furniture and equipment in laboratory work areas should be arranged so that an exit may be reached easily from any point. Two means of egress are required at a minimum for every laboratory room and they should be remote from each other and so arranged as to minimise any possibility that both may be blocked by fire or other emergency condition.

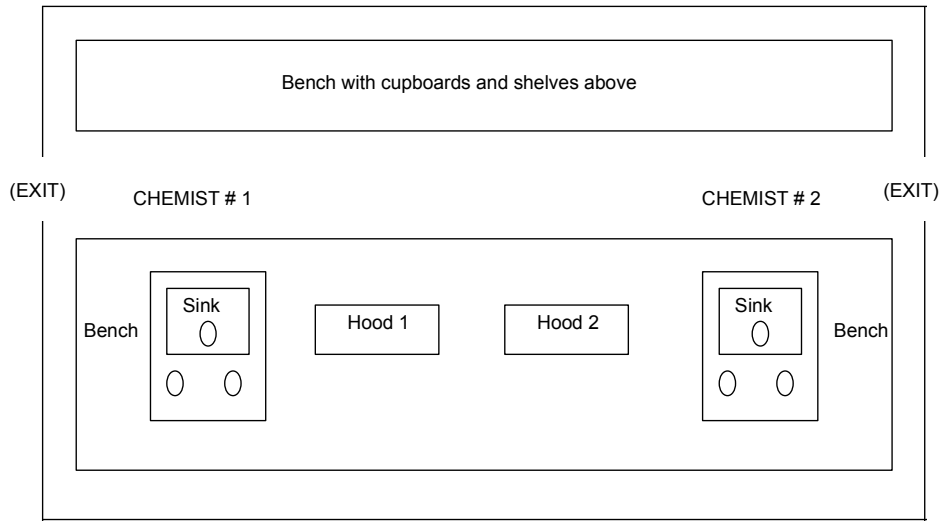


Fig. 5. Two-chemist/technologist chemical laboratory module

For a laboratory with more than 10 or 12 chemists/technologists, consideration should be given to using two person modules as shown in Fig. 5. These modules are safer than large open laboratory areas since chemical operations are separated from each other. Further consideration should be given to arranging the modules around a laboratory core which houses the more commonly used pieces of equipment (e.g. infra-red, gas chromatography), and arranging the analysts' offices around the outside of the laboratory modules. This arrangement keeps each chemist's/technologist's work area separate, allowing the analyst to work safely, while integrating his or her analytical work area with their office area and the more commonly used instruments in the laboratory core.

3.3.2. A Room for physical instrumental analysis

Before planning an instrument laboratory room (i.e., a room for physical analysis) it is necessary to determine the specific instruments (as well as their accessories) which are likely to be installed in it. The room should reflect the specifications of the instruments.

The physical specifications and service requirements of the instruments should be obtained from the instrument suppliers. On the basis of this information the size of the room needed can be determined and the physical layout of benches drawn, taking into consideration not only the area required for instruments but also the surrounding area that must be left open for operations and maintenance.

This room should also be temperature controlled and provided with ventilation ducts or hoods to remove gases formed during operation of instruments, such as gas chromatographs, etc. The room should be reasonably vibration free and be equipped with uninterruptible electrical power. As far as possible, this room should be open to allow for reconfiguration as new pieces of equipment are obtained.

In a larger laboratory, one room for all the large analytical instruments will not suffice. Instruments such as gas chromatographs (GC), gas chromatograph-mass spectrometers (GC-MS), nuclear magnetic resonance spectrometers (NMR), X-ray diffractometers (XRD), etc., will probably require their own room and specific technologist. These separate rooms are necessary for various reasons, e.g., health and safety considerations, special cooling requirements, low vibration areas, etc.

3.3.3. Balance room

The balance room should be designed to protect precision balances from corrosive atmosphere, dust and draughts. If possible, the room should have a filtered, low-velocity air supply and be vibration free. The balance table must be made of stone or concrete in order to reduce vibrations. While the balance room will not be occupied full time, it should be conveniently located.

Because of great improvements in balances in the last few years, it may not be necessary to have a separate balance room if the laboratory can afford some of these newer balances. These balances, because they are very shockproof and are small, fit well in the individual laboratories and save valuable analyst time.

The balance table for high precision balances (resolving to better than 0.001 g) should be of high mass construction, free standing and physically separate from surrounding benching, etc. (to prevent vibration transmission). Also, it should be designed to avoid the operator knocking or resting his/her feet on the balance table (low level rails), again to prevent upsetting the solving capability of the balance.

3.3.4. Storage facilities

3.3.4.1. General recommendations for chemical storage :

The main factors that should be considered in providing for the safe and efficient storage of hazardous chemicals are compatibility, optimum use of storage space, separate air supply and exhaust, safety, and convenience of storage and retrieval.

Storage racks and shelves should be designed to prevent breakage or leakage, which might cause damage or endanger those who enter or work in the storage area. Storing large and small containers on the same shelf can make retrieval difficult and breakage likely. For this reason shelves should be sized and spaced appropriately for storage of compatible materials by container size. Ventilation should be provided to prevent corrosion or dangerous concentrations of vapours.

National regulations may provide some important guidelines for the design of storage areas in order to minimize danger from fire, explosions and contamination of the environment. However, such regulations usually cannot address all of the safety and health aspects of chemical storage, particularly as new chemicals come into use and new hazards are discovered. Furthermore, national regulations are generally written for the storage of large containers and industrial quantities and, therefore, may not be appropriate for the storage of chemicals in small or break-resistant containers.

The type and size of containers to be stored will affect the need for special storage practices and safety procedures.

Ventilation is needed for chemicals and containers which may release dangerous quantities of vapours or gases which are flammable, corrosive, irritating or toxic.

Doors to chemical storage areas should be identified with the hazards of the materials stored in the area.

Another important safety consideration for storage areas is, of course, sufficient and explosion-proof lighting.

The laboratory designer should seek guidance from chemists with respect to the design of storage areas.

3.3.4.2. Location of storage facilities :

Storage facilities should be located where they will be safe, convenient and economical.

The storage facility should be separated from the rest of the laboratory and protected so that a spill or fire is not likely to spread beyond the storage area.

Within laboratory work areas, storage for working quantities should be provided under the fume cupboards in ventilated cabinets for volatile, flammable chemicals. Such storage should be limited to chemicals which are used frequently, quantities that are the minimum necessary and container sizes that are the minimum convenient.

Stockrooms or similar accessible supply areas should be provided for small amounts of frequently needed chemicals that are not stored in laboratory work areas. If these chemicals are flammable or reactive, or give rise to health concerns they should be placed in an isolated, independently ventilated room or preferably in an exterior fire-proof locked storage building.

3.3.4.3. Flammable liquid storage cabinets :

Special storage is commonly required for flammable liquids. Liquids which require such storage have flash point temperatures at or below 95 °C.

Depending on the quantities of such liquids within the laboratory they should be stored in either an approved storage room or in a storage cabinet. Laboratory facilities usually require a separate, fire-protected room used only for the storage of bulk quantities of flammable liquids.

Preferably, large stock supplies of flammable and hazardous liquids should be stored in a fire-proof locked building separated from the main laboratory area. If possible, no more than two or three 20-litre containers of each solvent should be on hand at any time.

Storage cabinets should be designed to insulate their contents so that in case of fire outside of the cabinet, the internal temperature will not exceed 165 °C within 10 minutes. There are commercially available double-walled storage cabinets which provide the minimum protection required.

There should be a mechanical system to provide effective ventilation and exhaust of hazardous and flammable vapours from chemical storage cabinets. The ventilation ducting of these cabinets should also be fire resistant.

3.4. Equipment and instruments

3.4.1. General considerations

When planning a Customs laboratory one should not underestimate the complexity of equipping the laboratory.

At the stage of establishing a new laboratory the requirements for equipment and instruments may seem large and complex since certain types of analysis may require several individual pieces of equipment and if even one is not available the analysis cannot be performed. On the other hand most of the instruments and equipment are common to different analyses so that once they are procured there comes a point at which productivity of the laboratory can rise sharply compared to investments in new instruments.

Plan the laboratory so that it is very flexible - design for the future !

The total operating expenses depend, of course, on the size of the laboratory and are related to many different factors. One of the important expenditures involves the maintenance, repair and replacement of equipment.

In the developing world, one of the major problems in carrying out effective laboratory operations is broken equipment. The degree of sophistication of equipment varies greatly from a straightforward pH meter to a complex instrument such as a spectrophotometer. It is therefore necessary that adequate provisions be made for obtaining professional repair services and replacement parts. It is false economy if analysts are being paid but cannot perform an important part of their work due to lack of maintenance.

Although Customs laboratories may require special equipment for specific tests under the Harmonized System, it should be noted that few Customs administrations (if any) possess all such instruments and apparatus, given that some are rarely used and are very expensive. Careful consideration should be given to cost and benefits when purchasing these special instruments and apparatus. It is recommended that laboratory facilities outside of Customs be found to carry out these special examinations.

Some of the basic instruments and equipment needed in a Customs laboratory are listed in Part I, Chapter 2, Sub-Chapter 1. Special instruments for specific examinations required for HS classification and additional equipment which may be needed are set out in Part I, Chapter 2, Sub-Chapters 2 and 3, respectively.

The needs of Customs laboratories vary from country to country, depending on differences in tariffs, the volume of and type of trade, etc. Therefore it is only possible to outline some of the instruments which may be useful for a standard Customs laboratory.

3.4.2. Fume cupboards

3.4.2.1. General considerations :

The major items of fixed equipment in a Customs laboratory are the fume cupboards. The use of solvents, and noxious chemicals in analysis require a greater use of fume cupboards than other types of laboratory work. The number of fume cupboards in the laboratory should be sufficient so that work is safe and all handling of volatile compounds can be done in the hoods.

A fume cupboard should be designed so that it ensures a high degree of personal safety. Remember that the fume cupboard is probably the most important piece of safety equipment available to the analyst. The hood should be large enough to accommodate most common operations. Fume cupboards may be purchased prefabricated with utility outlets and are available in standard sizes ranging from 90 cm to 1.5 m in length with larger hoods available on a custom-made basis. However, today with energy costs rising and because the size of the hood is directly proportional to its energy consumption it is not wise to select a larger-than-necessary unit.

It is advisable to make the hood as flexible as possible by placing many utility outlets in it, although costs may limit this possibility. It is necessary, however, if purchasing or constructing the hood locally to specify the type, quantity, and placement of every required service fixture such as for water, gas, electricity and drainage.

The installation of exterior controls for the utility service fittings is highly recommended. This permits access to the services from outside the hood while the unit is in operation. It is also prudent to have the fixture outlets colour coded for easy identification of each fixture.

Lighting inside the hood chamber is very important. Normally, a vapour-proof fluorescent light fixture is used, which gives a pleasant light as well as adequate illumination.

The fume cupboard is usually opened by lifting the vertically-rising front glass window which is counterbalanced with weights. The window is normally constructed of 6 mm laminated glass. The fume cupboards with windows that move from left to right instead of up and down are not recommended. (see Figure 6).

The material for the construction of fume cupboards is most important, especially if a fume cupboard must withstand acid fumes, in general, and perchloric acid fumes, in particular. The hood lining is normally made from 6-7 mm thick material that is both heat- and chemical- resistant.

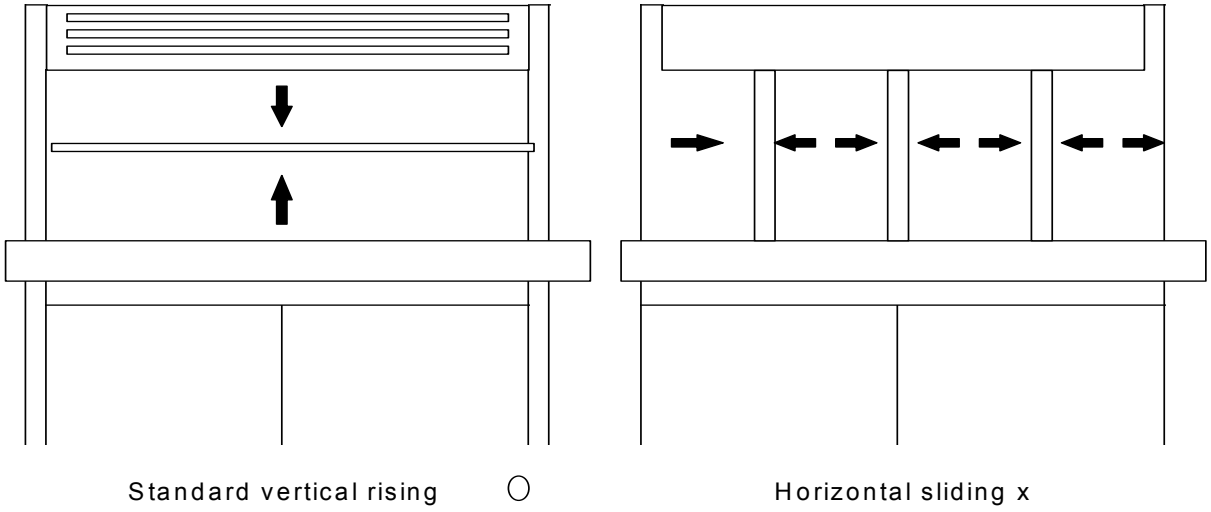


Fig. 6. Fume cupboard

Linings made of plastics or stainless steel are used. Figure 7 shows the schematic structure of a fume cupboard.

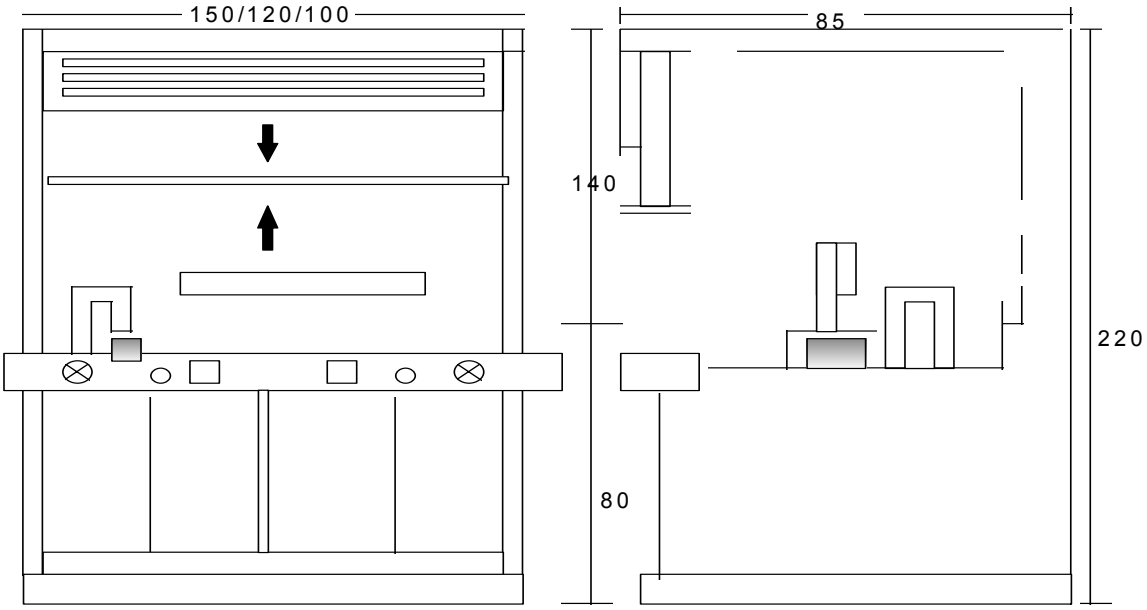


Fig. 7. Schematic structure of fume cupboard (Measures in centimetre)

Some recommendations for the materials and equipment for a fume cupboard are set out below :

Sliding window

- The sliding window should be made of 6 mm laminated glass and counterbalanced with weights

Worktop

- Acid-resistant stainless steel, ceramic material, epoxide resin or polypropylene

Lighting

- Fluorescent tube or any other explosion-proof source
- Switches on front panel

Taps

- 1 Cold-water tap
- 1 Gas tap
- 1 Compressed-air tap

All taps should be front-operated.

3.4.2.2. Ventilation of fume cupboards :

Generally the fume cupboard is so constructed that air flows in through the front opening and goes up along the rear wall of the hood's interior. The hood is normally designed with adjustable slots at the top of the chamber as well as at the countertop level. By opening or closing these slots the direction of the exhaust can be adjusted and controlled.

The total volume of air needed for a fume cupboard depends on the size of the front opening and the velocity of air circulating through the hood. If, for instance, the airflow through the open face of a fume cupboard is 0.5 m/sec (which is the minimum requirement), the width of the fume cupboard is 180 cm and the height of the face opening is 60 cm, the volume of air needed is 540 cm^3/sec or 1944 m^3/h.

The size of the fan motor of the exhaust duct must be sufficient to meet the requirements of the hood's ventilation and to overcome the static pressure generated by the ventilation duct (static pressure is the loss in efficiency caused by friction as the air passes through the exhaust system).

Static pressure varies directly with the length of the ventilation duct as well as with the number and type of bends. Incorrect determination of static pressure can lead to selection of incorrect exhaust fans. It is therefore recommended to use the assistance of a qualified engineer in designing the ventilation system.

The exhaust fan should be located as far away from the hood as possible, preferably on the roof of the laboratory, away and downwind from fresh air intakes for the building. Exhaust gas should be released outside through a filter, if possible. In this way negative pressure can be maintained throughout the duct system preventing duct leakage which may contaminate the laboratory.

The fume cupboard should be placed in an area in which cross-currents of air are minimal. One of the major factors in reducing hood efficiency is locating a hood near an active doorway. The movement of the door can draw vapours out of the chamber. Walking past a hood can also have a negative influence.

The chamber of the hood should be kept as clear and open as possible to allow air to pass through. A fume cupboard used for chemical work should not be used to store chemicals.

If any apparatus that does not have legs is housed in the hood it should be placed on blocks in order to allow air to flow beneath it. It is also good work practice to place the source of contamination at least 15 cm inside the hood as this will dramatically increase the margin of safety.

The ventilation system of the fume cupboard should be kept operating all the time, even over night. If a fume cupboard is not in use and the glass window is closed, ventilation can be reduced to half in order to save energy. Two-speed fans will also significantly reduce energy costs. Constant ventilation prevents dust from collecting in a fume cupboard and moisture from condensation in ventilation ducts. There should be a signal light in the fume cupboard which indicates when the ventilation is on (a small piece of thin paper attached to lower edge of the glass window also works as an indicator by vibrating if the ventilation is working).

It is well recognized that fume cupboards represent probably the single greatest safety feature in any laboratory. Therefore it is advisable for all laboratories using fume cupboards to have an instrument used to measure the air velocity so that periodic checks may be performed to ensure that the hood is operating up to its design capacity.

If there is more than one fume cupboard in the laboratory the capacity of the ventilation system of the laboratory should be such that incoming air volume is sufficient for all hoods to be in full use at the same time. Each fume cupboard should have a separate exhaust fan, as there is danger of cross-contamination from a common ducting system.

3.4.3. Bench system

The bench system for a chemical laboratory consists of a work surface, storage cabinets and knee-space below and shelving or storage cabinets above. The system includes service fixtures, sinks, electrical devices and the associated piping, drain lines and electrical conduits.

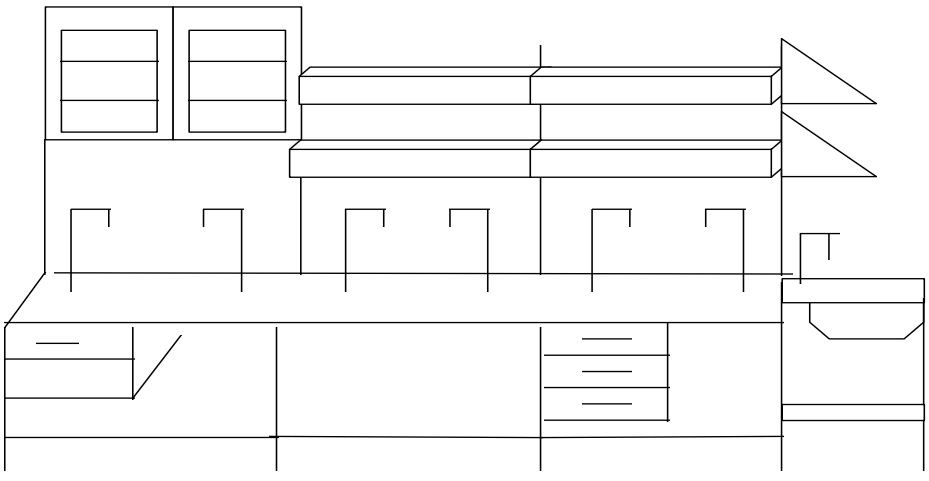
There are many choices available for the work surface. The primary reasons for selecting one surface over another are appearance and how well the finish resists the chemicals used without blistering, bleaching, staining or absorbing.

Epoxy top benches are recommended since they are highly resistant to the wide variety of chemicals that are used in a Customs laboratory. Plastic laminated countertops, which are much less expensive, are also widely used in areas where there is less exposure to solvents, strong acids, bases and heat.

For laboratories that require greater chemical or heat-resistant countertops, natural stone materials could be considered. Ceramic tile is sometimes used but its disadvantage lies in the many joints. In general, ceramic tile is not recommended.

Many laboratory furnishing manufactures offer a wide variety of laboratory furnishings of different standard sizes which allow adaptation to nearly any room size and shape. Bench modules are either free-standing or can be wall-mounted and are interchangeable allowing great flexibility.

Some examples of bench, cupboard and cabinet assemblies are reproduced in Figure 8.



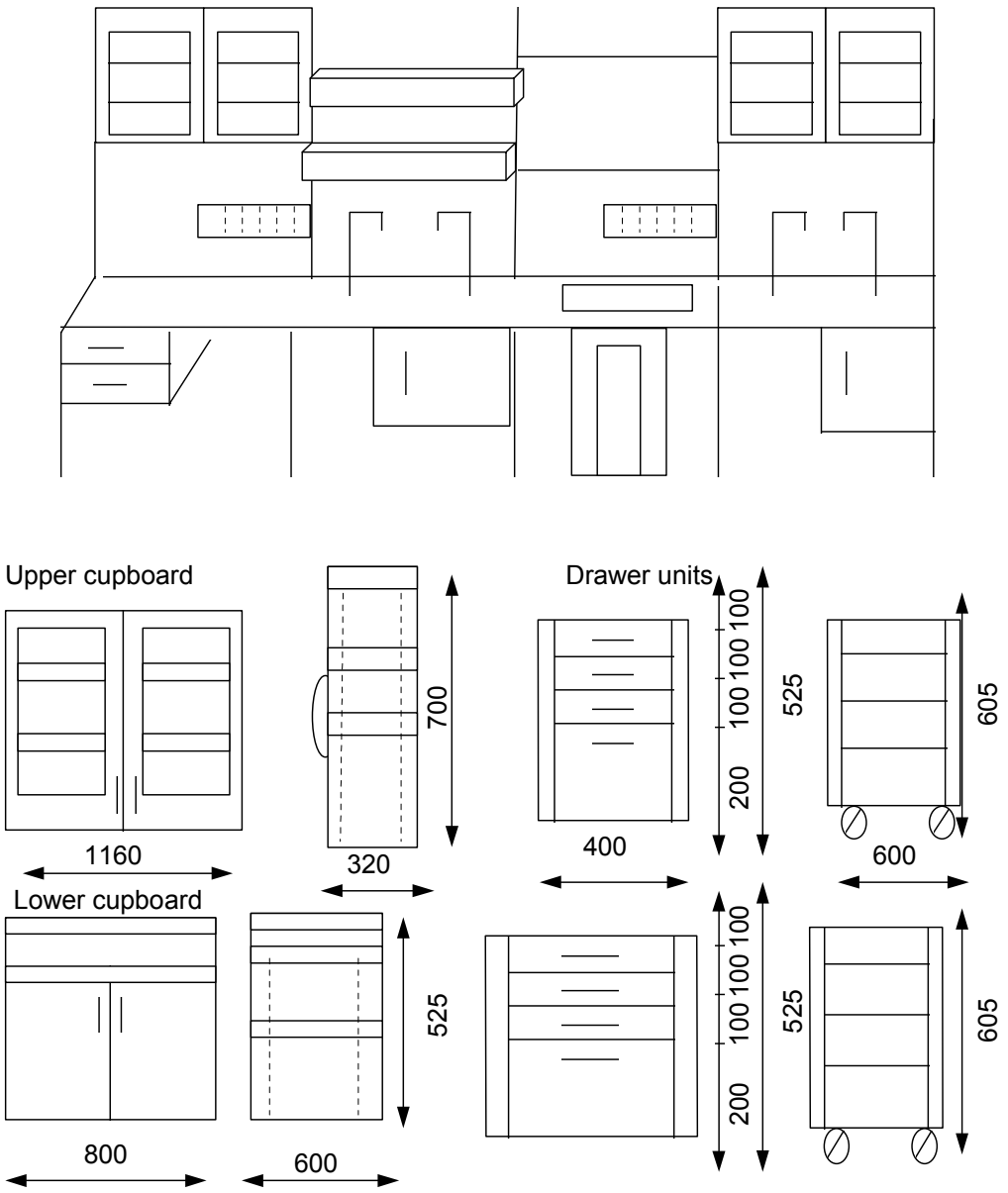


Fig. 8. Example of Bench, cupboard and cabinet assemblies

3.5. Ventilation and air-conditioning

The ventilation of a laboratory should be such that the whole volume of laboratory air is changed 6 to 12 times every hour. For instance, if the floor space of a laboratory is 60 m and the height is 2.5 m, the volume of air needed is 1800 m per hour. This amount of air is about the equivalent of air exhausted by one good-sized fume cupboard. The total volume of air that is required for a laboratory is dictated by the number and size of fume cupboards. This fact should be taken into consideration when designing the laboratory ventilation system.

The large quantities of air needed in a laboratory can best be introduced through perforated plate air outlets or diffusers which are specially designed for large air quantities. Air should not be introduced in the immediate vicinity of fume cupboards in order to avoid affecting the performance of these units.

Perforated ceiling panels provide a better air supply than ceiling diffusers in that system design criteria are simpler and easier to apply and the precise adjustment of fixtures is not required. Perforated ceiling panels should be sized so that panel velocity is less than hood-face velocity, preferably no more than two-thirds of hood-face velocity.

Natural ventilation, which may provide large quantities of air without filtering or air-conditioning is not generally suitable for laboratories.

In a standard laboratory air-conditioning is essential. An air-conditioning system is, of course, costly as it must be sufficiently powerful to provide a clean, filtered air supply which passes through fume cupboard exhaust fans as well as other outlets. Air-conditioning provides a stable temperature environment for sensitive and sophisticated analytical instruments. Most volumetric glassware is calibrated at 20 °C and must be recalibrated if used at significantly different temperatures.

The volume of incoming and exhausted air should be so regulated that the air pressure inside of the laboratory is somewhat lower than air pressure outside. This prevents odours from spreading from laboratory rooms to other parts of the facility.

Instruments such as gas chromatographs and atomic absorption spectrophotometers as well as vacuum pumps used to evaporate solvents must be provided with ventilation ducts or hoods to remove gases formed during operation, otherwise toxic levels of poisonous gases may accumulate in the laboratory atmosphere.

A simple and effective way to ventilate instruments is to install an exhaust duct on the wall about 40 cm above the instrument table and to connect a flexible duct (10 cm in diameter) to it just above the instrument. The flexible exhaust duct (also called an "elephant trunk") can then be bent in such a way that the opening comes close to the point of the instrument where gases are released.

3.6. Utilities

3.6.1. Electricity

3.6.1.1. General considerations

Many models of laboratory instrumentation available on the market today incorporate micro-circuitry type components. While greatly enhancing the capability, reliability and speed of the instrument, this type of hardware has also created problems. Such instruments are becoming increasingly vulnerable to disruption and abuse caused in great part by difficulties experienced with the electrical system of the laboratory. This makes the reliability of a laboratory's electrical service and distribution especially important. The electrical system in laboratory facilities should be designed to provide adequate, flexible, uniform and reliable power.

Periodic reconfiguration of laboratories may change overall power needs. Consequently, designers should estimate power needs not only for short-term projections, but also with long-term needs in mind.

Because of these short- and long-term needs, electrical distribution should provide the laboratory with as much flexibility as possible. The most important considerations are quantity of power, frequency and voltage levels. Generally,

most laboratory equipment is designed for the normal power provided by the utility company.

Many instruments draw relatively few amperes. Since the demand does not appear to be that high, there is a tendency for operators to attempt to connect more than one major instrument to single circuit. This practice should be avoided wherever multiple circuits are available in order to avoid overloading the circuits.

The outlets to which instruments are to be connected can be mounted either on the countertop, on the wall or on a shelf. However, it will be necessary to anticipate the actual number of outlets required at each instrument's location. The number of outlets in a laboratory must be sufficient since this adds considerably to the efficiency of analytical operations and to their safety.

3.6.1.2. Voltage stability

Ideally voltage should be supplied at its rated value and remain relatively constant. Unfortunately, fluctuations occur from time to time, generated either by the source or by local conditions within the laboratory.

Voltage fluctuations also can be created by electrical equipment located in or near the laboratory. Elevators, air-conditioners, furnaces and hot plates are capable of producing this effect under certain circumstances.

Many instruments are equipped with filter systems to minimize or avoid this problem, although they may not be completely effective. If there is a reason to believe that equipment malfunction might be caused by voltage fluctuation the problem can be analysed by means of special instrumentation.

The use of AC power conditioners as energy supply to instruments guarantees output voltages within +/- 5 % and noise filtering in lines as well.

Special attention should be paid to the location of computers in the laboratory. High-voltage lines under the floor or in ceiling spaces can cause monitor screens to flicker. Therefore, major electrical trunk lines should be kept to the perimeter of the building.

3.6.1.3. Grounding

Normally, laboratory instruments are tied into the common ground of the laboratory facility. However, it may be necessary, depending on specific circumstances, to create a separate or "isolated ground". If a common ground is utilized, the line should be inspected carefully by a qualified electrician to ensure that all ground connections are intact.

3.6.2. Utility gases

3.6.2.1. General considerations

The demand for high-pressure gases for instruments, e.g., hydrogen and nitrogen, has increased dramatically in laboratories in recent years.

The primary means of supplying instruments with these gases is via cylinders. This method of delivery has the advantage of creating an isolated source of supply in which purity and composition of the gas can be controlled.

As safe as these cylinders are, there is always a potential for an accident. Therefore, the storage of these cylinders in a laboratory is not recommended. An alternate approach is to create an area adjacent to the instrumentation laboratory as a "cylinder room".

The cylinder room must be wide enough to hold at least two cylinders of each type of gas used in the laboratory. The cylinders should be placed side by side instead of one behind the other, eliminating the need for unnecessarily moving a cylinder. Each cylinder should be strapped to a wall for safety. The depth of the room need only be enough to store the largest diameter cylinder.

Construction of a cylinder room should be based on the degree of hazard of the specific gases used. In most cases brick construction would be suitable. The doors should be constructed so that the entire length of the room can be exposed at one time.

Two similar gas cylinders can be connected together to allow for the use of an automatic low-pressure switch-over mechanism. In this way it is ensured that there will be no interruption or reduction of gas flow. When switch-over does take place, it should activate a flashing light in the laboratory thus alerting the operator of the need to deliver a replacement cylinder.

Gases are normally transported from the cylinder room to the instruments through flexible copper tubing. This tubing should be mounted directly to the wall in a fully exposed fashion as it enters the instrument room.

3.6.2.2. Gas system

One or more gas points are needed per bench. If natural gas is available from a utility company the gas system should be designed in accordance with the utility company's requirements. Where no street mains are available, gas must be provided via a liquefied petroleum "bottled" installation.

Wherever possible, gas mains and risers should run exposed rather than concealed in shafts or hung ceilings. This prevents the possible accumulation of gas in these closed spaces due to leakage from the gas piping system, which may explode if it occurs in the proper concentration and is subject to an igniting spark. Gas supply lines should be designed to supply small areas (e.g. 12 outlets) and each area should be controlled by a well marked shut-off valve. Handles for these valves must never be removed.

3.6.3. Compressed air

Compressed air is very useful in the laboratory. If obtained from a compressor machine, the optimum pressure should be between 5 and 6 kg/cm^2, and the air should be purified by filtering and drying before being used in laboratory instruments.

3.6.4. Water

Benches must be provided with several cold-water taps to allow for rinsing, condensers, etc. Hot-water taps can be restricted to those sinks where apparatus are washed.

Electric wires, water, sewer and gas piping require space and easy access for repair reasons. Because of this it is advisable for them to be mounted under the 20 - 40 cm wide separate centre-bench section between free-standing laboratory benches or, where benches are placed against a wall, between the wall and the bench (Figure 9).

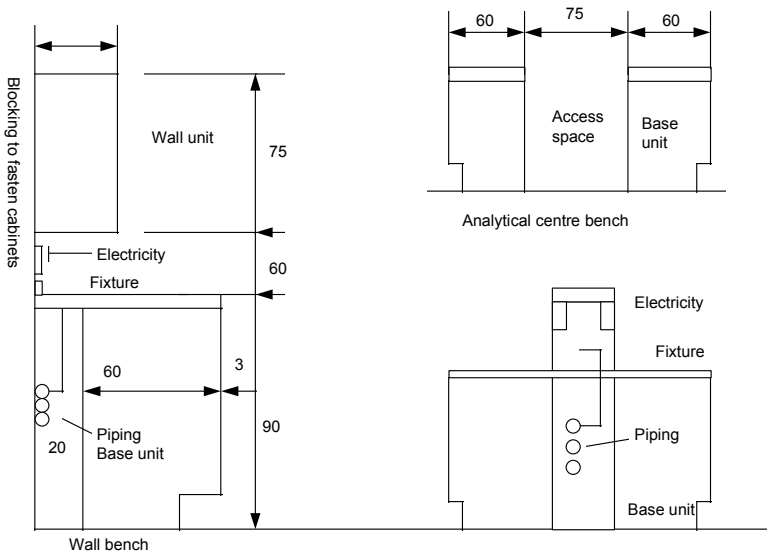


Fig. 2. Traditional casework system - wall supported. (Measures in centimetre)

3.7. Anti-pollution guidelines

Guidelines for waste disposal vary from country to country. Many countries do not allow any hazardous wastes to be disposed of through the sewer system. In these cases, chemical wastes and hazardous samples which have been analysed should be labelled with the proper safety labels (i.e. numbered, recorded and safely stored in an explosion-proof, well-ventilated, locked, exterior building) prior to disposal by a qualified company.

Design of an in-house waste disposal system depends on the types and quantities of laboratory waste water. Waste treatment concepts can range from simple dilution to full-scale on-site treatment facilities. Wastes may also be regulated by state or local authorities. Review of this matter with those authorities at an early stage in the design of the laboratory is advisable.

The following principals should be considered when designing process waste and vent systems :

(a) Waste and vent systems should be separate piping systems from storm water and sanitary waste drainage systems.

(b) Wastes from all fixtures and equipment where acids have been used should be neutralized before discharge into the sanitary drainage system.

(c) The system should be sized to anticipate future expansion and also provide for flexibility of the laboratory areas.

(d) Central acid-neutralizing sumps should be provided for areas with numerous sinks and located in an accessible place for easy maintenance.

(e) All waste piping connecting laboratory sinks and cup drains (where acids might be used) to acid-neutralizing sumps and all vent piping for these fixtures should be of acid-resistant materials.

Neutralization is accomplished by chemical reaction or dilution. Wastes are normally discharged to sumps filled with limestone or marble chips or, for large laboratories, to a treatment tank using an injected sodium hydroxide (NaOH) solution to raise the pH and sulphuric acid (H_2SO_4) to lower the pH (Figures 10 and 11).

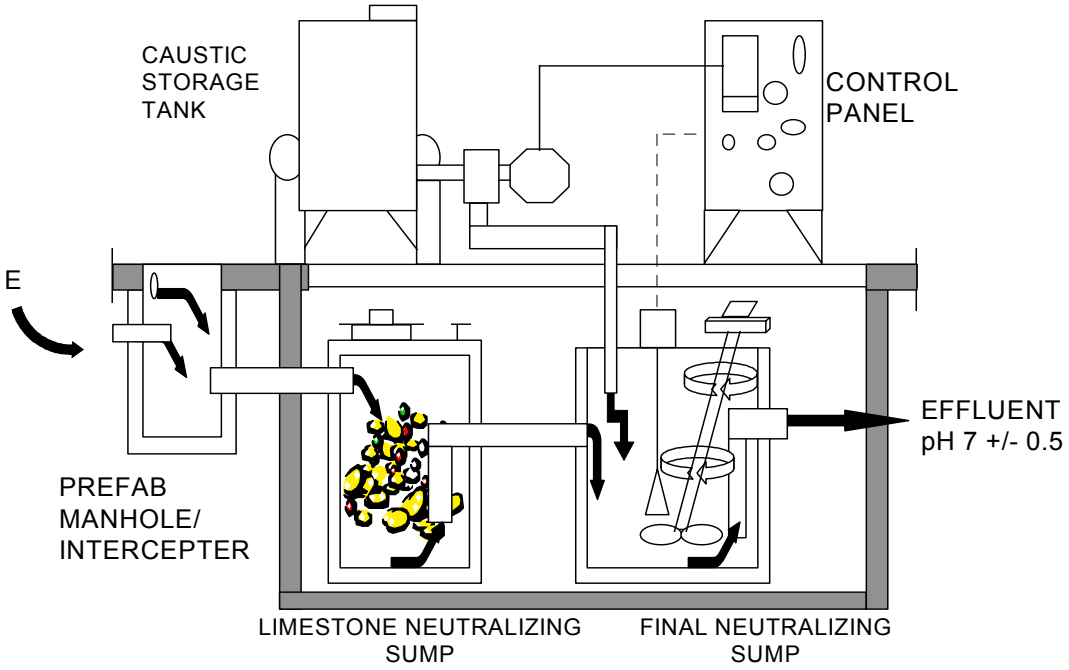


Fig. 10. Laboratory neutralization sump

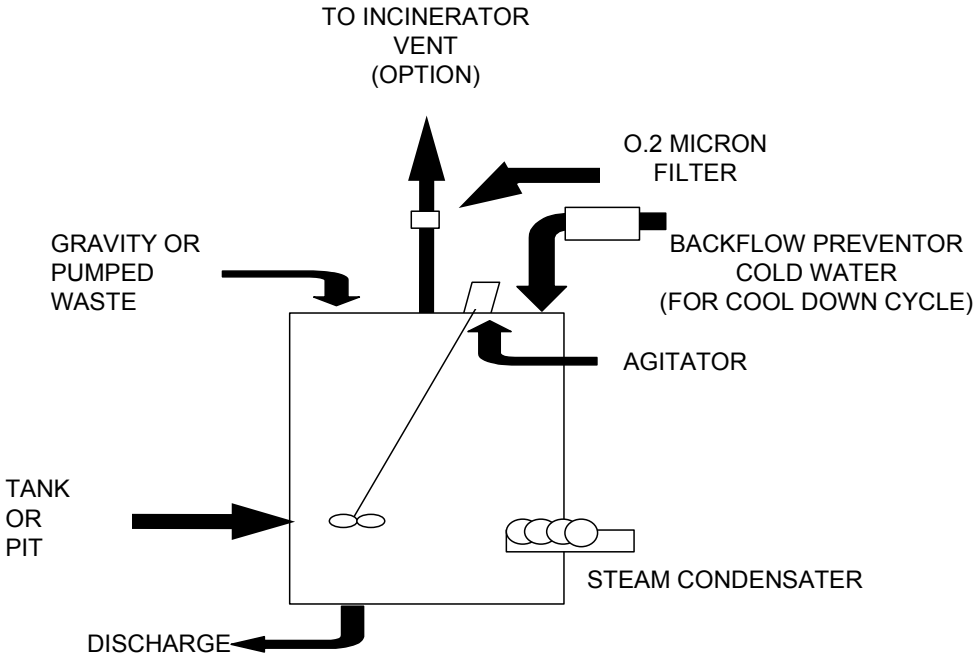


Fig. 11. Waste treatment tank schematic

In waste piping connecting laboratory sinks to neutralizing sumps high density polyethylene or polypropylene materials can be used, since these show good resistance to most organic and inorganic chemicals. Pipes and fittings should have screwed joints.

The method of sizing the laboratory waste and vent piping is the same as that used for sanitary waste and vent systems.

Acid-neutralizing sumps are usually constructed of chemical stoneware, acid-proof, tile-lined steel tanks or high-density polyethylene. The sizing of tanks is usually based on the number of sinks and cup drains used during peak periods.

Acid-neutralizing sumps should be sized on the following basis :

Diameter	x	Height	Users or sinks *
30 cm		30 cm	1
45 cm		30 cm	2
45 cm		60 cm	3 - 6
60 cm		90 cm	7 - 20
75 cm		142 cm	21 - 50
90 cm		175 cm	51 - 100

* Users : the number of people likely to use the sinks and/or cup drains during a peak-hour period.

Given the high cost of such facilities, it may be more practical in small laboratories to store waste water separately according to its content and to send it to specialists for treatment or to treat it in-house from time to time. Waste water containing the following substances should not be drained :

- Cyanates and cyanate complexes
- Chromium (IV) ions
- Mercury and its compounds
- Other heavy metal elements.

Dilute water solutions containing organic solvents which are easily decomposed by micro-organisms can be drained. Halogenated solvents or solvents containing halogen compounds should be stored and sent to specialists for treatment. Burning out of these substances is not recommended since toxic substances, e.g., dioxin, would be produced. It is possible to burn out other organic solvent wastes.

4. Administration

4.1. Budget

The budgetary system should be flexible. Contingency funds are important for the running of a laboratory. Budgets should be arranged so that funds are readily available for urgent supplies and repairs and the other day-to-day needs of the laboratory.

The Laboratory Head must have adequate control of the budget and operate it under clearly defined rules. These should be sufficiently flexible.

Budgets are typically planned for one year, but major equipment purchases should be planned for a three-to-five year period.

4.2. Purchasing

In case of a new laboratory the selection and specification of laboratory equipment together with the planning of space requirements (including flexibility for future expansion) should always precede, or at least go hand in hand with building design. Before selecting any equipment, a careful study should be made to determine :

(a) Which of the many possible suppliers and manufacturers of equipment are capable of providing installation and maintenance services.

(b) What experience potential suppliers have in dealing with the special conditions existing in less industrialized countries.

Equipment specifications must be prepared very carefully and clearly in order to avoid purchasing of incomplete or unsuitable apparatus. Information is needed, for instance, with regard to essential accessories not included with the basic instrument and optional accessories (with explanations for their particular application), essential operating supplies and recommended spare parts. Precise specifications are essential in obtaining reliable and comparable offers. For example, instruments equipped with a library data system (e.g., infra-red spectra, mass spectra, etc.) are recommended, since the library search system has recently been developed to identify unknown contents in samples.

Among the demands imposed on equipment suppliers, it is very important to include the standards certificate issued by international organizations, and to start to request standard written procedures and preventive maintenance, as well as manuals in the user's language.

Large laboratory supply companies offer a more comprehensive range of supplies, including a full range of instruments and auxiliary equipment, than is offered by individual manufacturers. Service facilities for the installation and maintenance of equipment can also be co-ordinated and provided much more easily by a large organization.

Complex apparatus requires special training for the operators. Engineers performing the installation usually provide some basic training. However, a more far-reaching programme of training may be necessary. In such cases, special training programmes should be arranged in advance of the purchase, preferably before the

equipment is delivered. The exact training requested should be specified in the contract to purchase the equipment.

Normally, the service representative will provide basic operator training while the instrument is being installed. A more specialized follow-up training is usually provided by the vender 6 to 12 months after installation.

4.3. Supplies management

Supplies routinely used by a laboratory include solvents, reagents, chemicals, glassware and other analytical materials. These supplies must be regularly replaced as used. Particular attention should be paid to supplies that are consumed and are very important to the daily operation of the laboratory.

There should be an accurate accounting system to record receipt, use and future need of supplies in order to be aware of the stock situation and the need to purchase replacements. Replacement orders should be made and the replacements received before the total depletion of items occurs. Some supplies may take months to obtain. The procurement process must take this time into consideration.

Stock records can be maintained in a variety of ways but a card system is perhaps the most versatile. The Supplies Record Card should contain such data as :

- Name of product
- Date purchased
- Where purchased
- Amount
- Expiration date (if any)
- Special storage requirements (if any)
- Amount dispensed
- Minimum reorder quantity (when the material reaches this level, it should be reordered as soon as possible).

Proper supplies management prevents the occurrence of the situation where analyses have to be stopped because a critical material is suddenly used up.

4.4. Equipment maintenance

It is important that all items of equipment be properly and promptly maintained and repaired when needed. The degree of sophistication of equipment varies from a straightforward pH meter to a complex instrument such as a spectrophotometer.

It may be difficult and expensive in developing countries to repair any instrument, sophisticated or otherwise. Therefore the routine maintenance of instruments is important since this should delay the day when outright repairs are necessary.

Under these circumstances it is recommended that laboratory analysts or technicians be given training in repair and maintenance techniques. Training can best be provided by instrument suppliers.

If adequate service is not readily available locally, serious consideration should be given to the purchase of a service contract. The usual service contract involves checking the instruments at specified intervals and performing necessary maintenance. Service contracts are expensive and should be considered when the expense can be

justified. Services of expert from the suppliers could also be made up of. A basic in-house maintenance capability should be developed if possible.

Recording the history of an instrument's maintenance and repair is important. It provides a summary of the instrument's operation over a given period and provides a justification for the replacement of old and worn out instruments with new ones when an instrument costs more to maintain than it is worth.

4.5. Training

The following staff training may be required :

(a) Training on the Harmonized System. Chemists must have knowledge of the Harmonized System in order to determine, carry out or direct the necessary analyses.

(b) Training on certain basic techniques (e.g., titrimetry, gravimetry, TLC, IR, GC) especially for assistant analysts.

(c) Training on analytical methods for specific products (e.g., petroleum products, foodstuffs, organic chemicals, textiles, metals).

(d) Training on safety and emergency measures.

(e) Training in the use of computers.

(f) Training on sampling procedures for field Customs officers

(g) Languages

(h) Automatic data processing

(i) Customs legislation

(j) Quality assurance

(k) Statistics.

4.6 Information systems

It is recommended to acquire an "information system" with an adequate database designed to store and integrate the technical data of analysed samples and other data generated in other areas within the analytical process, and to allow users from the administrative, support and analytical staff to consult and recover information in accordance with their specific needs.

Final reports of analyses performed can be keyed directly into the system, allowing information recovery by multiple means : name and synonyms, physical-chemical data, properties, HS classification, uses of the product and data from samples analysed in the past.

Administrative and support staff can obtain the necessary information to prepare statistical reports on the work performed and to control and supervise operations and the laboratory.

5. Safety and anti-pollution measures

5.1. The safety programme

The common responsibility requires that the best possible working conditions and safest possible environment is provided in which to carry out analytical procedures.

The Head of the laboratory and supervisors have overall responsibility to ensure safe working conditions in the laboratory, including the responsibility :

(a) To ensure that workers know and follow chemical hygiene rules.

(b) To ensure that protective equipment is available, in working order and used according to established guidelines.

(c) To provide appropriate training.

(d) To know the current legal requirements concerning substances.

(e) To determine the required levels of protective apparel and equipment.

(f) To establish a general security plan for all staff in the Customs laboratory (e.g., scheduling of fire safety drills, scheduling of medical examinations, etc.).

(g) To introduce certain techniques for identifying risk (e.g., use of radiological exposure monitoring badges, pest control, etc.).

One senior analyst should be appointed the "Safety Officer" and given the responsibility to monitor safety procedures, practices and equipment on a routine basis. In a large laboratory, the Safety Officer may be assisted by a committee of two or three analysts.

The duties of the Safety Officer (and committee where appropriate) should be detailed in the laboratory Safety Programme. This programme should indicate safety requirements, hazards, equipment and emergency procedures. Items which should be included in a laboratory Safety Programme are discussed in the following paragraphs.

5.2. Basic safety rules

Each laboratory should develop its own set of General Safety Rules, and make sure that all members of staff are aware of them by supplying personal copies of the rules and by posting copies on notice-boards. The rules should be changed and developed in the light of experience. Some that should be included are :

(a) Become familiar with the location and use of emergency equipment (e.g., fire extinguishers, eyewash fountains, safety showers, first aid cabinets). Know where to go in case of fire.

(b) Before beginning a sample analysis, review possible hazards connected with the assignment and take necessary precautions to eliminate or counteract the hazards.

(c) Use, as appropriate, the safety equipment provided for protection (e.g., safety goggles to protect eyes, face shields, various types of gloves). Wear a laboratory coat routinely since its purpose is to serve as protective clothing.

(d) Bring all accidents and hazardous conditions to the attention of the supervisor immediately.

(e) Be extremely careful of loose clothing, neckties, scarves, dangling jewelry (such as necklaces) and long hair when using revolving or reciprocating equipment. Keep such items bound or confined so that they will not be entangled in the equipment.

(f) Turn off laboratory services (gas, water, etc.) at the service cock when not in use. Changes in pressure may suddenly dislodge tubing connected to an apparatus and lead to an accident or possible injury.

(g) Always use mechanical aids, such as safety bulbs or pipette fillers, when pipetting hazardous material. Never use the mouth.

(h) Use fume cupboards for any analytical operations involving significant amounts of solvents, or when noxious fumes will be generated.

(ij) Keep the work area neat and tidy, with all containers labelled with their contents.

(k) Any chemicals, whether toxic or not, which come in contact with the skin must be washed off immediately and completely.

(l) Keep fire escape routes and doors clear at all times. Do not block, even temporarily.

(m) No one should work alone in the laboratory, so that assistance is available in the event of an accident.

(n) The last person to leave the laboratory at the end of the working day must check that all equipment which should be turned off has been (this does not release each individual operator from his or her duty to turn off equipment no longer in use).

(o) Do not smoke in any of the laboratory areas.

(p) Do not eat or drink in any areas of the laboratories other than those designated for this purpose.

(q) Use biological safety cabinets for products that pose a biological hazard.

(r) Ensure that personnel exposed to radiation wear radiological exposure monitoring badges.

5.3. Fire safety

5.3.1. General considerations

A laboratory must be regarded as one of the more likely places in which a fire will occur. Therefore, a laboratory facility should be designed so that the building and its contents will be protected from fire and so that any fire that occurs can be extinguished, with no injury to personnel and with minimum loss to building and contents.

It is also prudent to have the local chief fire officer visit the building, be familiarized with the problems and advise the laboratory staff about local fire regulations and further safety measures that should be taken.

Fire and smoke detectors should be installed in the building and connected so that in the event of fire, an alarm is given at a fire station.

Staff must have clear instructions of the action to be taken in case of fire. Such instructions should be posted prominently in various parts of the building. Occasionally, there should be a fire drill to check that everyone knows what to do in case of a serious fire and to make sure that the building can be evacuated in an orderly and rapid manner. Fire evacuation must include switching off the electricity supply to the affected area, since short-circuits may result in a further fire hazard.

When new staff is introduced, they should be trained in safety procedures, be shown where fire extinguishers, blankets, hoses and buckets are kept and how the sprinkler system works, if there is one.

5.3.2. Fire extinguishers

Laboratory facilities should be provided with portable fire extinguishers that can be used by laboratory employees to put out small fires effectively. Fire extinguishers must provide adequate fire extinguishing capacity for litre bottles of flammable solvents (liquids).

Multi-purpose extinguishers are often recommended for areas where fires may involve different classes of materials. However, pressurized dry chemical extinguishers which are suitable for general use, should not be used around computers or other sensitive electronic equipment. It is nearly impossible to clean the dry chemical out of such instruments and the instruments could be irreparably damaged.

Many hand-held carbon dioxide fire extinguishers on the market have the required capacity and are often preferred because they are suitable for small solvent fires and leave no residue.

Fire extinguishers should be located near the doors of laboratory work areas, either just inside or just outside. This location is preferred because a person who seeks the extinguisher in case of fire will be heading toward the exit.

It is recommended that all laboratory buildings be provided with standpipes and 4 cm hose, for use by occupants to supplement fire extinguishers, and that the hoses be equipped with special nozzles.

5.3.3. Spill kits and respiratory protection

If spill control material is to be provided, it should be of a type and in a quantity adequate to provide safe control of potential spills. Storage of spill control material should be located in a corridor or other area away from those in which spills are likely to occur so that a spill does not prevent access to emergency equipment.

Respirators with the proper filters should be used when dealing with toxic fumes from analyses or spills. They should be available at one central point (i.e. spill cart) in the laboratory.

Electrical equipment may be the source of a fire due to faulty wiring, inadequate grounding, failure causing sparks or local overheating or switch gear sparks. All electrical equipment should carry a fuse which blows at an amperage slightly in excess of that required by the equipment. It is commonplace to find that equipment drawing only one or two amps have a 13 or 15 amp fuse which is unlikely to be of any use if anything goes wrong.

Flammable vapour near electrical equipment represents a fire hazard unless the instrument has been rendered "explosion proof" by sealing those sections where a spark could occur. Motors must be serviced regularly. Lack of attention may result in overheating and subsequent fire. Centrifuging of volatile flammable solvents is hazardous if the centrifuge motor is not explosion proof, especially if the tube breaks.

5.4. Emergency water

Readily available supplies of water are needed for emergency flushing of chemicals from the eyes and body in every work place in which irritating, corrosive or toxic chemicals are used. Emergency showers and eyewash stations should be standard features in every laboratory.

Time is of the essence in emergencies. The location of and distance to an emergency shower are important and there should be no door in the path of travel between the spill area and the shower. The first few seconds are most critical to preserve life and avoid major injury. The distance should therefore be no greater than 30 metres from any location in the laboratory to the nearest shower. That is the maximum distance. Shorter distances are advisable.

Eyewash stations are used to rinse the eyes and face in case of chemical spill. Close proximity is essential since an individual must be able to find the eyewash blinded and unaided within seconds of the spill. Locating such devices at a sink in the work area will take less floor area, provide an economical and convenient water supply and drainage and is easy to find in an emergency. Never hook up the eyewash to a hot water supply. If possible, provide water within the temperature range of 10 to 20 C.

5.5. Chemical hazards

Many chemicals are hazardous and should be handled carefully. Hazardous chemicals can be flammable, explosive, toxic, corrosive or poisonous and can be identified with the help of technical literature and manufacturers leaflets. It is desirable to check on the nature of chemicals and to take adequate precautions before handling chemicals in order to avoid accidents. The suppliers of chemicals should be asked for Material Safety Data Sheets (MSDS) every time (new) chemicals are ordered. Any laboratory should, of course, have the necessary first aid facilities.

Chemicals can be harmful (1) when coming into contact with the skin or eyes, (2) when inhaled or (3) when swallowed.

Contact of corrosive chemicals with the skin or eyes can cause irritation, discoloration of the skin and even burns. Serious effects can be avoided in most cases by rinsing immediately with running water. Treatment with dilute solutions of neutralizing chemicals is sometimes effective (e.g., the use of a diluted ammonia solution in the case of acid burns).

Contact of hazardous chemicals with the eyes is, of course, extremely dangerous and it is, therefore, highly advisable to wear goggles or spectacles while doing analytical work.

The inhalation of toxic or poisonous chemicals can result in the quick absorption of chemicals into the body. For example, hydrogen cyanide gas is immediately absorbed into the blood and is extremely toxic. Highly concentrated gases may lead to asphyxiation. Breathing deeply, coughing and artificial respiration can be effective countermeasures in some cases.

Swallowing of chemicals is usually the most dangerous of accidents. Toxic chemicals such as acids, alkalis and heavy metal ions directly dissolve or coagulate stomach proteins and are absorbed by the body. The most effective treatment is to make the victim vomit as soon as possible. Treatment with neutralizing chemicals is sometimes effective. For example, if acid is swallowed, sodium bicarbonate solution should be administered. In the case of heavy metal ions, the victim should be made to vomit.

It should also be noted that there are chemicals which are suspected or known carcinogens. These chemicals must always be handled with extreme care, using all available protective gear.

To the extent possible, hazardous chemicals should be stored away from the laboratory and away from other chemicals with which they violently react.

All reagent bottles, flasks or other containers must be properly labelled, even if the contents are considered harmless. Warning stickers can be used in addition to the container label to highlight hazards.

Before sending containers (beakers, etc.) containing hazardous material to be washed, they must be rinsed or otherwise treated by the analyst to remove the hazard. The analyst is the only one who knows of the hazard and how to eliminate it.

When emptying acids and cleaning solutions into drains, the sink should first be filled with water before pouring the acid or cleaning solution into the sink; only after this should the water be drained. The tap should be allowed to remain on full for a few additional minutes. Dilution should always be sufficient to reduce the acid concentration to a pH of between 5 and 7.

5.6. Physical hazards

There are many physical hazards in a laboratory. Most are avoidable by the use of common sense. It is advisable, however, to prepare some instructions for new employees and periodically to remind others. The following are some suggested instructions for glassware handling :

(a) Do not use broken or chipped glassware or return it to storage. Always use gloves in handling broken glass.

(b) Remove sharp or jagged edges from glassware before using it. Fire polish the edges on all glass tubing.

(c) Broken glass in sinks presents a definite hazard since glass may not be visible in the presence of water. When broken in the sink, remove it promptly. Furthermore, consider the possibility of the presence of broken glass when reaching into the sink for any purpose.

(d) In handling beakers, support them by grasping the sides, never the top. Support large beakers (one litre or more) from the bottom when in use.

(e) When heating liquids in glass by means of a gas flame, protect the glass from direct contact with the flame by use of wire gauze or centred wire gauze.

(f) When placing liquids in bottles which have a positive closure, reserve more than 5 % of the volume as air space to allow for expansion due to temperature changes.

In vacuum operations, glassware under vacuum should be protected from physical shock which might cause cracks and result in collapse with explosive violence. Flat-bottom flashes should not be subjected to vacuum unless constructed with heavy walls specifically for such service. Vacuum should be relieved before attempting to disassemble equipment.

5.7. Safety and emergency equipment

5.7.1. General considerations

"Safety" equipment is that designed to protect and/or prevent injury and is used before an accident happens. "Emergency" equipment is used after an accident (or other emergency) to minimize the injury or damage. Therefore, eye goggles are "safety" and eyewash fountains are "emergency" equipment.

Using these definitions, the following are lists of safety and emergency equipment.

(Safety equipment)

- Rubber aprons
- Eye goggles
- Face shields
- Disposable plastic gloves
- Bench shields (portable, clear plastic)
- Pipetting bulbs
- Heavy rubber carriers for acid and alkali bottles
- Metal safety cans for flammable solvents
- Metal solvent storage cabinets (about 160 litres capacity) (for storage of solvents used daily in the laboratory)
- Respirator filter masks (for dust or fumes).

(Emergency equipment)

- Hand-held fire extinguishers
- Fire blankets (wall mounted)
- Eyewash stations (built-in fountains or portable kits)
- Spillage absorbent kits for both acids and solvents (available commercially or can be assembled locally)
- Emergency showers
- Respirator masks with oxygen supply
- First aid cabinets.

All such equipment is useless if not available or not in serviceable condition. The Safety Officer should periodically check both the storage locations (if not in use) and the condition of all safety and emergency equipment. The laboratory staff should be trained in the use of the equipment and use must be enforced when necessary.

5.7.2. Emergency showers and eyewash stations

Some of the most important emergency facilities in a laboratory are eyewash stations, emergency showers, sprinklers and emergency exits.

Eyewash stations should be located near analytical work stations. It is, in fact, desirable to locate eyewash stations next to each sink and by the emergency showers, for easy and quick access.

Emergency showers should be located within a few seconds walk of the analytical work stations. The distance should be no greater than 30 metres from any location in the laboratory to the nearest shower.

In order to be effective, emergency showers (see Figure 1) need to release a large volume of water at low velocity (120 - 180 litres per minute). Because of the large volume of water, many installations have floor drains below the shower, although this is by no means standard practice. Floor drains are expensive and dry out because of infrequent use, allowing sewer gases to enter the laboratory.

Emergency shower valves should be installed so that they can be actuated by a person of any height. One very effective way is to run a cord down a clear wall so that it can be found easily and pulled.

Sprinkler systems are desirable and often required by law or regulation. They must be located carefully since some chemicals react violently to water or create inflammable gases when mixed with water. Sprinkler systems should be adjusted carefully in order to function correctly.

The minimum flow rate for plumbed eyewash equipment should be at least 12 litres per minute and, preferably, 24 to 36 litres per minute.

Cold water (about 10 °C) is recommended for emergency flushing of chemicals from eyes and body, as well as for emergency treatment of thermal burns. Cold water is generally best because it will slow the reaction rate of the chemical splashed, constrict blood vessels and minimize circulation of absorbed chemicals (hot water can increase the injury).

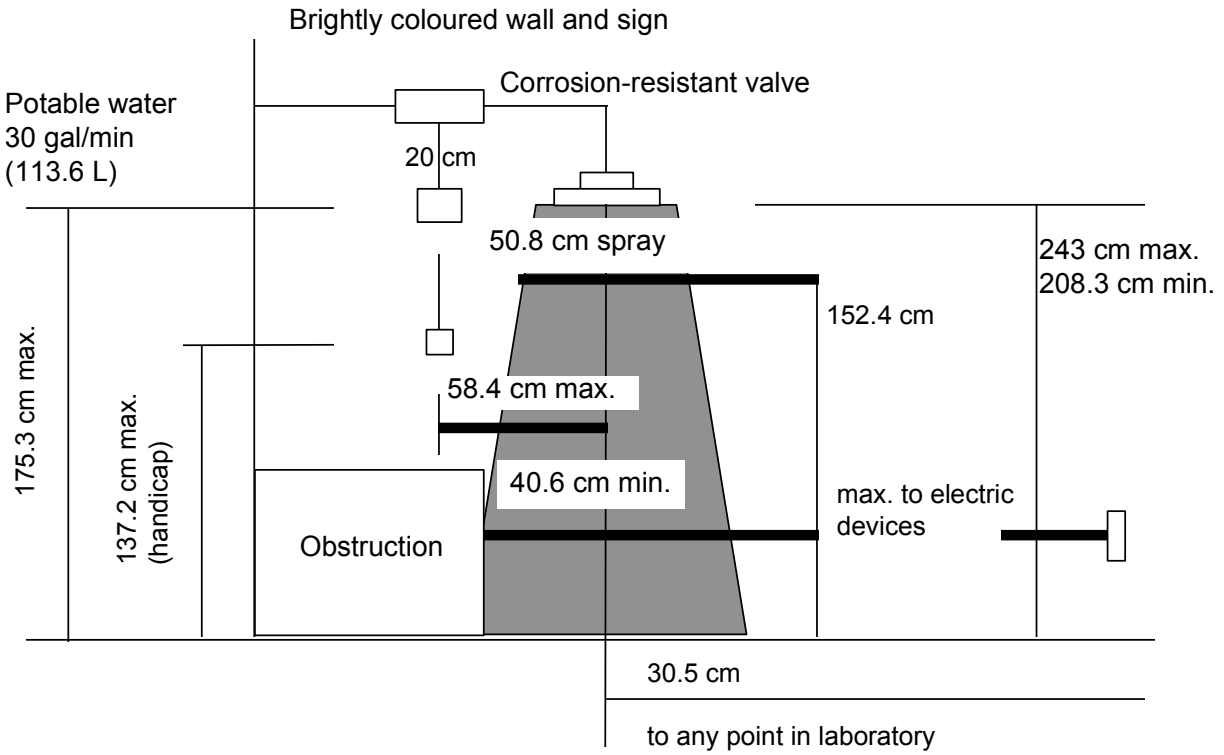


Fig. 1. Emergency shower - critical dimensions

Finally, two emergency exits should be located at the opposite ends of rooms for chemical analysis. They should be installed to allow the doors to push open in order to permit easy escape.

5.8. Training on safety and emergency measures

The Safety Officer should conduct emergency drills at least twice a year. Demonstrations of safety and emergency equipment should be included in such training. All safety and emergency equipment should be checked and, if inoperable, should be repaired or replaced as soon as possible.

New employees should be briefed by the Safety Officer on the location and use of safety and emergency equipment, the location of hazardous chemicals, ways to avoid or minimize accidents, escape routes and how to notify fire authorities.

5.9. Anti-pollution measures

Every employee in a laboratory should understand the importance of anti-pollution measures. Analysts should be familiar with the drainage equipment in the laboratory, its functions and efficiency. (See also Section 3.7 of this Guide on anti-pollution guidelines). Before used chemical compounds are drained, they should be neutralized, precipitated or separated, thus creating non-hazardous compounds of a consistency which may be drained. For example, cyanates and cyanate compounds should be burned or decomposed by the use of oxidising agents, such as chlorine or ozone before introduction into the drainage system.

Certain hazardous chemical wastes require special disposal by professionals. For example, heavy metal compounds, etc. should be stored separately and their disposal should be entrusted to qualified companies.

For the purposes of pollution control the analytical staff should co-operate closely with the building support staff, professional disposal companies and the sewer authorities.

6. Sampling

6.1. General considerations

In this section the normal sampling and sample preparation methods for analysis are described. If standard sampling procedures are laid down in ISO 2859 1-3, etc. (see III, Chapter 1 : International Standards and Methods), the sample collector should prepare the sample according to those procedures.

During sample collection, care must be taken not to damage the goods. It is recommended that the owner of the goods, or his or her representative, should be present when sampling is performed to the extend necessary.

Chemical analysis leads to successful practical results only when the sample for examination has a composition that is representative of that for the entire shipment.

The amount of the sample to be taken for analysis is usually predetermined. The sample collector must collect at least the minimum amount of goods necessary for laboratory purposes. In the case of an airtight container containing packages for retail sale, the smallest packing unit may generally be regarded as a suitable sample for analysis.

Since the sample being analysed must be representative of the shipment, the method of obtaining it is important. Most non-liquid materials, and sometimes liquid ones, may be far from homogeneous and careful work is necessary to obtain a useful sample from a very large shipment. The procedures vary somewhat with the nature, size and homogeneity of the original material and it would be difficult to provide set rules for general applicability. In cases where the shipment being sampled is large, small portions of the sample should be drawn systematically throughout the shipment with a view to combining them into one easily handled sample having the same average composition as the entire shipment. If the sample collector cannot mix the sample portions technically or cannot identify whether or not they are homogeneous, he or she should send each sample portion separately.

Liquid samples stored in a can or a drum should be drawn out after shaking, mixing or stirring sufficiently because they are not always homogeneous. Powder, particles or muddy samples which are packaged in a container should be gathered from the portion which is not in direct contact with the air. These samples should generally be taken from more than two separate containers. However, this rule need not apply to uniform commodities such as canned or bottled products.

Samples such as petroleum compounds or molasses which are stored in a tank or cistern should be taken from each of three layers (top, centre and bottom), after the tank or cistern is filled and the contents are allowed to stabilize.

To prevent or minimize contamination, decomposition, or matrix change, care should be taken with the sampling methods, sampling apparatus and sampling containers (e.g., containers made of polyethylene or polypropylene with double caps (one being a screw cap) to avoid loss and contamination of the content) being used. For samples which are sensitive to the atmosphere (moisture, carbon dioxide, etc.), rapid sampling is required. Clean and dry sampling apparatus and containers should be employed every time. In particular, dark containers should be used for samples which are sensitive to sunlight.

Once the sample has been collected, stringent precautionary measures should be taken to prevent the samples being exchanged or tampered with. Such measures may include sealing the sampling container or packaging with an official seal. Each sample should also be labelled immediately to indicate the name, number, sampling date, location, etc., in order to avoid any confusion.

Labels must be attached to samples in such a way that they cannot be torn off or damaged when the seals are broken to open the sample. The identification label must remain intact throughout the analysis.

Samples that are flammable, explosive, toxic, corrosive or poisonous should be handled carefully and a special marking such as "Dangerous" or "Hazardous" should appear on the sampling containers.

In the case of perishable goods, the containers should be prominently marked as "perishable" and the samples should rapidly be dispatched to the Customs laboratory.

Samples should be securely packed and sent to the laboratory as soon as possible with the accompanying sampling certificate which should give all necessary details concerning the sample, such as :

(1) Name, number, quantity (total net weight or unit size) and quality of sample

(2) Port of loading and unloading

(3) Date of arrival and sampling

(4) Name of the sample collector and client

(5) Number and date of declaration

(6) Name, address and telephone number of importer or agent, consignee and shipper

(7) Country of origin

(8) Use and price (f.o.b., c & f or c.i.f.)

(9) Sampling method

(10) Purpose and items of analysis

(11) Tariff number of declaration.

(12) Available technical information about the sample (e.g., technical literature, safety data sheet, catalogue, CAS number, composition, chemical structure, typical analysis results or specifications), etc.

Samples for analysis may be taken at many Customs stations. Therefore it is important for chemists to inform the officers concerned on how best to take a representative sample and on the necessary safety and security precautions related to the handling and storing of samples.

6.2. Dispatch of the sample to the laboratory

The sample should be dispatched with great care to ensure that it arrives at the laboratory in the same condition as when taken. The containers to be used and the manner in which the dispatch is to be handled depend very much on local conditions.

6.3. Sample receipt

When a sample is received in the laboratory, the laboratory staff should first identify the nature of the sample from the accompanying sampling certificate in order to store the sample properly. All samples (with accompanying documents) must be registered, marked and stored properly as soon as they arrive at the laboratory. The system shall ensure that the necessary information to identify the sample (including its origin) is recorded. It shall also ensure that there can be no confusion of samples (or related documents) during the analytical operations.

A sufficient level of security must be maintained, so that unauthorized persons cannot get access to the samples or the result.

Perishable goods should be stored in refrigerators or freezers to avoid deterioration before analysis.

The Customs laboratory should have a system for tracking the sample through its initial storage, its analysis and its subsequent disposal. This is usually done by means of a record-keeping system based on a unique number assigned to the sample at the time of sampling or on arrival in the laboratory. This number may be sequential, e.g., 0001 to 9999.

The record must show each movement of the sample, e.g., its receipt, its assignment to a laboratory analyst for analysis, its subsequent storage and its eventual disposal.

One of the administrative staff should be given this record-keeping function and be closely supervised by a senior administrator.

The use of a card record system rather than a book is recommended since cards are more flexible and may be organized in groups under different headings. Certain items of information should be included on each card :

(1) Sample number.

(2) Product name.

(3) Date sampled.

(4) Date received at the laboratory (the "date received" would normally be the date that the request is received; however, if the request and sample are received separately, the date received should be the latter of the two since that represents the earliest date that the laboratory could begin work on the request).

(5) Method of storage (dry, refrigeration, freezing, dark room, etc.).

(6) Storage location (coded for easy reference).

(7) Date assigned for analysis.

(8) To whom assigned (the analyst should initial to show receipt).

(9) Date returned (from analyst).

(10) Final disposition or disposal of the sample, including the method and date.

The above sample record is to record only physical movement and location, not the analytical results. The reason is that the analytical worksheet and the sample record are usually in two different locations.

The sample record card should be in duplicate. One copy goes to the sample co-ordinator who holds on to the sample until the analyst is ready to perform the analysis, and another copy to the laboratory supervisor who assigns the sample. This sample assignment could be done manually, by writing on the copy of the sample card the initial of the assigned analyst and date, or through computer. It is then given to the analyst. When the analyst is ready to analyse the sample, he or she retrieves the sample from the sample co-ordinator who fills out the original copy of the sample card with the relevant information, such as the name of analyst, date of assignment, date sample was given to the analyst, etc. After analysis and preparation of the laboratory report, the analyst returns the card with the remaining sample to the sample co-ordinator. The co-ordinator fills out the original copy of the sample card with information such as the date the sample returned, final disposition or method of disposal and date. This card should then be filed separately from the laboratory worksheet and the laboratory report.

Sample record cards should be prepared and kept in the custody of one person until the case is brought to a close and the sample is destroyed. This function is critical to the operation of the laboratory and therefore the person employed in this post must be reliable. However, this task should not usually occupy a person full-time and therefore could be combined with other duties in the laboratory, such as store-keeping of laboratory chemicals, glassware and other equipment, etc.

6.4. Sample storage and disposal

Sample storage, both initial and reserve, is critical to a sample analysis. Improper storage can completely invalidate any analytical results. Ideally, the sample should be stored in such a manner as to prevent any change in the attributes being examined, from the time of sampling, through analysis, and into reserve storage.

The usual storage areas are dry (room temperature storage), refrigerated or frozen. In order to minimize exposure to changes during storage it is important to use proper sampling containers for the samples.

Samples are normally kept for a period of time after analysis in case further information or analysis is requested or the results of the laboratory's work are disputed. For this purpose, the remaining samples should be returned to the sample co-ordinator after analysis. Samples should be kept at least as long as the time allowed for an appeal from the importer or exporter. Samples for which analysis is susceptible to dispute should be stored in a separate area to reduce the chance of their being inadvertently destroyed.

Sample disposal is, or can be, a relatively simple matter. Problems only arise when there is a hazard involved in destruction or when the sample itself presents a hazard to the disposer. The analyst should know of any hazards involved and should inform the person in charge of the sample storage of any special disposal requirements.

6.5. Analysis assignment

In a large laboratory, the assignment of a request for analysis might be a two-stage process, i.e., initial assignment to a Section Head who then assigns the work to a chemist within the Section. In a smaller laboratory the assignment of work might be assigned directly to a chemist.

(Note : The sample should be retained in a "sample room" until the chemist is ready to begin work on the sample since it is strongly recommended that chemical and other potentially hazardous samples should not be handled in the general office environment).

The chemist who performs the analytical work must identify and define analytical problems and establish results in accordance with the methods to be used. Generally, it is preferable to use a method that has been subjected to collaborative study. If the analyst has difficulty with such a method, it is likely to be due to a deficiency in training or in facilities, rather than the method itself. If a method has been in use and found to give reliable results it should not be changed to another until the new method has been shown to be of equal or better reliability in that particular laboratory.

It should not, however, be forgotten that a reliable method is only a precondition to obtaining the right answer. The ability of the analyst to apply the method properly is equally important.

7. Operation

7.1. Safety rules

It is essential for Customs laboratory staff to analyse the requested sample safely and to furnish accurate and reproducible results after the examination.

All staff at Customs laboratories should observe the safety rules laid down in "Safety and Anti-pollution Measures" (see Section 5). Analytical staff should wear a laboratory apron or coat, laboratory shoes and protective eyeglasses; they should know the location and proper use of safety equipment such as fire extinguishers, fire blankets, safety fountains or showers, and equipment for handling spills. It is also recommended that the laboratory be kept clean and that all chemical reagents, apparatus and samples be put away when staff are finished with them. If they get into difficulties, analysis staff should report to their supervisor or colleagues immediately.

7.2. Basic rules to be followed in the laboratory

Each experiment should be understood thoroughly in advance, and appropriate experiment programmes and time-tables should be prepared by each analyst.

It is essential to use the minimum amount of reagents and samples necessary for each experiment. To avoid contamination of the stock bottles, appropriate amounts of reagents, chemicals or solutions should first be taken to each test-tube or beaker from the stock bottle, and these materials should be used for each experiment. When the experiment is over, dispose of reagents, chemicals, etc., properly. Never return them to the stock bottles.

Walk slowly and be careful when handling any apparatus in the laboratory. Do not heat any apparatus for quantitative analysis and any glassware at the point where two thicknesses of glass meet. Wash your hands before and after every experiment and check the labels or markings at least twice before taking anything from a bottle or container.

It is, of course, essential to keep all rooms clean and to put away apparatus, sample containers and tools after each experiment. Before locking up, someone must check that, in all the rooms, the gas and liquid valves are closed and electrical instruments switched off.

7.3. Investigation of samples and analytical methods

With reference to the nature and quantity of the sample, it is essential to draw as much information as possible from the accompanying certificate and relevant documents. Past data relating to a similar sample should be used, if possible. If the analyst wants to obtain additional information on the sample, he or she may ask the importer or exporter. If necessary, simple screening tests are carried out in advance to ensure a successful experiment. On the basis of all this information, the analyst can draft an experiment programme for systematic analysis. As far as analytical methods are concerned, validated methods should be employed.

In addition, standard test methods for samples analysed frequently should be developed to facilitate analytical work and to ensure accuracy and precision of data.

7.4. Procedures for using laboratory apparatus

Since many different types of apparatus are used for analytical experiments, it is necessary to understand the purpose, use, capacity, etc., of each of them. When an experiment is finished, it is recommended that the apparatus be cleaned and stored properly.

7.4.1. Procedures for using glassware apparatus

It is necessary to check all glassware to determine whether there are any cracks or chips. If so, the glassware should be disposed of.

The analyst must wash glassware carefully with a brush in warm water and detergent, then rinse completely with tap water and finally rinse with a minimal amount of distilled and/or deionized water until the water spreads out evenly on the clean glass. In the case of quantitative glassware, a brush and cleanser should not be used. After analysing oils or fat with any apparatus, the analyst should wash the apparatus with an organic solvent in which the sample is soluble. The analyst may use a solution of CrO_3 or $K_2Cr_2O_7$ in concentrated H_2SO_4; however these solutions must be handled carefully.

(Volumetric flasks)

A volumetric flask is used to dilute a certain volume or weight of sample or solution with diluent to a certain volume.

The proper method of reading a meniscus in volumetric measurements of liquid with quantitative glassware is with the eye on a level with the meniscus, and where the bottom of the meniscus is used to indicate the volume of the liquid.

When handling the volumetric flask, the sample or solution should be diluted in stages, shaking the flask to ensure thorough mixing. The diluent should be added slowly so that the bottom of the meniscus is even with the middle of the calibration mark at eye level.

The solution should then be thoroughly mixed, keeping the stopper securely in place.

(Burettes)

A burette is used to determine the accurate volume of a standard volumetric solution which is used for volumetric analysis.

When using a burette for quantitative analysis, it must first be rinsed a few times with about 10 ml of standard volumetric solution. It should then be filled with standard volumetric solution and the stopcock should be opened several times until all air is removed from the tip. Finally, it should be filled to just below the zero mark with standard volumetric solution and the volume of standard volumetric solution can be read. An erlenmeyer flask, in which a constant volume of the test sample solution is poured, is placed on white paper under the burette. The standard volumetric solution may be added from the burette rapidly until it is almost at an end point; the flow of the solution should then be reduced until individual drops fall into the flask. Add the last few drops slowly, shaking the flask to ensure thorough mixing.

(Transfer pipettes)

A transfer pipette is used to transfer a constant volume of a solution.

When using a transfer pipette, it is desirable to use a safe pipette controller to avoid any problems. The solution should be drawn up past the calibration mark. The outside of the pipette should then be wiped with tissue paper and the solution allowed to flow out until the bottom of the meniscus is just at the calibration ring; to pick off the last drop adhering to the outside of the tip, touch the side of the flask with the tip. Then withdraw the pipette from the flask and hold it over the container into which the solution is to be transferred. Allow the pipette to drain in a vertical position, with the tip against the side of the container. Allow 15 to 20 seconds for drainage after it appears that most of the solution has drained out. The liquid remaining in the tip of the pipette should not be blown out, if the pipette is calibrated for this amount to remain.

7.4.2. Analytical balance

Weighing is necessary to measure the weight of the sample, standard substance and reagent in any analysis. In Customs laboratory, the analyst deals with a broad range of weights, from a few kilogrammes down to a few milligrammes or less. The analyst uses mass rather than weight, because mass is an invariant value worldwide. In Customs laboratories, a single-pan balance is more useful than a double-pan balance for reasons of rapidity. More recently, electronic analytical balances have been introduced into Customs laboratories. No weights or knife edges are involved. The weights are automatically indicated by placing a weighing bottle on the pan after setting the balance to zero. There are two kinds of weighing: "rough" and "accurate". The former is normally used when it is not necessary to measure the accurate weight, where reagents are simply added to adjust the solution conditions. On the other hand, the accurate weight of the samples or reagents may be necessary, particularly, in quantitative analysis. Accurate weighings are performed on an analytical balance, usually to the nearest 0.1 mg.

Avoid vibration or jarring when determining accurate weights and keep the analytical balance clean to protect all parts from dust or contamination. Hot or cold samples (or reagents) must be brought to room temperature in a cool desiccator to avoid the influence of the atmosphere, and they should then be weighed at room temperature. The door of the balance case should always be kept closed, except when the weighing bottle is being put in or taken out. The sample should be weighed in the weighing bottle (or dish) or on powder paper. If the sample includes volatile constituents or tends to absorb moisture in air, it should be dried and weighed in a weighing bottle which is capped to prevent evaporation or moisture absorption during weighing. After setting the balance to zero, the analyst should weigh the empty weighing bottle, which has been preheated for more than two hours in an oven at an agreed temperature (e.g., 110 ± 2 °C) and has been cooled in a desiccator, or powder paper (1), then weigh the weighing bottle or powder paper with the sample added (2). The sample's weight is as follows:

$$\text{Weight of sample} = (2) - (1)$$

If the weight of an empty weighing bottle or powder paper is already set at zero on the balance, the weight of the sample is displayed directly. During weighing, the sample should not be added to the weighing bottle on the pan or be removed from the weighing bottle on the pan. The weighing bottle should not be handled directly with the hands. It is desirable to handle the weighing bottle with tongs, a piece of

paper or clean gloves. If the sample or reagent spills over the pan, the analyst should immediately clean it up with a brush or with a paper towel or tissue paper.

7.5. Screening analysis

7.5.1. Appearance and nature of sample

When an analyst receives a sample, the appearance, weight and nature of the sample should first be observed. The appearance and nature of the sample should be examined using the sense of sight, smell or hearing. The state, colour, smell, size, appearance, weight, etc., of the sample should be noted in writing. If necessary, wet samples should be dried and the dry matter tested.

Next, the solubility in various solvents, the melting point, the boiling point, the specific gravity, etc., should be examined if necessary.

7.5.2. Colour test and precipitate reaction

7.5.2.1. Apparatus

The reagent bottles, test-tubes, test-tube support, test-tube holder, centrifuge tube, porcelain spot test plate, glass stirring rods, platinum wire, burner, wire gauze with asbestos centre, medicine dropper, funnels, paper filter, spatula, pH test paper, etc., should be prepared in advance.

7.5.2.2. Reagents

Each experiment should be understood thoroughly and the necessary reagents should be prepared by analyst in advance. Each reagent must be prepared according to the interpretation of each test method. Some reagent solutions are unstable and their period of use limited; freshly prepared reagents should be employed whenever possible or necessary. Even if the solution is stable, the analyst ideally should prepare a quantity of that solution once or twice a year to avoid any problems. It is also desirable to prepare reference chemicals or materials for the control experiments.

7.5.2.3. Purpose

These tests are carried out to provide a rapid screening of ions, functional groups, compounds and elements included in the sample; they are very useful for a systematic analysis.

7.5.2.4. Test

A preliminary test should be carried out in advance if the analyst has enough sample and reagent, so as to provide a guideline for the following test. In parallel with the test of the sample, the analyst should carry out a control and blank test using the same quantities of all reagents to ensure the detection of the analyte. The suitable amount of the sample or reagent prescribed in the analytical methods should be used in order to avoid any problems. The analyst should note (in writing) the degree of colour change and the amount of precipitate for future testing.

7.5.2.5. Methods

Some testing methods generally used in Customs laboratories are as follows :

Beilstein test : halogens

Alkali metal fusion method : halogens, sulphur, nitrogen, phosphorous

Liebermann reaction : phenol (also reaction with ferric chloride)

Simon reaction : secondary amines

Chromotropic acid-sulphuric acid reaction : form-aldehyde

Precipitation reaction : $SO_4^{2-} + Ba^{2+} \Rightarrow BaSO_4\downarrow$

$$Ag^+ + X^- \Rightarrow AgX\downarrow$$

X : Halogen

These and other testing methods are set out in many technical publications (see Chapter 4. Technical literature and reference books).

7.6. General concept of chemical equilibrium

The chemical reaction between the reacting species A and B is generally as follows :

$$aA + bB \leftrightarrow cC + dD$$

a, b, c and d : coefficients
C and D : reaction products from the chemical reaction between A and B

When this system is at equilibrium, the equilibrium constant *k* is described as

$$k = \frac{[C]^c\,[D]^d}{[A]^a\,[B]^b}$$

[A] : molar concentration of species A,

e.g., the equilibrium constant for water
($2H_2O \leftrightarrow H_3O^+ + OH^-$) is

$$k = \frac{[H_3O^+]\,[OH^-]}{[H_2O]^2}$$

Because the activity of water is constant in dilute solutions (~ 55.3 *M*), the self-ionisation constant k_w is as follows :

$$k_w = [H_3O^+]\,[OH^-] = 1 \times 10^{-14} \text{ (at room temperature)}$$

The pH of the solution is defined as

$$pH = -\log[H_3O^+] \text{ or } -\log[H^+] = 14 - \log[OH^-] = 14 - pOH$$

On the other hand, if the solubility of one species is limited and exceeded, e.g., [C] ~ 0, this species is called "insoluble" and the chemical reaction between the species A and B proceeds to the right and the precipitate C occurs in the solution.

$$aA + bB \rightarrow cC\downarrow + dD$$

In a redox reaction, oxidation involves the loss of electrons by species and reduction involves the increase in electrons. The reaction is as follows:

$$a\underline{Ox} + ne^- \underset{\text{Oxidation}}{\overset{\text{Reduction}}{\leftrightarrow}} b\underline{Red}$$

The potential of this reaction depends on the concentration of the species and the relationship between them is described by the Nernst equation as follows :

$$E = E_o - \frac{2.3026RT}{nF} \log \frac{[Red]^b}{[Ox]^a}$$

E : Reduction potential
E_o : Standard potential
R : Gas constant (8.3143 V coul eq^{-1})
T : Absolute temperature (K : 274.16 + °C)
n : Number of electrons
F : Faraday constant (96.487 coul eq^{-1})

Molarity calculations are as follows :

$$\text{moles} = \frac{\text{sample weight (g)}}{\text{molecular weight (M.W.)}}$$

$$\text{M (molar concentration)} = \frac{\text{mol}}{\text{l}} = \frac{\text{mmol}}{\text{ml}}$$

Normality calculations is as follows;
Eq. = mol x number of reacting units*

*) e.g., reacting unit ; HCl = 1, H_2SO_4 = 2, NH_3 = 1,

$$N \text{ (normal concentration)} = \frac{\text{Eq.}}{\text{l}} = \frac{\text{mEq.}}{\text{ml}}$$

7.7. Gravimetric analysis

There are mainly two types of weighing. One involves a sample being chemically converted into another substance of low solubility and known composition, which is then filtered, dried, sometimes ignited, and weighed. The other involves one component in the sample being volatilized, collected and weighed; similarly, after one component is removed, the remaining residue may be weighed.

7.7.1. Precipitation method

Precipitation methods with inorganic reagents are very useful for quantitative analyses for Cl^-, SO_3^{2-}, etc., in a cost-effective manner.

Organic reagents have been used more selectively in the precipitation method for inorganic ions.

However, these analyses are not specific to a particular ion but rather form precipitates with groups of ions; for example, the chloride ion is determined by precipitation of the salt by Ag^+ to form AgCl, but other halogen (iodide and bromide) ions in the sample will also be coprecipitated and the resulting analysis will be high.

$$Cl^- + Ag^+ \rightarrow AgCl\downarrow \text{ (precipitate)}$$

and $Br^- + I^- + 2Ag^+ \rightarrow AgBr\downarrow + AgI\downarrow$
(precipitate : impurity)

A test sample solution is placed in a container (beaker) and a small excess of reagent is added to the test sample solution, inducing precipitation of a reaction product. The precipitate is transferred onto a filter and properly washed with a solvent or solution in which the precipitate is insoluble. The precipitate is then dried and weighed.

There are several means of increasing the particle size of the precipitate to increase purity and optimise the effectiveness of filtration, including the following

- heating the sample container gently in advance;
- using a dilute sample and reagent;
- adding the reagents slowly, while stirring;
- cooling the sample solution gradually to room temperature.

Application : Quantitative analysis of e.g., halogen ions, sulphuric acid ions and silicone oxide. Ion-chromatography is now employed in advanced Customs laboratories to determine halogen ions and sulphuric acid ions.

7.7.2. Volatilization method

Volatilization methods are used for the determination of dry residue and volatile matter in water or volatile organic solvent solutions.

(Determination of dry residue)

A 1 g sample is accurately weighed in a sample container (weighing bottle (containing a glass rod), etc.) which has been preheated for more than two hours in an oven at a temperature of e.g., 110 ± 2 °C and then cooled in a desiccator. The container is then placed on/in a water bath to evaporate the volatile matter. After wiping the outside of the sample container with tissue paper, the residue is dried in the oven, again at a temperature of 110 ± 2 °C, to constant mass (see ISO 759, 3251, etc.).

$$\text{Dry residue (\%)} = \frac{\text{Constant mass of residue}}{\text{Weight of original sample}} \times 100$$

(Determination of volatile matter)

In the case of aqueous solutions, the Karl Fischer method (ISO 3733) has often been employed in Customs laboratories to determine water. For the determination of volatile organic solvents, the following distillation methods can be employed : ASTM D 86, 850, 938 and ISO 918, etc.

7.8. Volumetric analysis

This technique is based on the measurement of the volume of a standard volumetric solution (a reagent) that must be titrated to react completely (equivalence point) with all material in a sample to be analysed. The following experiment should be carried out at constant temperature, since the volume of standard volumetric solutions depends on the temperature.

There are two types of preparation methods for obtaining standard volumetric solutions. If a standard substance is sufficiently pure, a weighed quantity of standard substance is dissolved and diluted to a specific volume. Otherwise the standard volumetric solution is standardized indirectly by titrating a weighed quantity of primary standard or secondary standard solution.

The end point of titration is generally detected by a colour change (brought about by an added indicator) or other physical properties such as conductivity, electrical potential, etc.

The following are required for volumetric analysis :

- The equilibrium constant is very large and the reaction proceeds quantitatively;
- The reaction speed is rapid;
- An appropriate indicator, etc., to identify the end point is often present;
- There should be no component in the sample to interfere with the reaction.

7.8.1. Acid-base titration analysis

This technique is very useful for determining the concentration of acid or base in a sample solution in a cost-effective manner.

If a marked change in pH cannot be obtained, a back-titration method is employed. In the case of a weak acid or base, which cannot be determined in water, non-aqueous titration techniques can be employed.

The sample is titrated with a standard volumetric solution of a strong acid or a strong base. A titration curve is constructed by plotting the pH of the solution as a function of the volume of the standard volumetric solution added. An example of a titration curve is shown in Fig. 1. The end point is usually indicated by a sharp change in pH that occurs at the equivalence point. Indicators and a pH meter are used to detect the end point. The concentration of acid or base in the sample solution is calculated as follows;

$$M = \frac{N \times y \times 1000 \times d}{u \times 1000 \times x}$$

u : Number of reacting units
e.g., HCl = 1, H_2SO_4 = 2, NH_3 = 1,
M : Molar concentration of sample solution
x : Volume of sample solution (ml)
N: Normal concentration of standard volumetric solution
y : Volume of standard volumetric solution at the end point (ml)
d : Degree of dilution

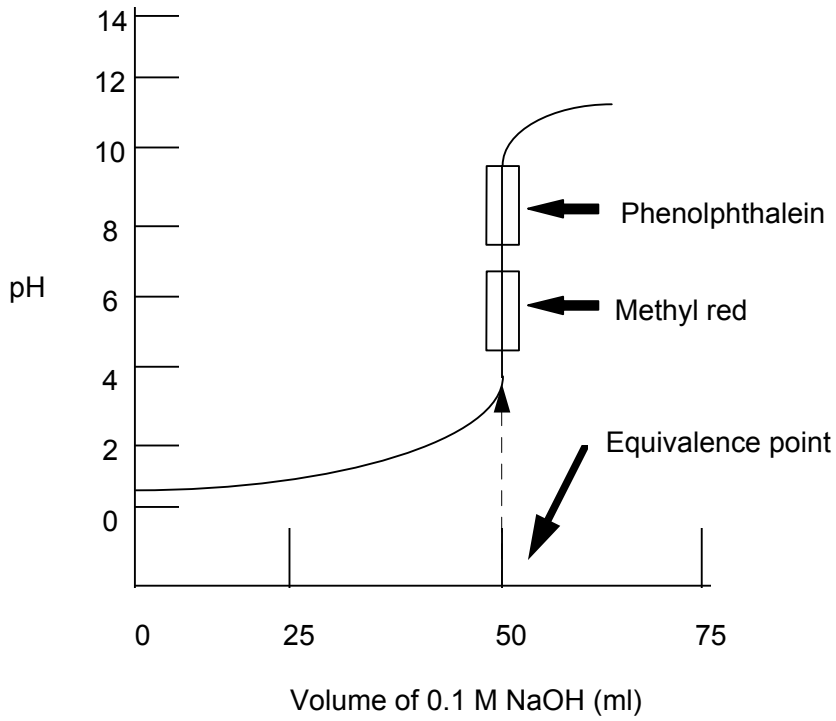


Fig. 1. Titration curve for 50 ml of 0.1 M HCl versus 0.1 M NaOH

a. Preparation of standard base solutions

A saturated NaOH solution is prepared with distilled deionized water and NaOH, and stored in a dark room. Several days later, this solution is filtered to remove Na_2CO_3 and diluted to appropriate concentration with distilled deionized water. As a primary standard, potassium acid phthalate is employed to standardize the NaOH solution. Phenolphthalein is normally used as an indicator. The standardized NaOH solution should be stored in a plastic bottle with a screw-cap. Commercially available standardized NaOH solution is also widely used in Customs laboratories.

b. Preparation of standard acid solutions

Concentrated HCl is diluted to the appropriate concentration with distilled deionized water. This solution is standardized by titrating a primary standard (sodium carbonate or tris(hydroxymethyl) aminomethane (THAM)). A standardized NaOH solution is also employed to standardize the HCl solution as a secondary standard. Commercially available standardized HCl solution is also very convenient.

c. Indicators

Transition ranges of pH and colours of some common indicators are shown in table 1.

Table 1 Indicators

Indicator	Colour*	pH*
Bromophenol blue	yellow ↔ blue	3.0 - 4.6
Methyl orange	red ↔ yellow	3.1 - 4.4
Methyl red	red ↔ yellow	4.4 - 6.2
Bromothymol blue	yellow ↔ blue	6.0 - 7.6
Phenolphthalein	colourless ↔ deep-red	8.5 - 9.0

* Source : The Merck Index, Eleventh Edition.

d. Titration procedures

A sample is generally diluted to the appropriate concentration with distilled water by means of pipettes and volumetric flasks; this solution is called the test sample solution. A constant amount of reference, blank and test sample solutions should be titrated with the standard volumetric solution in advance in the presence of a few drops of the indicator to ascertain the colour change of the indicator and to determine the approximate concentration of the test sample solution. Titration of the blank and the test sample solutions should subsequently be carried out. The standard volumetric solution should initially be added rapidly and, on nearing the end point, it should be added slowly drop by drop; the flask should be shaken frequently to ensure thorough mixing.

Application : Quantitative analysis of e.g., inorganic acids and bases, organic carboxylic acids, organic amines, amino acids, drugs. Ion-chromatography is often employed in advanced Customs laboratories to determine inorganic and organic acids, etc. The Kjeldahl analysis for determining nitrogen is well known.

7.8.2. Precipitation titration analysis

The Mohr method is well known as the most commonly used precipitation titration. This technique is used to determine the concentration of anion in a sample solution. The sample is titrated with a precipitating agent (standard volumetric solution). Two types of indicators are generally used for the detection of the end point. One indicator reacts with the precipitating agent in excess after the equivalence point and forms a coloured compound. Another is adsorbed on the surface of the precipitate at the equivalence point and a colour change takes place. An example of the Mohr method is as follows :

$$n\mathrm{Cl}^- + \mathrm{Ag}^+ \rightarrow\rightarrow\rightarrow\rightarrow n\mathrm{AgCl}\downarrow \xrightarrow[\mathrm{CrO_4^-\ (yellow)}]{\mathrm{Ag^+\ (in\ excess)}} \mathrm{Ag_2CrO_4}\downarrow \text{ (red)}$$

a. Indicators

Some common indicators are shown in table 2.

Table 2 Indicators

Indicator	Colour*	pH*
(Mohr method) Potassium chromate	yellow $\leftrightarrow$ red	
(Fajans' method) Fluorescein Dichlorofluorescein	yellowish-red orange	7 - 8 4

* Source : The Merck Index, Eleventh Edition.

b. Titration procedures

A sample is generally diluted to the appropriate concentration with distilled water by means of pipettes and volumetric flasks; this solution is called the test sample solution. A constant amount of reference, blank and test sample solutions should be titrated with the standard volumetric solution in advance in the presence of a few drops of the indicator to ascertain the colour change of the indicator and to determine the approximate concentration of the test sample solution. Titration of the blank and the test sample solutions should subsequently be carried out. The standard volumetric solution should initially be added rapidly and, on nearing the end point, it should be added slowly drop by drop; the flask should be shaken frequently to ensure thorough mixing.

Application : Quantitative analysis of e.g., Cl^-, Br^-, I^-, SCN^-.

7.8.3. Complexometric titration analysis

This technique can be used to perform quantitative analyses of certain metal ions in a cost-effective manner; however, it requires skilled and experienced analysts and is also time consuming. ICP (see 7.12.1.2) or AAS (see 7.12.2) methods are now often employed in advanced Customs laboratories on account of their accuracy,

rapidity and simplicity (these techniques do, however, require expensive and complex instrumentation).

This technique is a complex formation titration to determine the concentration of a certain metal ion with an electron pair donor (ligand). Ethylenediamine tetraacetic acid (EDTA) is well known as a complexing agent (ligand) and usually indicated as "H_4Y". Since the selectivity of EDTA depends on the pH of a test sample solution, varying the pH of the solution with a buffer solution and the use of masking agents make it possible to titrate different groups of metal ions.

a. Preparation of high-purity EDTA and standard volumetric solutions

A high-purity EDTA solution is prepared by drying disodium ethylenediamine tetraacetate dihydrate at 80 °C for two hours. In a back-titration, high-purity metal solutions are used as standard volumetric solutions and the kinds of metals to be used depend on the sample element to be analysed.

a. Indicators

Eriochrome Black T (BT, EBT; HIn^{2-}) is well known as a typical indicator for this technique. Calmagite and Xylenol orange are also used as indicators.

$$\underset{\text{red } (\leq \text{pH } 6)}{H_2In^-} \overset{pK_a=6.3}{\leftrightarrow} \underset{\text{blue (pH 7-11)}}{H_2In^{2-}} \overset{pK_a=11.55}{\leftrightarrow} \underset{\text{red orange } (\geq \text{pH } 11)}{H_2In^{3-}}$$

b. Back-titration procedure

After pre-treatment of a sample, a constant volume of EDTA solution is added to the test sample solution and after adjusting to the appropriate pH of this solution with hexamethylene tetramine, the excess EDTA is titrated with the standard volumetric solution.

Application : Quantitative analysis of e.g., metal ions

7.8.4. Reduction-oxidation titration analysis

This technique is based on an oxidation-reduction (redox) reaction. While oxidizing agents tend to take on one or more electrons and be reduced to a lower oxidation state, reducing agents tend to bring the reverse change. For example :

$\underline{MnO_4^-}$ (pink) + $8H^+$ + $5e^-$ $\leftrightarrow$ Mn^{2+} (colourless) + $2H_2O$
(Oxidizing agent : Mn^{+7})

This technique is often employed in Customs laboratories for quantitative analyses (e.g., sucrose, MnO_4^-, etc.) and is accurate, useful, and less costly; other more accurate, more rapid and simpler methods (e.g., high-performance liquid chromatography (HPLC) for quantitative analysis of sugars, ion chromatography for quantitative analysis of certain inorganic and organic ions, etc.) have now been developed.

a. Preparation of standard oxidizing solution

Potassium permanganate solution is well known as a standard oxidizing solution. This solution is boiled and placed in a dark room overnight. Next morning it is filtered to remove the impurities (MnO_2) and then standardized by titrating a weighed quantity of primary standard, sodium oxalate ($Na_2C_2O_4$), in an acid solution.

b. Preparation of standard reducing solution

Sodium thiosulphate solution is widely used as a standard reducing solution and is stable in air for a long time. This solution is prepared from pure sodium thiosulphate and is also commercially available.

c. Indicators

Potassium permanganate solution is also used as a self-indicator.

A starch indicator is used for titration involving iodine. In the presence of starch, any occurrence of I_2 in a redox reaction changes a colourless solution to a dark-blue one.

Other redox indicators are shown in table 3.

Table 3 Other redox indicators

Indicator	Reduced form*	Oxidized form*	Solution*
Methylene blue	blue	colourless	1 *M* acid
Indigo tetrasulphonate	colourless	blue	1 *M* acid
Diphenylamine	colourless	violet	1 *M* H_2SO_4

* Source : The Merck Index, Eleventh Edition.

d. Preparation of test sample

Sample elements to be analysed should be converted to the appropriate oxidation state for titration, by adding certain oxidizing or reducing agents. The excess of these agents can be removed before titration. Reference and blank solutions should also be prepared in the same manner. The preoxidizing and prereducing agents generally used are shown in table 4.

Table 4 Preoxidizing and prereducing agents

Agents	Oxidation state	Removal method
(Prereducing agents) Sodium sulphite and sulphur dioxide Stannous chloride Pb Zn	 Th(III) → Th(I) Fe(III) → Fe(II) Cu(II) → Cu(I) Fe(III) → Fe(II) U(VI) → U(IV) Sn(IV) → Sn(II) $2H^+$ → H_2	 Boiling or bubbling with CO_2 Adding $HgCl_2$ (excess) Filtration Filtration
(Preoxidizing agents) Perchloric acid Potassium persulphate Permanganate Bromine Hydrogen peroxide	 Cr(III) → Cr(IV) Cr(III) → Cr(IV) V(IV) → V(V) V(IV) → V(V) Cr(III) → Cr(IV) Ti(I) → Ti(III) Co(II) → Co(III)	 Boiling the diluted solution Boiling Adding hydrazine and boiling Boiling Boiling

e. Titration procedure

A constant amount of reference, blank and test sample solutions should be titrated with the standard volumetric solution in advance in the presence of a few drops of the indicator to ascertain the colour change of the indicator and to determine the approximate concentration of the test sample solution. Titration of the blank and the test sample solutions should subsequently be carried out. The standard volumetric solution should initially be added rapidly and, on nearing the end point, the standard volumetric solution should be added slowly drop by drop; the flask should be shaken frequently to ensure thorough mixing.

Application : Quantitative analysis of samples which have oxidation and reduction ability, e.g., metal ions, saccharides, aldehydes.

7.9. Electrolytic analysis

This technique is based on the transfer of electrons from one species to another. In redox reactions, oxidation involves a loss of electrons by species and reduction involves an increase in electrons.

This technique can be used to perform quantitative analysis of certain metal ions in a cost-effective manner; however, it requires skilled and experienced analysts and is also time consuming. ICP (see 7.12.1.2) or AAS (see 7.12.2) methods are now often employed in advanced Customs laboratories on account of their accuracy, rapidity and simplicity; these techniques do, however, require expensive and complex instrumentation.

7.9.1. Potentiometric analysis

This technique is used to ascertain the concentration of sample elements by determining the potential between reference and indicator electrons without the passage of current in the cell. The saturated calomel electrode (SCE) is well known as a reference electrode. There are two types of indicator electrodes : metallic electrodes such as silver, copper and mercury, and membrane electrodes such as the glass electrode used for pH meters.

7.9.1.1. Potentiometric titration

Standard and test sample solutions should be prepared including a high concentration of electrolyte. For the preparation of a standard solution, any species in the sample which affect the determination of sample elements to be analysed should be added to this solution. An appropriate amount of solution is added to a beaker on a stirrer. After setting with the indicator and reference electrode and switching on the stirrer, the standard and test sample solutions are titrated with a standard volumetric solution. A standard volumetric solution should initially be added rapidly and, on nearing the end point, standard volumetric solution should be added slowly drop by drop. The burette and potential reading should be recorded. The titration is continued through the end point. The end point is determined by interpolation or from the titration curve. After standardization of the standard volumetric solution by a primary standard, the concentration of the sample is calculated. Whenever transferring the electrode from one solution to another, thorough washing with ionized water and carefully wiping with clean tissue are required.

7.9.1.2. pH meter

This is a device for determining the pH of a sample solution. As glass electrodes tend to break, careful handling is required.

The voltage of this cell E is as follows :

$$E = k - \frac{2.3026RT}{F} \log \frac{1}{[H^+]} = k + a\log[H^+] = k + a\text{pH}$$

$$\text{pH} = \frac{(E-k)}{a}$$

This technique can normally be calibrated at two points : one at pH 4.00 or 9.22 and another at pH 6.88 in the pH buffers at 20 °C. When transferring the electrode from one solution to another, wash thoroughly with deionized water and wipe carefully with clean tissue. After adjusting of the pH, the potential of the test sample solution is then determined and the pH value of the test sample solution is indicated on the display. Once finished, the electrode should be thoroughly washed with deionized water and capped with deionized water to prevent it from drying.

7.9.2. Voltametry analysis

7.9.2.1. Polarography

Polarography is a kind of voltametry and a dropping mercury electrode (DME) is used as working electrode. The plot of current (μA) versus voltage (V) is called a polarogram. An example of a polarogram is shown in figure 2 (not reproduced). Half wave potential $E_{1/2}$ depends on the species being analysed and the diffusion current i_d is proportional to the concentration of the analytical species.

Preparation of calibration curve and determination of concentration of sample elements

After preparation of several kinds of standard solutions and test sample solutions, each polarogram is determined under the same conditions. A calibration curve of the diffusion current i_d (y axis) versus the concentration of metal ion in standard solutions (x axis) is first plotted. The induced equation is calculated as follows :

$y = b + ax$

a : slope of calibration curve
b : intercept of y axis

The concentration of the sample element is then calculated on the basis of the above equation.

7.9.2.2. Amperometric titration

In this technique, the current passing through a cell at fixed potential is measured at different points in a titration and plotted against the titrated volume of standard volumetric solution. From the equivalence point, which is given by extrapolation to the intersection of the two lines, the concentration of the sample elements can be calculated.

7.9.2.3. Electrophoresis

This technique is extremely good for identifying the species of water-soluble proteins. A variety of supports may be prepared or are commercially available, including paper, gels such as starch, agar and polyamide.

After developing and colouring, characterized chromatograms are available; a detailed comparison of the sample chromatograms with standard chromatograms of proteins makes it possible to identify the kind of protein.

Application : Qualitative analysis of water-soluble proteins to identify the species of meats, plants, etc.

7.10. Separation of mixture samples

Separation procedures are essential for identifying the composition of traded commodities. In the case of mixture samples which consist of more than two kinds of constituents, analysts are often required to separate and to identify each constituent qualitatively and quantitatively. Though certain separation procedures are laid down in 7.7 "Gravimetric analysis", further separation procedures especially for organic samples are described in this section.

7.10.1. Separation procedures by appearance

This separation technique is occasionally possible for mixture samples where each constituent has a different appearance, e.g., a different colour, size, crystal form, etc. Analysts can separate these constituents by hand with a pincette or using a sieve with various sizes of aperture (see Chapter 11, Notes 2 (B) and 3, and Chapter 16, Note 2).

7.10.2. Separation procedures using filtration

This is a convenient and widely used technique for the separation of insoluble matter in a liquid sample. A paper, glass or membrane filter may be used, according to the kind of insoluble matter in the sample. The sample is passed through the filter and the insoluble matter is trapped in the filter. The insoluble matter is washed with a solvent or solution in which it is not soluble. The residue is then dried and analysed. The filtrate is also collected and used for further analysis.

7.10.3. Separation procedures using distillation

This separation technique is often used for samples which consist of more than two constituents having different boiling points. For example, a volatile component in the sample may be removed by first heating on/in a water bath in a fume hood (allowing most of the volatile matter to evaporate) and then heating in an electric drying oven/vacuum drying oven to obtain a dry residue. (see heading 27.07; ASTM D 86, 850 and 938; and ISO 759, 918, 3251, etc.)

(Distillation apparatus and separators)

These are used for distillation and the separation of components in samples. It is necessary first to check all glassware for any cracks or chips. If any are found, the glassware should be disposed of. When a flammable or volatile sample is analysed, the sample should be heated with a water bath or an electric heater in a fume cupboard and not directly with a gas burner or other open flame. Do not forget to ensure that water flows freely through the reflux condenser. When distillation is finished, the temperature of the sample residue should be lowered slowly at room temperature, and the apparatus and separators should be cleaned with an appropriate solvent.

7.10.4. Separation procedures using solvent extraction method

These techniques are based on the difference in solubility of two compounds in a solvent.

The mixture is dissolved in a solvent and filtered. The insoluble product recovered on the filter is dried and the solute is evaporated to dryness.

The soxhlet system makes it possible to extract completely from a mixture the components that are hot-soluble in a volatile solvent : the solvent, placed in a flask, is volatilized then condensed in a thimble containing the mixture; when part of that mixture has been dissolved, the solvent returns to the initial flask where it is once again volatilized. The dissolved components, which are non-volatile, are concentrated in the flask until completely extracted.

The Kumagawa system, which operates on similar lines, is also widely used.

7.10.5. Separation procedures using centrifugation

This technique is very useful for separating the precipitate from a "muddy" sample solution. The sample is poured into a container for a centrifuge, and water is poured into another container to equal the sample container in weight to ensure equilibrium in the centrifuge. The precipitate is "concentrated" at one end of the sample container by centrifuge force, e.g., at 2000 rpm for 10 minutes. The precipitate is allowed to settle and physically separated from the solution by decantation or filtration. Sample solutions containing volatile matter require the use of a sample container with a cap.

7.10.6. Separation procedures by means of chromatography

Chromatography is a very convenient separation technique for complex samples and is based on the difference of distribution of each component between two phases, i.e., the "stationary phase" (solid or liquid) and the "mobile phase" (liquid or gas). A sample placed at the beginning of the stationary phase, is "developed" by the mobile phase which flows through the stationary phase and each component is separated according to its characteristic tendency to be "retained" by the stationary phase. This procedure is explained in detail under section 7.11 (Chromatography).

7.10.7. Other

The precipitation procedure, electrolysis, ignition to ash, etc., are also used for the separation of samples in Customs laboratories.

7.11. Chromatography

7.11.1. Column chromatography

This technique is inexpensive and widely applicable to the analysis of organic preparations in Customs laboratories. If a high-performance liquid chromatograph (HPLC) (7.11.5) is available in the laboratory, column chromatography would be less used. However, this technique remains useful and worthwhile (e.g., for preparative-type analysis, etc.).

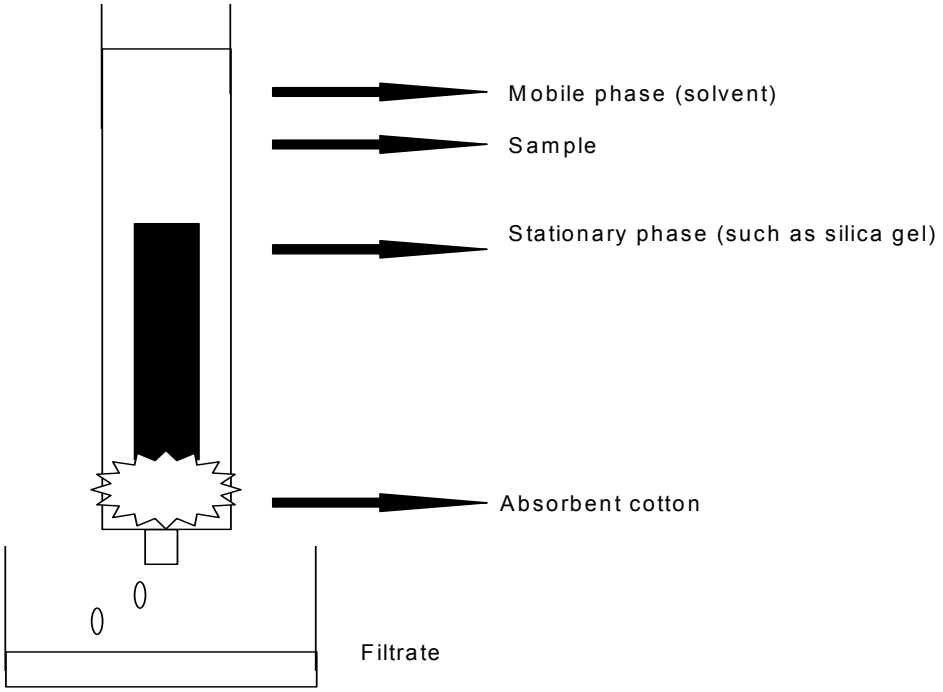


Fig. 2. Diagram of column chromatography

A diagram of column chromatography is shown in Fig. 2. A sample is placed at the top of the column which is packed with the small uniformly-sized particles of polar adsorbents such as silica gel or alumina (stationary phase); absorbent cotton wadding is placed at the end of the column. Successive elutions take place with solvents (mobile phase) of gradually increasing polarity. Each component in the sample is developed slowly downward according to its polarity. Non-polar materials, e.g., hydrocarbons, are initially eluted with non-polar solvents, e.g., n-hexane. More polar materials are retained on the column until a sufficiently high polar solvent passes through the column. The eluent is fractionated and dried. Dry residues are weighed and analysed to identify the contents and composition of the sample. The analytical conditions, e.g., the column (diameter and length), the adsorbent, the sample weight, the kind of eluent and the detection method should be noted.

Application : E.g., separation of petroleum oils from lubricating preparations; separation of each component in surface-active preparations; separation of mixtures of chemicals.

7.11.2. Paper chromatography (PC)

This is an economical and qualitative method for examining a mixture and establishing the number of components. This method is less costly than the TLC method (7.11.3), though it is time consuming and less sensitive than TLC.

A diagram of PC is shown in figure 3. The stationary phase is a piece of filter paper. A 0.1 - 1 g sample is dissolved in 10 ml volatile solvent. About 1 - 5 μl of the sample solution is spotted on a paper plate by means of a capillary tube and the solvent is allowed to evaporate. Each spot must be at least 2 cm away from the next one and at least 2 cm from the bottom of the plate. It is recommended that location of the origin of spots be marked on the plate in advance with a pencil. The

plate is then suspended in a chromatographic chamber, the bottom 1.0 to 15 cm (below the line of sample spots), solvent (mobile phase). Each spot is "developed" as the solvent front migrates upward (by capillary action) through the plate, reaching a height of 15 - 20 cm on the plate. The plate is then removed from the chromatographic chamber and dried thoroughly in a fume cupboard. The movement of the spots is generally detected by exposing the plate to ultraviolet (UV) light or iodine vapour or by spraying the plate with colouring agents.

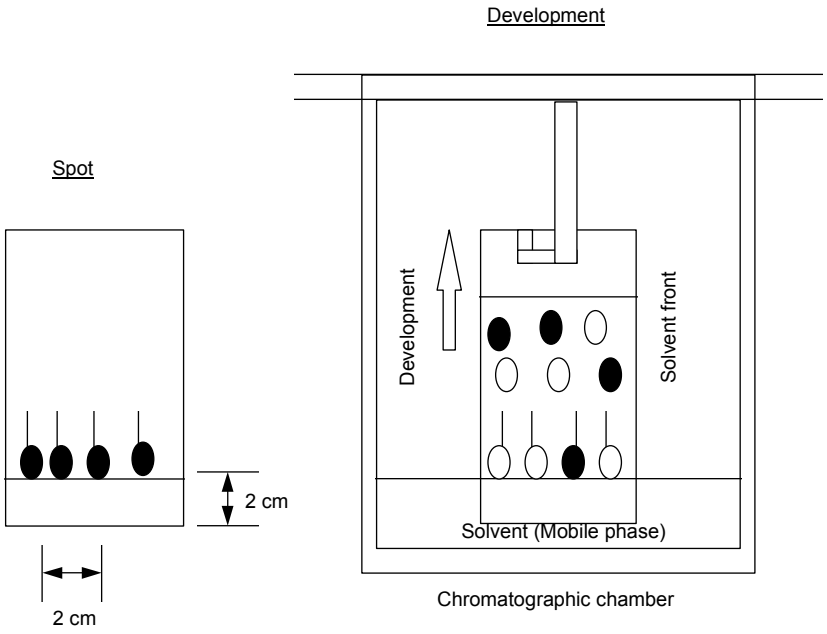


Fig. 3. Paper chromatography procedure

The Rf value (distance the sample or its components has travelled from the origin divided by the distance the solvent front has travelled) of each component in the sample is compared with the values of standard substances. Apparent identification is made by comparing the Rf value with the standard substances. Sometimes each spot is cut from the plate, extracted with an appropriate solvent, and analysed to identify the chemical structure. The analytical conditions, e.g., the system, the plate, the mobile phase, the detection method and the spot volume, should be noted.

Application : Separation of e.g., saccharides, organic acids and amino acids in food preparations; mixtures of chemicals.

7.11.3. Thin-layer chromatography (TLC)

This is an excellent, inexpensive and qualitative technique for rapidly examining mixture samples and achieving good separation. High-performance thin-layer chromatography (HPTLC) is also employed in Customs laboratories. The HPTLC plate enables more rapid and more accurate determination than normal TLC.

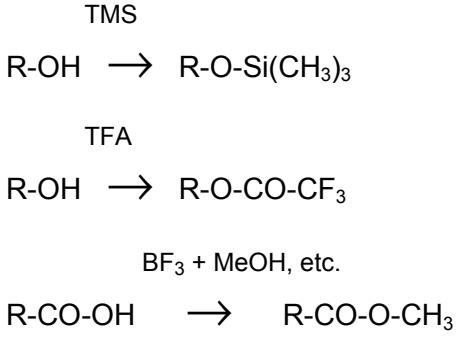
The stationary phase is a thin layer of adsorbent material supported on a glass or plastic plate. Sometimes, this plate is dried in an oven to make it active. A 0.05 - 0.5 g sample is dissolved in 10 ml volatile solvent. About 1 μl of the sample solution is spotted on the plate by means of a capillary tube, and the solvent is allowed to evaporate. Each spot must be at least 1 cm away from the next one and 2 cm away from the bottom. It is recommended that the location of the origin points of spots be marked on the plate in advance with a pencil. The plate is placed in a chromatographic chamber containing a suitable developing solvent (mobile phase); and each spot is then developed until the solvent front reaches 10 or 15 cm. For successful separation, it is essential to select an appropriate mobile phase. The plate is removed from the chromatographic chamber and dried thoroughly in a fume cupboard. The spots are generally detected by exposing the plate to ultraviolet (UV) light or iodine vapour, or by spraying the plate with colouring agents.

The Rf values (see 7.11.2 above) of the spots are calculated and compared with the Rf values of standard substances. Apparent identification is made by comparing the Rf value with the standard substances. Individual spots may be extracted with an appropriate solvent and analysed to identify the chemical structure. Standard thin-layer plates or sheets may be commercially available. The analytical conditions, e.g., the plate, the spot volume, the mobile phase and the detection method, should be noted.

Application : Separation of e.g., saccharides, organic acids and amino acids in food preparations; mixtures of chemicals; drugs.

7.11.4. Gas chromatography (GC)

GC is an excellent method for determining volatile mixtures qualitatively and quantitatively, and is an essential requirement for Customs laboratories. GC is better than any other chromatography system in terms of analytical speed, sensitivity and reproducibility. For non-volatile and unstable substances, trimethylsilicification (TMS), acetylation (e.g., trifluoroacetic acid(TFA)) and methyl esterification are employed.



The gas chromatograph instrument is equipped with a carrier gas (N_2 or He) cylinder and its line, a hydrogen gas cylinder (for FID detector) and its line, an air compressor and its line, columns and a recorder (integrator) to calculate the retention time (Rt) and peak area.

The nature of the components in a sample (e.g., boiling points, whether non-volatile components such as non-volatile acid, alkali or oxidizing agent, or reactive components are included, etc.) should be investigated in advance and an appropriate column should be selected according to the nature of the sample, in order to ensure a successful experiment. If non-volatile components are injected, the analyst should sweep an injection portion. The sample is generally diluted with a volatile solvent (e.g., diethyl ether, etc.) to an appropriate concentration to keep the column and injection portion free from contamination. The degree of dilution depends on the composition of the sample.

The stationary phase is generally an adsorbent or particles of porous support impregnated with a non-volatile liquid. This phase is packed in a glass or stainless steel column, or maintained/chemically bonded in the inert wall of a capillary column. Before a capillary column is used, its splitter, connector, etc., must be prepared. 0.1 - 5 μl of the test sample or its solution is rapidly injected into the injection port with a micro-syringe, through a rubber septum. The injection port is adequately heated to convert the sample or its solution to the vapour state. The vapour state components are introduced by carrier gas (mobile phase) to the column. Each component is developed and separated by its distribution between the gas and liquid (solid) phases through the column, in the same manner as the foregoing chromatographic processes. Eventually, each component reaches the detection portion separately and is automatically detected and recorded.

For GC detectors, a flame ionization detector (FID) is widely used in Customs laboratories, though a thermal conductivity detector (TCD) and an electron capture detector (ECD) may also be used.

The time between the injection and the detection of each peak is called the "retention time (Rt)". A comparison of Rt between a standard substance and an unknown substance may make it possible to identify the unknown peaks.

For quantitative analysis, the internal standard procedure is widely used in Customs laboratories. An internal standard (IS) substance should be pure and its structure must resemble that of the component to be determined. It must be possible to separate the peak of the IS from all peaks of the sample sufficiently. The sample and the standard substances are dried, if necessary. A variety of standard solutions, which include a constant amount of IS, are prepared where the object component in the sample is present in concentrations varying from 0 % to about 10 %. The test sample solutions are prepared in the same way as the standard solutions. A constant volume of the standard solution and of the test sample solution is injected rapidly into the GC, under the same conditions. A plot of peak area ratio (standard substance/IS) versus weight ratio (standard substance/IS) shows a good correlation. An example of the calibration curve is shown in figure 4. Since the peak area is roughly proportional to the concentration, calibration curves obtained from the standard solutions are linear with excellent correlation factors. The concentration of the object component in the sample is calculated from a calibration curve.

The analytical conditions, e.g., the system, the column (name, diameter and length), the mobile phase, the gas flow (split ratio for the capillary column), the temperature programme, the detection method, the injection volume should be noted.

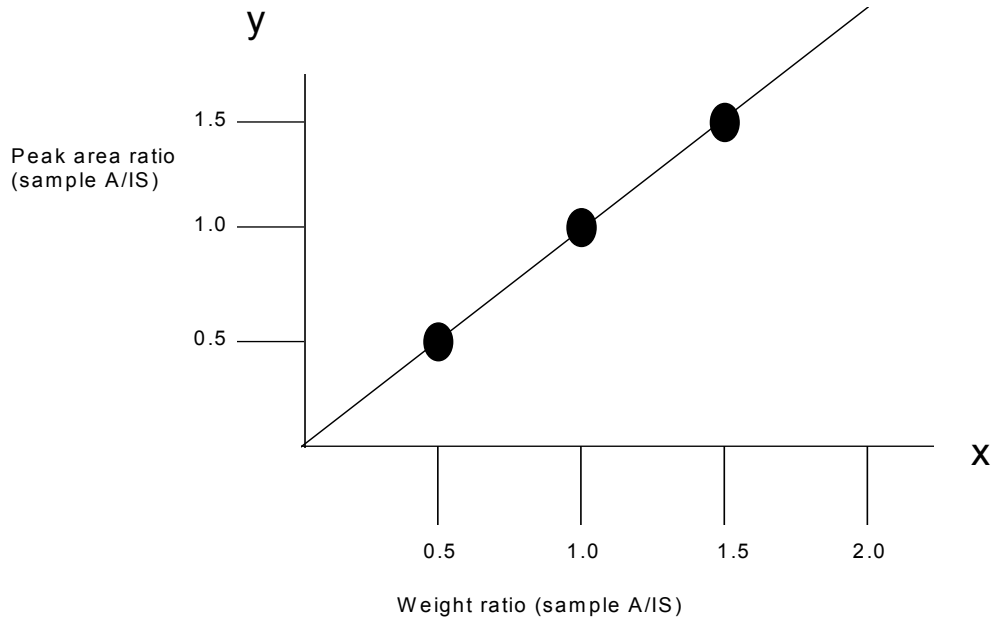


Fig. 4. A calibration curve for peak area ratio (sample A/IS) versus weight ratio (sample A/IS)

Application : Qualitative and quantitative analysis of e.g., fats and butters, constituting fatty acids of fats and butters (methyl esterification), aromatic components in foods, beverages and spirits, alcohols, saccharide (TMS), vegetable oils, wax, theobromine in cocoa, petroleum oils, volatile chemicals, medicaments, essential oils, perfumery, sorbitol (derivative), fungicides, oligomers, drugs.

7.11.5. High-performance liquid chromatography (HPLC)

HPLC is an excellent method for determining organic or inorganic mixtures qualitatively and quantitatively, particularly non-volatile and unstable mixtures which cannot be analysed by GC. In many Customs laboratories, this technique is employed instead of volumetric methods for the quantitative analysis of saccharides.

A high-performance liquid chromatograph instrument is equipped with a pump to provide high pressure (0.1 - 9.9 ml/min, approximately 400 pascal), columns (with pre-column), and a detector and recorder (integrator) to calculate the retention time and area of detected peaks.

The nature of components in a sample (e.g., polarity, etc.) should be investigated in advance and an appropriate column should be selected according to the kind of sample, to ensure a successful experiment. The sample is generally diluted with mobile phase to an appropriate concentration to gain a good separation and to keep the column free from contamination. The degree of dilution depends on the composition of the sample.

There are generally two types of stationary phase: a normal phase and a reversed phase. In the case of the former, less polar mobile phase is used. For the latter, more polar mobile phase is employed. A column packing with chemically-bonded stationary phase is commercially available, ensures high pressure and high-speed conditions and avoids removal of the stationary phase. These phases are packed in a straight stainless steel column. A test sample solution is prepared as 0.1 - 10 % solution in the mobile phase to be used. All solvents should be HPLC

grade. All mobile phases and the test sample solutions must be filtered through 0.2 - 0.45 micron filters (e.g., membrane filter) before use. If an aqueous mobile phase is employed, it is necessary to carry out the de-air procedure of the mobile phase with a supersonic instrument or under vacuum conditions. 0.5 - 10 μl of the sample solution is generally injected through the external sample loop into the column. An automatic injection system is convenient for injecting a constant volume of the test sample and standard solutions. Each component is developed and separated in the column in the same way as the foregoing chromatographic process. Eventually, each component reaches the detection portion separately and is automatically detected and recorded.

For HPLC, an ultraviolet (UV) detector is widely used in Customs laboratories, and a differential refractometer (RI) detector, a fluorescent detector and an amperometric detector may also be employed. A more recent development is the diode-array detector (DAD) which gives a high degree of sensitive and selective detection.

The time between the injection and the detection of each peak is called the "retention time (Rt)". Apparent identification is made by comparing the Rt of the sample with that of the standard substance.

As the peak area is roughly proportional to the concentration, the analyst can determine the amount of substance quantitatively, in the same way as with GC. The analytical conditions, e.g., the system, the column (name, diameter and length), the mobile phase, the flow rate, the detection method, the injection volume, should be noted.

Application : Qualitative and quantitative analysis of e.g., fats, fatty acids, amino acids, saccharide, vegetable oils, theobromine in cocoa, vitamins, hormones, proteins, peptides, steroids, antibiotics, organic chemicals, medicaments, sorbitol, fungicides, oligomers, drugs.

7.11.6. Size exclusion chromatography (SEC)

This is an excellent technique for qualitative and quantitative analysis of a sample which contains components of different molecular size (e.g., polymers, oligomers, etc.). The average molecular weight of the sample can be estimated using this technique. The prepared fractions (preparative-type system) are of a sufficient quality to be utilized for the following analysis.

This system is also called "gel permeation chromatography (GPC)" and is similar to HPLC mentioned above.

The stationary phase is a molecular sieve which has different sizes of pores formed by the cross-linking of polymer chains. The stationary phase is packed in a straight stainless steel column and a few columns are connected in series, and then used for determination. The distribution of each component depends on its size and shape. All solvents should be HPLC grade. A sample is dissolved at about 5 % solution in the mobile phase. The test sample solution must be filtered before injection. Several ml of the test sample solution are injected through the external sample loop onto the column. Immediately following the injection, the external sample loop should be cleaned with the mobile phase. Each component is developed and separated according to its size and shape. Eventually, each

component reaches the detection portion separately and is automatically detected and recorded.

For SEC (GPC) detectors, an ultraviolet (UV) detector connected in series with a differential refractometer (RI) detector is widely used in Customs laboratories.

The time between the injection and the detection of each peak is called the "retention time (Rt)". Since log(molecular weight (M.W.)) values generally vary inversely to the time of elution, a comparison of the Rts of an unknown substance and a standard substance, the average molecular weight of which is already known, makes it possible to estimate the average molecular weight of the sample.

A fraction collector automatically fractionates the elution at regular intervals. The dry matter of each fraction is weighed and analysed to clarify the content of the sample and the composition.

Application : e.g., synthetic polymers and oligomers, hormones, proteins, peptides, steroids, polysaccharide, nucleic acids, organic chemicals, etc.

7.11.7. Other

Ion-exchange chromatography, super-critical-fluid chromatography and counter-current chromatography are also widely available.

7.12. Optical and spectrophotometric analysis

7.12.1. Emission spectrophotometry

Emission spectrophotometry is an excellent technique for qualitative and quantitative analysis of elements in samples, especially inorganic samples. With this technique, the existence and intensity of the characteristic and essential spectra lines derived from each element are determined.

7.12.1.1. Emission spectrophotometry

This method has many advantages in qualitative analysis, since the analyst can determine many elements in a sample simultaneously in a short time and with good accuracy. The cost of the analysis is reasonable. This method is also used for quantitative analysis. However, analysts need to be very careful when preparing the sample.

A small amount of sample, generally solid, is placed in a graphite electrode, subjected to energy by an electron arc or spark and is vaporized. Each element in the sample is raised to an excited energy level. When it drops back to the ground state, photons with energy equal to electronic transitions are emitted. They are separated by a prism or grating, and an atomic spectrum arranged in order of wavelength is produced. A photographic film or plate is generally used as a detector. By measuring the existence and intensity of the characteristic and essential spectra lines derived from each element, the analyst can identify the kinds of elements in the sample. Iron is generally used as the standard substance.

Application : e.g., qualitative analysis of elements in a sample, especially an inorganic sample.

7.12.1.2. Plasma emission spectrometry (inductively coupled plasma (ICP))

This technique is extremely good for quantitative analysis of inorganic compounds. Test sample solutions are easily prepared. This technique exhibits low concentration detection limits and is extremely sensitive in respect of many elements. ICP analysis can be used to detect more than 70 % of elements, including boron, silicon, titanium and phosphorous. Through the use of high-temperature plasma, much chemical interference is eliminated. The dynamic range is extremely broad (i.e., four to six orders of magnitude). It enables quantitative *multi-element analysis under the same conditions*, which is a major advantage over AAS (7.12.2).

Most advanced Customs laboratories employ this technique, since it is more accurate, simple and less time consuming than volumetric and electrolytic analyses.

Samples are dissolved in inorganic acid (or acid solution) and the solution is transferred to a volumetric flask and diluted to an appropriate concentration. A variety of standard solutions of pure metals for making a calibration curve are prepared when the object component in the sample is present in concentrations varying from 0 ppm to about 30 ppm. The standard may be prepared solutions by an analyst or are commercially available as 1000-ppm standard solutions.

The concentration depends on the element to be analysed. The matrices should be added to these solutions to match those of the test sample solutions. These solutions are prepared in the same way as the test sample solutions.

The test sample solutions and the standard solutions are introduced to the inductively coupled plasma (ICP), which is argon plasma, through a spray chamber nebulizer at a constant flow rate under the same conditions. Excited elements in this plasma emit characteristic photons with energy equal that resulting from the transition back to the ground state. They are separated by a grating or monochromator, and each atomic spectrum is determined by a photomultiplier tube manually set, by the analyst, at the wavelengths for specific elements.

By measuring the intensity of each spectrum, a calibration curve is initially prepared for each element. These calibration curves are linear with excellent correlation factors, and the concentration of elements in the test sample solution can therefore be calculated with good accuracy.

Application : Qualitative and quantitative analysis of many elements in samples, especially inorganic samples (e.g., quantitative analysis of low levels of tungsten and vanadium in steel).

7.12.2. Atomic absorption spectrophotometry (AAS)

This technique is well-suited to quantitative analysis. It is easy to prepare the test sample. This technique exhibits low concentration detection limits and is extremely sensitive in respect of many elements. Through *the use of a different radiation source for each element*, much chemical interference is eliminated, but the fact that the radiation source has to be changed frequently is a major disadvantage of this technique.

Test sample solutions and standard solutions are prepared using the same procedure as for ICP. These solutions are introduced to the flame through a burner and converted to atomic vapour under the same conditions. Though some are thermally excited by the flame, other residual atoms in the ground state absorb characteristic radiation for each element; the source of this radiation is a hollow-cathode lamp. The intensity of absorption is determined by a photomultiplier through a grating. A calibration curve is obtained from standard solutions, and the concentration of element in the sample can then be calculated.

Application : Quantitative analysis of many elements in samples, especially inorganic samples.

7.12.3. Absorption spectrophotometry

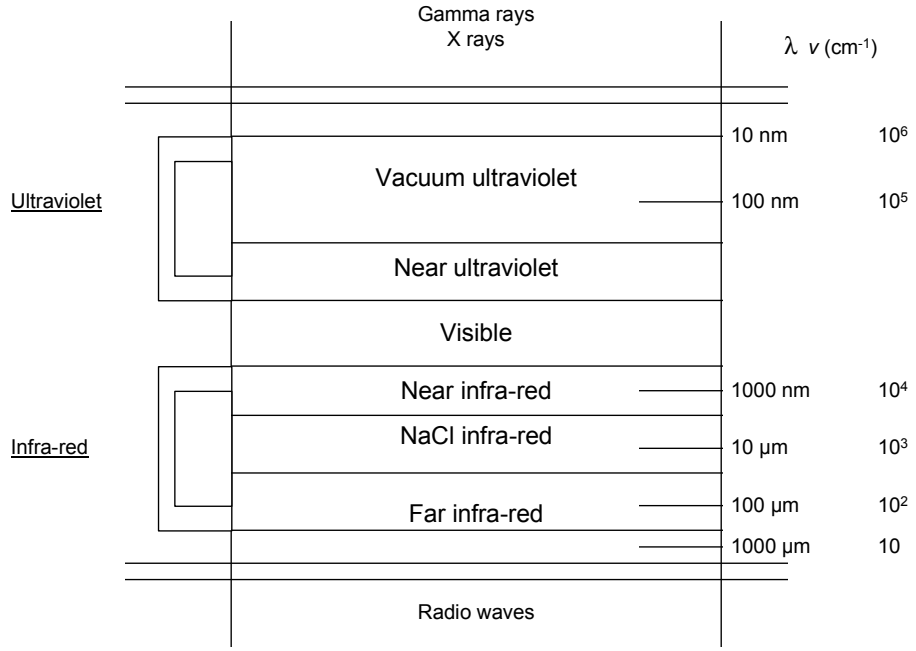


Fig. 6. Electromagnetic spectrum

The energy of the electromagnetic radiation E is related to the frequency ν (s^{-1}) or wavelength λ (cm) as follows :

$$E = h\nu = \frac{hc'}{\lambda}$$

h : Plank's constant, 6.62×10^{-27} erg/s
c': Velocity of light (3×10^{10} cm/s)

The Beer-Lambert law is described as follows :

$$A = -\log T = -\log \frac{I}{I_0} = \varepsilon\, l\, c$$

A : Absorbance
T : Transmittance
I : Power of transmitted radiation
I_0 : Power of incident radiation

ε : Molar absorptivity
l : Path length
c : Concentration of sample solution

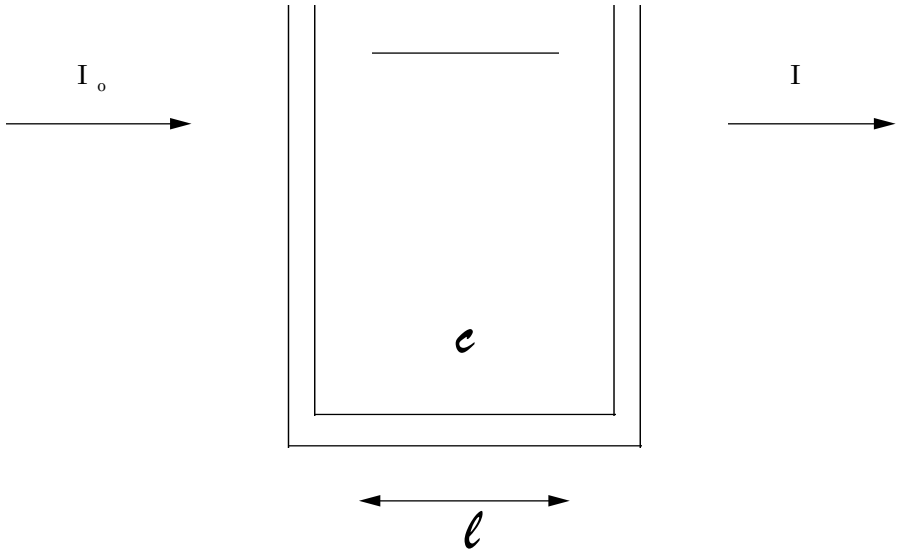


Fig. 7. Absorption of radiation

7.12.3.1. Ultraviolet (UV) and visible spectrophotometry

This technique is widely used for quantitative analysis, since the relationship between the intensity of the absorption and the concentration of the sample follows the Beer-Lambert law accurately. This technique is also used to identify the sample by comparing the spectra of the sample and the reference/standard.

To analyse a sample by determining its UV and visible spectra is called "UV and visible spectrophotometry". The absorption of radiation energy in the UV and visible region causes electron transition of molecules from the ground state to the excited state. In fact, one of the primary uses of UV and visible spectrophotometry is to determine the extent of conjugation within the molecule. The intensity of the absorption varies according to the wavelength. Each molecule has a characteristic UV and visible spectrum.

A deuterium discharge lamp (D_2 lamp), which covers the UV region from 168 nm to 400 nm, and a tungsten filament incandescent lamp (W lamp), which covers the visible region from 320 nm to 3,000 nm, are generally used as sources. The analyst should select the source on the basis of the region of spectrum to be determined. After stabilization of the source, the determination should be started.

The polychromatic radiation from the source is separated to each wavelength radiation through a monochromator, such as a grating.

A sample cell made from quartz, in the form of a square cuvette (e.g., 1 cm thick), is widely used as a sample container. The analyst should select an appropriate solvent which has little absorption in the wavelength region to be determined. More than two cells should be prepared, one for reference and the others for a test sample and standard solutions. Before the determination, background corrections of the cells should be carried out. The solvent is added to

one cell as reference and the test sample solution or standard solutions for a calibration curve are added to the other cells. If necessary, a centrifugation separation procedure should be carried out, in advance, for the test sample solution and standard solutions. The relationship between appropriate concentration and the value of ε is shown in Table 1.

Table 1 Appropriate concentration of test sample solution on the basis of ε value

ε	c (mole/l)	A (absorbance)
10^1-10^2	10^{-2}	0.1 - 1.0
10^2-10^3	10^{-3}	0.1 - 1.0
10^3-10^4	10^{-3}	0.1 - 1.0

If the solvent tends to be volatile, the analyst should use a cell with a cap. The analyst should initially wash cells with the solvent and then with the test sample solution; finally the test sample solution is poured into the cell (but not up to the top) and the outside of the cell should be carefully wiped with a piece of clean tissue paper. Hold the sides, which UV and visible light does not pass through, and keep the transmittance sides of the cell clean. After use, these cells should be carefully washed with the solvent and stored in a wide-mouthed reagent bottle to which sufficient alcohol or water has been added.

(Double-beam technique)

This technique is generally used for determining UV and visible spectra. An electronic recorder is an indispensable tool in this device. After proofreading of the wavelength, adjustment of the indicator and background corrections of the cells, the reference cell and sample cell are inserted in the sample rooms respectively. The test sample solution is scanned in the region to be determined. Any difference of the transmittance between the reference and sample is detected by a detector tube (phototube or photomultitube) in each wavelength and the spectrum is simultaneously recorded.

(Single-beam technique)

This technique is capable of greater precision and is employed for quantitative analysis. The procedure is similar to that of the double-beam technique. A reference cell is initially inserted in a sample room and transmittance or absorbance is set to 100 (%T) or 0 (A). A sample cell is subsequently inserted in the sample room in place of the reference cell and the transmittance or absorbance value is determined individually; the value should be noted by the analyst.

Application : Qualitative and quantitative analysis of samples including molecules which have paired non-bonding outer-shell electrons, such as nitrogen, oxygen and sulphur, or having electrons in the π orbital (e.g., double bonds, triple bonds and aromatic rings, etc.).

7.12.3.2. Infra-red (IR) spectrophotometry

IR spectrophotometry is based on the study of the interactions between a substance and electromagnetic radiation with a wavelength of between 2.5 and 25 μm.

The energy is insufficient to modify the electronic energy of the molecule; the excitations observed will be confined to vibrations and rotations.

The absorption spectrum obtained represents the transmittance (proportion of light transmitted through the sample) in terms of the wave number.

Certain vibration frequencies may undoubtedly be associated with the presence in the molecule of certain well-defined groups. The IR spectrum provides a real "identity card" for the molecule. The region 1,500 cm^{-1} to 400 cm^{-1}, which is particularly rich in information is known as the molecule's fingerprint.

Spectra libraries have been set up and make it possible automatically to identify pure compounds.

7.12.3.2.1. Quantitative analysis :

The intensity of absorption is linked to the intrinsic properties of the molecule and to the number of molecules present in the beam. This enables quantitative analysis applications by applying the Beer-Lambert law.

7.12.3.2.2. Instruments

There are two types of IR spectrometers : the dispersion spectrometer and the Fourier transform spectrometer, which uses optical interference.

The operating range is between 4000 cm^{-1} and 400 cm^{-1}.

7.12.3.2.3. Technique for the preparation of samples :

a. Liquid spectra

Pure liquid :

These can be examined very simply in the form of a capillary film obtained by placing one or two drops between two appropriate plates and introducing these into observation cell.

To make the measurements easier to reproduce and more quantitative, thin cells (0.005 - 0.1 mm) may be used depending on the intensity of absorption of the vibration to be studied.

Spectra of dissolved substances :

In this way, it is possible to examine solid, liquid or gaseous substances, which have to be dissolved in an appropriate solvent (i.e., one which has a low level of absorbency and is within the region limits of the IR field under examination).

The most widely used solvents are : CCl_4, CS_2, C_6H_{12}, $CHCl_3$. They must be completely anhydrous.

The double-beam technique can be used to compensate the absorption of the solvent : for this, a cell of the same thickness as used for the sample beam

has to be placed in the reference beam; it is filled with pure solvent. In this way, only the bands attributable to the solute are visible. However, examination is not possible in regions of high absorption of the solvent. Indeed, all the energy being absorbed, no light will come either from the sample beam or from the reference beam, and the apparatus will merely plot a straight line.

The examination of aqueous solutions poses many different problems :

- the constituent material of the plates which must be sufficiently transparent to IR and also be water-resistant (AgCl, CaF_2);

- very high absorption of water in a wide area.

b. Gas spectrum

A sample is introduced in a gas cell which should be capped at both ends by discs of substances that are transparent to IR rays (e.g. NaCl).

c. Solid spectra

Examination by transmission

(Examination in the form of a film)

The methods of obtaining a suitable film depend largely on the substance to be examined :

- evaporation of a solution on a disc;

- melting and compression (in a heating press) then solidification by cooling of the film obtained (method often used for polymers).

(Examination in the form of a dispersion)

- Dispersion in a paste

The particles obtained by grinding are dispersed in a substance whose viscosity is such that a paste-like slurry is formed which is observed between two NaCl plates.

For this purpose, the oils are used; Paraffin oil ("Nujol") and fluorocarbon oil (fluorolube).

When examining the spectrum, account has to be taken of the absorption of the oil used.

- Dispersion in a pellet of alkali halide :

This is the commonest method used for solids

The finely ground particles are mixed with KBr powder, also ground to obtain as homogenous a mixture as possible; the whole is rendered transparent by recrystallization under very high pressure (10 tonnes/cm^2). Generally, 1 mg of solute is used in 300 mg of KBr.

Examination by reflection :

There are several techniques for depositing the sample on a reflective surface and establishing the spectrum :

- specular reflection

- diffuse reflection

- attenuated (total) reflection (ATR).

These techniques are useful where very little of the matter is available.

(Analysis of IR spectrum)

It is necessary for Customs laboratories to have standard/reference IR spectral data and publications in order to identify an unknown substance. If standard/reference substances such as pure chemicals, drugs and polymers, are available, the IR spectra should be determined and be stored. It is, of course, desirable for the IR data to be arranged on the basis of the Harmonized System/molecular weight/molecular formula. If the structure of a sample can be assumed from the accompanying certificate and relevant documents, the analyst should compare the sample spectrum with standard/reference spectra of the same or similar substances. In the case of insufficient information from the accompanying certificate, the analyst can identify the functional groups (e.g., benzene ring, alcohol, ether, carbonyl, sulphonate, etc.) empirically. On the basis of these analyses and other data such as appearance, melting point, colouring test, etc., the analyst can analogize the structure of the sample component and compare the sample spectrum with the spectra of similar substances. By comparing both spectra, especially in a fingerprint region, the structure of the sample can be identified. The correlation tables between functional groups and the IR region are set out in many publications. The sources of IR spectral data generally used in Customs laboratories are as follows :

Aldrich Library of IR Spectra : Aldrich Chemical Co.

The Sadtler Handbook of Reference Spectra : Sadtler Research Lab. Inc.

Application : Qualitative analysis of organic and inorganic substances. Quantitative analysis of the composition of e.g., polymers.

7.12.4. Other

The microscope Fourier transform infra-red detector, the raman spectrophotometer and the spectropolarimeter are generally used in Customs laboratories.

7.13. Electromagnetic analysis

7.13.1. X-ray diffraction analysis

This technique is well-suited to qualitative analysis of inorganic samples, particularly mineral products, on the basis of information with reference to the array of atoms in a crystal, crystallite or grain, as available from an X-ray diffractometer.

When struck by X-ray, a reinforced X-ray intensity is detectable in the vicinity of the crystal, provided that the angle of reflection is equal to the angle of incidence. The following equation is given :

$n\lambda = 2d\sin\theta$
n : Integer 1,2,3 (1 is applied to a powder and multi-crystal sample)
λ : X-ray wavelength
d : The interplanar spacing
θ : The angle of incidence and reflection

(Preparation of sample)

a. Powder and mass sample

Such samples are placed in a mortar and ground to extremely small particles by a vigorous back-and-forward motion of a pestle across the mortar. Strong hand pressure and diligence in grinding at this point are essential for preparing small particles, since insufficient guiding of the sample causes line broadening. The sample powder is hand-pressed to an aluminium plate or a glass sample plate.

b. Metal sample

The surface of a metal sample is smoothed and flattened by polishing; it is then cut to an appropriate size to fit an aluminium plate.

(Analysis of data)

Data from the X-ray diffractometer scan for a sample are compared to standard patterns. The following reference source is widely used in Customs laboratories :

The Joint Committee for Powder Diffraction Standards (JCPDS) Powder Diffraction File

The matching of line positions and relative intensities between the sample and a standard pattern makes it possible to identify the sample. In the case of mixtures, overlapping X-ray diffractometer scans are observed, except where there is formation of a solid solution.

Application : Qualitative analysis of mineral products, ores, slags, inorganic chemicals, textiles, metals, fertilizers, etc.

7.13.2. Mass spectrometry (MS)

This is one of the most useful techniques for identifying molecular structure, especially molecular weight, in Customs laboratories. Since this technique is very sensitive, very small amounts of sample can be employed to identify the molecular structure. It can also be incorporated with a gas chromatograph (GC-MS). GC-MS is one of the most useful instruments for identifying each component in a complex sample, drugs, etc.; however it is expensive and the maintenance is difficult. GC-

MS equipped with a library data system is recommended, since that makes it possible to identify each peak in a sample automatically and very quickly.

In high-vacuum conditions, volatilized molecules in a sample are bombarded by high energy, e.g., a beam of electrons (usually 70 eV; EI method), to form molecular ions (radical cations/anions), some of which are then fragmented into smaller ions or radical ions (fragment ions). These ions are then separated according to their mass-to-charge ratio (m/z) by means of electronic and magnetic fields (e.g., a high-resolution double-focusing mass spectrometer), and detected in proportion to their relative abundance. A quadrupole mass analyser is also commercially available at a reasonable cost. Observed data (mass spectrum) are displayed or plotted as relative abundance versus mass-to-charge ratio. These fragmentation patterns provide detailed information with reference to molecular structure.

(Ionization method)

The electron impact (EI) method is widely used. The chemical ionization (CI) method, field ionization (FI) method, secondary ion mass spectrometry (SIMS), etc., are employed to determine the molecular weight of unstable or non-volatile substances.

(Introduction of sample)

a. Direct inlet (DI) method

A very small amount of non-volatile liquid or solid sample is placed on the end of a probe and the probe is directly inserted into an ionization chamber. The sample is vaporized and ionized in the ionization chamber.

b. Gas chromatography-mass spectrometry (GC-MS) method

GC-MS uses a mass spectrometer incorporating a gas chromatograph and is one of the most useful methods for identifying each component of a complex sample.

The nature of the components in the sample (e.g., boiling point, whether non-volatile components are included, etc.) should be investigated in advance and, after GC analysis, a GC-MS analysis should be carried out in the same conditions. A 10 - 100 mg sample is dissolved in 1 ml of a suitable solvent (e.g., diethylether). 0.01 - 1 μl of the solution is injected into a GC with a micro-syringe. Each component reaches the ionization chamber separately and is ionized. Ionized molecule ions and fragment ions are detected in a detection chamber. A total ion chromatogram, which is similar to a gas chromatogram, is initially displayed/plotted and a mass spectrum of each peak can automatically be displayed/plotted.

(Analysis of data)

The highest ion in a mass spectrum generally corresponds to the molecular ion; however, no molecular ion is observed in the case of an unstable molecular ion. Since the fragmentation patterns are usually characteristic of types of compounds, identification is obtained by a comparison of the mass spectrum determined and reference/standard spectrum under the same conditions.

Application : Qualitative analysis of organic samples, such as alcohols, saccharides (TMS), petroleum oils, volatile chemicals, medicaments, essential oils, perfumeries, fungicides, oligomers, especially drugs, and aromatic components in food preparations, beverages and spirits, etc.

7.13.3. Nuclear Magnetic Resonance (NMR) spectrometry

This is one of the major techniques for the study of the molecular structure of organic samples in Customs laboratories. The necessary instrument is very expensive and difficult to maintain; however, NMR is probably the best means of analysis and identification of complex molecular structures.

NMR is employed with atomic nuclei which have angular momentum, e.g., ^{1}H, ^{13}C, ^{31}P, etc. For example, three different types of information, i.e., chemical shift, spin-spin coupling and peak area (quantitative determination), are available from a ^{1}H-NMR spectrum. Since the information is characteristic of the type of molecular structure, NMR is an excellent analytical technique to identify the structure of organic substances.

A deuterated solvent (e.g., deuterated chloroform or deuterated water) is generally employed to dissolve the sample as an external lock for the NMR signal. Tetramethylsilane (TMS) is widely used as reference for comparing chemical shift; its chemical shift is defined as zero. A 1-5 % solution of the sample is placed in a sample tube (e.g., 5 mm in diameter) and the sample tube is placed in a magnetic field; the NMR spectra can then be determined.

Application : Qualitative and quantitative analysis of saccharides, petroleum oils, organic chemicals, medicaments, fungicides, oligomers, polymers, drugs, etc.

7.13.4. Electron microscope analysis

Electron microscopes have a wide range of magnification (20 X to 100,000 X) and provide information such as particle size, shape and texture of solid materials. Observation of the surface of samples enables analysts to identify the nature of each sample, its processing condition, etc.

There are several types of electron microscope available, e.g., the conventional transmission electron microscope (CTEM), the scanning electron microscope (SEM) and the scanning transmission electron microscope (STEM). Secondary electrons, which are excited by the primary electron beam from a source, are collected and amplified. An image of the sample surface is generated by scanning with the primary electron beam and is displayed on a cathode ray tube (CRT).

A mass sample is cut to a square piece (e.g., 1 cm $\times$ 1 cm), taking care not to damage the surface to be scanned. Other types of sample (e.g., powders, particles or crystals) are mounted on a supported holder, after preparation of carbon or metal evaporated films only for the non-conductive sample. The sample holder is placed on a mounting stage for analysis.

Application : Surface analysis of mineral products, ores, slags, inorganic and organic chemicals, starches, textiles, plastics, raw hides and skins, leathers, furskins, wood articles, precious stones and metals, drugs, etc.

7.14. Other

An elemental analyser (C,H,N,O), a densimeter/pycnometer, a thermal analyser, a surface tension meter (see Chapter 34, Note 3), a melting point analyser, a refractometer, etc., are also employed in Customs laboratories.

8. Preparation of laboratory worksheet and laboratory report

8.1. Records

A bound notebook should be used in the Customs laboratory. All data, calculations and results should be recorded during the actual experiment or later the same day in a manner readily understandable to all.

It is usually recommended that any relevant notes and comments be written in this notebook for further reference.

Laboratory notebooks constitute original data, which are important to keep, especially for cases where the Customs laboratory report is disputed. Reference is made to them on laboratory worksheets and laboratory reports. They must therefore be systematically labelled (e.g., with a code consisting of the laboratory room number or the analyst's abbreviated name and a sequential number). Old laboratory notebooks should be stored in a central location.

8.2. Reporting results

On the basis of experiment data, reference data and calculation results, an accurate, detailed and understandable laboratory worksheet and report should be prepared for each sample analysed, in accordance with sections 8.3. (Laboratory worksheet) and 8.4. (Laboratory report).

Original data sheets with details of the experiment conditions, a photograph, calculations, reference data, etc., should be appended to that report.

8.3. Laboratory worksheet

After completion of the analysis, a laboratory worksheet and a laboratory report should be produced for each sample tested.

The following information should appear on the laboratory worksheet :

(1) Relationship to any previous sample analysed by the laboratory.

(2) Product description (physical appearance, any significant markings or packaging, the preparation of the analytical sample, etc.).

(3) Relevant excerpts of information from the manufacturer or importer.

(4) All analyses performed on the sample data analysis and the results of the analyses, whether positive or negative (e.g., physical properties, chemical name, chemical structure, chemical composition, key components of mixtures, type of polymer, textile or paper, where relevant).

(5) Any other factual information that is relevant to the classification of the goods or to any other question raised.

(6) Additional background information, e.g., encyclopedia references, journal articles.

(7) Comparison with similar products previously analysed by the laboratory.

(8) Interpretation of data, conclusions and a brief rationale for opinions and conclusions.

(9) Reference to any supporting information in texts, encyclopedias, journals, etc.

The laboratory worksheet should be filed in such a way that it can be easily retrieved. This may be facilitated by storing the information on a computer. It is important that all work data are accessible to the reviewer in the event of a question so that the entire sequence of work can be reconstructed later, if necessary. Calculations must also be clear, with all equations stated (results must be reported only to a number of significant digits justified by the accuracy and precision of the method).

Typing errors may occur when transferring data and results to a laboratory worksheet or a laboratory report. Data stored on a computer may accidentally be lost and, if no special precautions are taken, data may easily be altered on a computer without allowing the detection of such changes at a later stage. It is therefore essential to print out all instrumental analyses. Laboratory worksheets and reports should always contain a reference to where the original data can be found.

8.4. Laboratory report

The formal typed laboratory report is the end product of all laboratory work and therefore must be complete and accurate.

The following structure is recommended for laboratory reports :

(1) Name of Customs laboratory

(2) Series number and date of the report

(3) Name of the client

(4) Name and number of the sample

(5) Information related to the need of the Customs officers.

(6) Opinions and conclusion of the technical work performed by the analyst, with explanations.

(7) Signature and title of analysts accepting technical responsibility for the test report and date of issue.

(8) Reference as to where the original data can be found.

9. Quality assurance

9.1 Introduction

One of the main purposes of a Customs laboratory is to produce high-quality analytical data through the use of analytical measurements that are accurate, authorized and adequate for Customs purposes. To achieve this objective, the laboratory's activities must be carried out in a cost-effective manner under a planned and documented quality system.

Insufficient attention to the quality of the work product often causes serious deficiencies in Customs laboratory operations. Controls and checks are necessary to ensure quality. These entail not only a thorough knowledge of the Customs laboratory's purpose and operation, but also a dedication to standards of excellence on the part of the supervisory and analytical staff.

Since Customs laboratory reports may be used before the courts, they must be not only scientifically credible, but also legally defensible. To achieve this, Customs laboratories will find it necessary to operate with a quality assurance system that includes extensive documentation of their activities.

The United States Federal Register's "Good Laboratory Practice Regulations" (21 CFR Part 58), European Standard EN 45001, the OECD's "Principles of Good Laboratory Practice", etc. (see III, Chapter 1 : International standards and methods) would be extremely useful when drafting an implementation plan for a quality assurance system in any laboratory.

9.2 Quality assurance programme

Personnel, laboratory design, management of equipment and supplies, training of staff, safety and antipollution measures and sampling are documented in detail in other draft sections.

9.2.1. Objectives of a quality assurance programme

The main objectives of a quality assurance system in Customs laboratories are as follows :

- To facilitate uniform application of the Harmonized System.
- To facilitate enforcement activities.
- To develop the quality of Customs laboratory practices and operations.
- To establish or identify authorized and reliable methods for Customs purposes.
- To produce reliable and accurate analytical results by using authorized and reliable analytical methods.
- To maintain a continuing assessment of the quality of data reported by analysts.
- To achieve high-quality Customs laboratory performance in a cost effective manner.
- To improve sample and record handling.
- To carry out safety and anti-pollution measurements.
- To carry out safe operating practices.
- To give appropriate training.

9.2.2. Quality assurance committee

A quality assurance committee must develop a quality assurance plan and achieve the objectives of the quality assurance programme. The committee is composed of representatives from the supervisory and analytical staff, preferably with a quality assurance co-ordinator serving as a chairman of the committee.

First and foremost, the committee examines and evaluates laboratory activities in terms of the following quality assurance elements :

- Programme objectives.
- Work planning for quality.
- Organizational structure.
- Policy of management.
- Laboratory design.
- Procurement of laboratory facilities.
- Supplies' management.
- Equipment maintenance.
- Safety and anti-pollution measurements.
- Training of staff.
- Sampling.
- Sample and data handling.
- Personnel practices.
- Customs laboratory operations.
- Standards and methods employed in Customs laboratory (selection and evaluation).
- Reporting procedures.
- Technical literature and reference books.
- Statistical procedures.
- Audit procedures.
- Evaluation of costs and benefits for laboratory performance.
- Improvement of the quality assurance programme.

The committee develops quality assurance requirements on the basis of its findings and it documents quality assurance procedures to meet its requirement. The committee is important for ensuring that laboratory staff are properly involved in the planning and development stages of the programme.

The committee decides on the format of a quality assurance manual and assigns the writing of various sections to competent individuals. Every quality activity, such as policies, organizational objectives, functional activities, etc., is to be documented in this manual. Each section of the manual is discussed and modified, if necessary, by the committee. The latter implements the programme, monitors it and make changes, if necessary, to achieve the quality goals set for the operation of the system. All activities should be documented.

9.2.3. Statistical applications

9.2.3.1. Treatment of scatter

To obtain reliable analytical results, chemists usually divide a sample into several test samples and then analyse each of these; they repeatedly determine the end points of test sample solutions in titration analysis. Some scatter of analytical data based on unavoidable error and essential variation is normally allowed for. Variation of analytical data based on random errors usually shows the normal

distribution around a true value. However, the distribution of analytical data sometimes shifts to one side or the other, due to specific factors such as determinate errors (instrument errors, operational errors, method errors, etc.).

Treatment of analytical data should be carried out on the basis of statistical procedures. For example, the average $\boldsymbol{x}$ of a series of values x_1, x_2,...., x_n, is obtained simply by adding all the individual values and dividing the sum by the number of values n.

$$\boldsymbol{x} = \frac{\Sigma_n x}{n} = \frac{x_1 + x_2 + ... + x_n}{n}$$

It is important to know the degree of scatter and the most widely used measures for determining scatter are as follows :

The sample variance : $V = \Sigma(x_i - \boldsymbol{x})^2 / (n - 1)$

The standard deviation : $\sigma = (V)^{1/2}$

The coefficient of variation : $CV = (\sigma/x) \times 100$ (%)

On the basis of the measurement of scatter, whether certain analytical data fall within a certain distribution (e.g., confidence limit) should be considered from the standpoint of probability.

The precision of a measurement can be improved by increasing the number of observations. When a chemist performs a series of replicate analyses, one of the results sometimes deviates considerably from the others. In that case, a decision has to be taken as to whether the results should be rejected or retained. Statistical application is one useful means to determine whether the scatter of analytical data is based on unavoidable error and essential variation or significant errors in analytical technique.

The significance of σ in relation to the normal distribution curve is shown in Figure 1. The mathematical treatment from which the curve was derived shows that 68 % of the individual deviations fall within one standard deviation from the mean $\boldsymbol{x}$, 95 % are less than twice the standard deviation, and 99 % are less than 2.5 times the standard deviation. Consequently, 68 % of the individual values will fall within the range $\boldsymbol{x} \pm \sigma$, 95 % will fall within $\boldsymbol{x} \pm 2\sigma$ and 99 % will fall within $\boldsymbol{x} \pm 2.5\sigma$. The normal distribution curve assumes no determinate errors, but only random errors. Determinate errors, in practice, shift the normal curve from the true value.

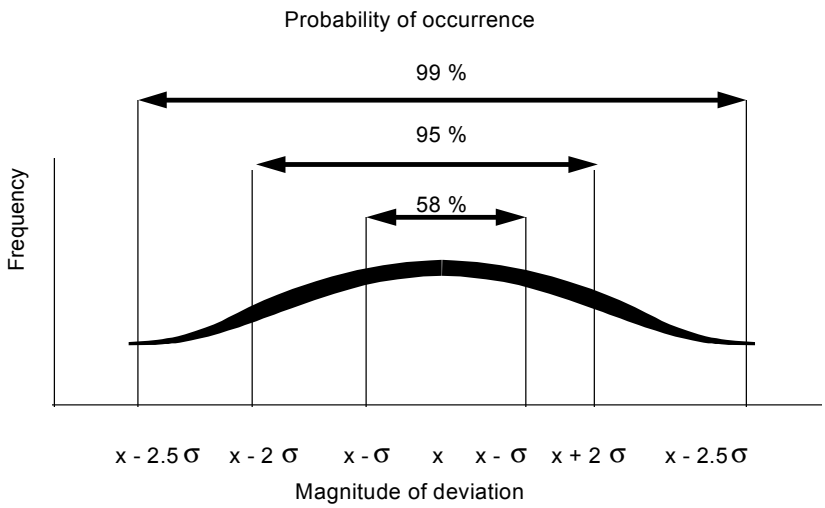


Figure 1 Normal error curve

A control chart is one of the most useful statistical tools in the quality assurance programme and is

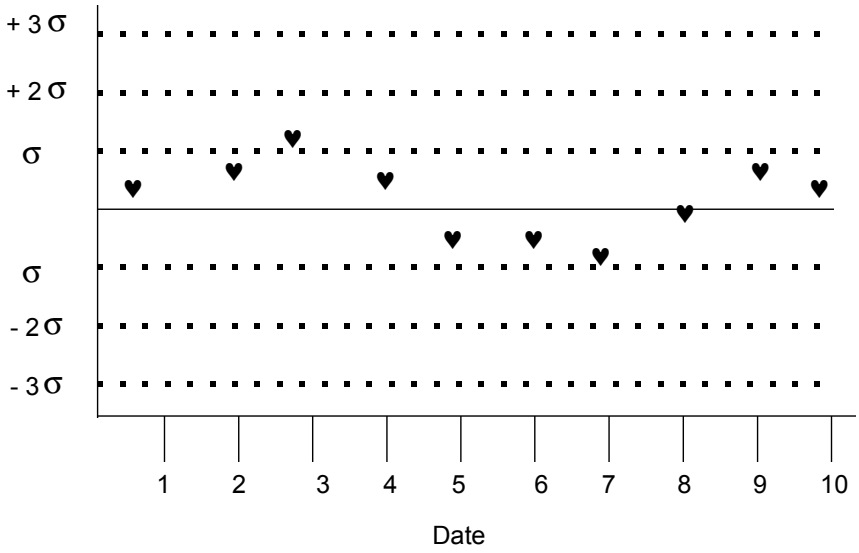


Figure 2 Example of control chart

frequently used for routine analysis. An example of a control chart is shown in Figure 2. The control chart consists of a centre line representing the known and assumed value of the control and one or two pairs of limit lines, the inner and outer control limits.

Day-to-day results of the analysis are plotted on the chart to ascertain whether the analysis is in the range of statistical control.

For a detailed description, professional literature and publications (e.g., ISO Standards 2602, 2854, 3207, 3301, 3494, 3534, etc.) should be consulted.

9.2.3.2. Significant figures

Digit treatment of data corresponds to the purpose of the experiment and the analytical accuracy of the instruments used. For example, chemists can read 10 % of the minimum scale in the case of analog-type instruments.

In a calculation process of addition or subtraction, a standard digit is the maximum one (see Example 1). In the case of multiplication or division, the accuracy of the results is controlled by the most inaccurate digit (see Example 2).

Example 1

$$\begin{array}{r} 207 \\ 20.7 \\ +)\ 12.8 \\ \hline 240.\underline{5} \end{array}$$

Example 2

$$2.3 \times 0.71 = 1.6\underline{33}$$

The figures underlined are indefinite and they are rounded off.

If the digit following the last significant figure is greater than 5, the number is rounded up to the next higher digit. If it is less than 5, the number is rounded down to the present value of the last significant figure :

$240.\underline{3} \Rightarrow 240$
$240.\underline{7} \Rightarrow 241$

If the last digit is a 5, the number is rounded off to the nearest even digit :

$240.\underline{5} \Rightarrow 240$
$241.\underline{5} \Rightarrow 242$
$249.\underline{5} \Rightarrow 248$

However, if there is another digit following the 5, the figure is rounded off as follows :

$240.\underline{50} \Rightarrow 240$
$240.\underline{51} \sim 240.\underline{59} \Rightarrow 241$

9.2.3.3. Handling of analytical data

It is important that analytical data be arranged in such a way as to produce an effective and successful conclusion, which can be widely applied. Tables, graphs and mathematical treatment are effective means of achieving this.

(a) Table

A table indicates a qualitative relationship. Contents are expressed according to size against analytical conditions and the placing of the decimal point is important.

(b) Graph

When chemists set out analytical data in a graph, the relationship between variables becomes apparent. The results can be recorded on section paper, semi-logarithmic graph paper, log-log graph paper, etc. If analytical data give linear plots, chemists can estimate the analytical value of an objective component by extrapolation and intrapolation. On the basis of a slope of calibration curve (a) and intercept of y axis (b), the chemist can give coefficients of the analytical equation. The analytical results correspond to the following equation :

$$y = ax + b$$

(c) Mathematical treatment of analytical data

It is desirable to produce an analytical equation from the analytical results. An analytical value (differential value, integral value, etc.) is directly calculated from the equation and the phenomenon in experiment is accurately expressed in the form of the equation both qualitatively and quantitatively.

The linear equation above is one of functions. The method of least squares is widely known to give the best straight line through a series of analytical results. The best straight line occurs when the sum of the squares of the deviations of the data from the line are minimum. Calculations of *a* and *b* are made as follows :

$$a = \frac{\Sigma(x_i - \boldsymbol{x})(y_i - \boldsymbol{y})}{\Sigma(x_i - \boldsymbol{x})^2} \approx \frac{\Sigma x_i y_i - [(\Sigma x_i y_i)/n]}{\Sigma x_i - [(\Sigma x_i)_2/n]}$$

$$b = \boldsymbol{y} - a\boldsymbol{x}$$

Where **x** is the mean of all values of x_i and **y** is the mean of all values of y_i, and n is the number of the data points.

If the analytical results do not give straight plots, the chemist must find a simple function which fits with the calibration curve of the analytical data.

9.2.4. Quality control of qualitative analysis

Qualitative analysis is as important as quantitative analysis for a Customs laboratory. The key purpose of qualitative analysis is to identify clearly the kind and structures, etc. of the components in a sample.

In parallel with testing or determination of the sample, the analyst usually carries out control and blank tests under the same conditions, to ensure the detection of the target substance. However, there are many substances which show the same colouring reactions (e.g., colouring tests, etc.), Rf values (thin-layer chromatograms

(TLC)), retention times (e.g., gas chromatograms (GC), high-performance liquid chromatograms (HPLC), etc.), spectra (e.g., ultraviolet and visible (UV-VIS), mass, infra-red (IR) spectra, etc.), etc.

Consequently, the analyst usually carries out double or triple checks using other techniques (e.g., other colouring agents, other columns, other mobile phases, etc.).

Attention must also be paid to detection limits and sensitivity. The detection limit is generally defined as the quantity (or concentration) required to give a signal equal to three times the standard deviation of the blank. Sensitivity is also defined as the quantity (or concentration) required to give a certain intensity of colouring reaction, spot, peak, etc. In the case of too diluted a sample solution or an insensitive detection method, the reaction or determination tends to produce a negative result. Consequently, it is important to prepare a sample solution in an appropriate concentration and to use highly sensitive detection methods for each component in the sample.

9.2.5. Sampling

It is extremely important to collect representative samples from many types of shipments at the examination stage and to prepare a test sample for analysis in the Customs laboratory with the minimum composition change, as described in the section on "Sampling".

In order to accomplish these procedures safely and consistently, a sampling plan should be prepared, developed and documented for the various types of goods traded, covering delivery of the samples to the Customs laboratory and production of samples for analysis as well as sample storage and disposal. Laboratory staff should be included in the discussions.

To ensure correct sampling and sample preparation and to avoid unnecessary repetitive analyses, good communication is necessary between the clearance or field officers and the laboratory staff.

9.2.6. Audit procedure

A Customs laboratory should include performance and system audits in its quality assurance programme.

The performance audit is to examine and evaluate the overall analytical activities of the analysts; it should include worksheet or notebook review, field analyst work review, the duplication of sample analysis, and intra- and inter-laboratory comparisons.

The system audit is a field inspection or assessment of the laboratory's control system and should be well-planned and conducted by a trained audit team in order to enhance the quality of the laboratory's activities.

Qualified persons, who are familiar with the quality assurance programme and are skilled scientists, should be selected and trained as auditors.

The following items should be examined and evaluated by the audit team :

- Performance of quality assurance activities.

- Confirmation of reliability of analytical methods.
- Sample analysis and analytical reporting procedures.
- Safety and anti-pollution measurement.
- Sample and data handling.
- Management and procurement of laboratory facilities and supplies.
- Staff training and evaluation, etc.

After the audit has been completed, provisional recommendations to enhance the quality of the laboratory's activities are made by the audit team to the assigned staff and the laboratory director. The provisional recommendations should be discussed at a meeting to avoid any problems or misunderstanding.

The audit report should point out imperfections in laboratory activities and recommend corrections (e.g., changes in laboratory procedures, improvement or purchase of laboratory facilities, need for staff training, etc.).

A draft report should be prepared as soon as possible and be submitted to the laboratory for review and checking of its factual accuracy, in advance. After any corrections have been made to the report, the final version should be submitted to management and appropriate action should be taken on the basis of the report's recommendations.

9.2.7. Inter-laboratory comparison

Intra-laboratory checking sometimes does not produce sufficient conclusions for evaluating and upgrading the Customs laboratory's performance. In such cases, inter-laboratory comparison may be useful. The main objectives of this are as follows :

- To develop analytical techniques among laboratories.
- To confirm the reliability of analytical methods.
- To examine the grade of standard materials or chemicals, including reliability.
- To highlight the laboratory's particular capabilities.
- To exchange technical information.
- To investigate optimum analytical methods, such as official analysis methods.
- To facilitate international trade through the acceptance of analytical data.

When carrying out this type of activity, international standards (e.g., ISO 5725, 10011-1, 10011-2, 10011-3, etc.) could be extremely useful.

Chapter 2 : Laboratory equipment, instruments and apparatus

INTRODUCTION

Set out below are tables listing various equipment, instruments and apparatus needed for a Customs laboratory. The tables are divided into three groups :

- Basic equipment, instruments and apparatus
- Special instruments and apparatus required for HS classification
- Other instruments and apparatus which may be needed in an advanced Customs laboratory.

The number of items of equipment, instruments or apparatus required depends, of course, on the number of analysts working in a laboratory. For the purposes of these tables it has been assumed that a standard Customs laboratory employs 12 analysts.

This Chapter of the Customs Laboratory Guide also contains a section on instrument maintenance, etc.

Note)

"*" indicates basic equipment or apparatus which is essential to start up a Customs laboratory. The number of items, given in brackets following the "*", is the minimum and more will be required depending on the size of the Customs laboratory.

"**" indicates essential instruments to start up a standard Customs laboratory, and equipment or apparatus used with those instruments. The number of items (the minimum) required are given in brackets following the "**".

The ability of a developing country's Customs laboratory to purchase instruments, equipment and apparatus will depend on its resources. Basic items should be purchased to establish a minimum capability dependent on the types of analyses required. Additional supplies should only be purchased as workflow dictates or where experience shows them to be necessary.

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1. Basic equipment, instruments and apparatus

1.1. Basic equipment

Equipment	Number (for a laboratory of 12 chemists)	Remarks
Chemical experimental table	6	* (1)
Physical experimental table	9	* (1)
Balance table	6	* (1)
Storage cabinet, normal type	3	* (2)
Storage cabinet, safety type	6	* (1)
Fume cupboard	6	* (2)
Air conditioner	1	For infra-red room** (1)
Sink	6	* (2)
Chair, for laboratory	20	* (5)
Office table and chair	12	* (3)
Fire extinguisher	6	* (2)
Emergency shower	2	* (2)
Eyewash station	2	* (1)
Refrigerator	1	For storage of samples, reagents, etc.* (1)
Alarm	6	* (2)
First-aid box	1	* (1)
First-aid medicines	1 set	* (1)
Cylinder	10	H_2, O_2, N_2, He, etc.** (4)
Bookshelf	3	* (2)

1.2. Glassware

Glassware		Number	Remarks
Beaker	10 ml	30	
	20 ml	30	
	50 ml	250	* (30)
	100 ml	360	* (30)
	200 ml	60	* (10)
	300 ml	30	
	500 ml	15	* (2)
	1000 ml	6	* (1)
	2000 ml	5	
Conical beaker	200 ml	60	* (5)
	300 ml	30	
	500 ml	10	
	1000 ml	5	
Tall beaker	100 ml	15	* (5)
	200 ml	15	
Erlenmeyer flask	10 ml	15	
	50 ml	150	* (10)
	100 ml	250	* (15)
	200 ml	150	* (10)
	300 ml	30	
	500 ml	15	* (5)
	1000 ml	6	* (1)
	2000 ml	5	
Filtering flask	300 ml	3	* (1)
Long-neck Kjeldahl flask	200 ml	30	* (3)
Round-bottom flask	50 ml	10	
	100 ml	20	* (2)
	200 ml	10	* (2)
	500 ml	10	
	1000 ml	5	
Short-neck Kjeldahl flask	10 ml	20	
	25 ml	10	* (5)
	50 ml	10	* (5)
	100 ml	20	* (5)
	200 ml	10	
	500 ml	10	
	1000 ml	5	
Volumetric flask, clear	10 ml	30	
	25 ml	30	
	50 ml	30	* (5)
	100 ml	90	* (10)
	200 ml	15	* (5)
	250 ml	15	* (5)
	500 ml	9	* (1)
	1000 ml	9	

Glassware		Number	Remarks
Volumetric flask, amber	50 ml 100 ml 250 ml 500 ml	30 30 30 10	* (5)
Culture dish		30	* (2)
Graduated cylinder	10 ml 25 ml 50 ml 100 ml 250 ml 500 ml 1000 ml	15 15 15 15 6 6 6	 * (2) * (2) * (2)
Colour comparison		30	** (5)
Watch glass	60 mm 120 mm	30 30	** (5)
Centrifuge vessel		10	** (6)
Dropping bottle with bulb and pipette		15	* (5)
Drying tube, straight U bend		10 10	* (2)
Reagent bottle, narrow mouth, amber	60 ml 250 ml 500 ml	30 30 30	* (10)
Reagent bottle, wide mouth, plain	30 ml 120 ml 500 ml	15 15 15	* (5)
Reagent bottle, narrow mouth, plain	60 ml 250 ml 500 ml	30 30 30	* (10)
Reagent bottle, wide mouth, amber	30 ml 120 ml 500 ml	15 15 30	* (5)
Specific gravity bottle		6	* (2)
Specimen bottle		200	
Test tube	18 mm 30 mm	1500 75	* (100)
Vial, with polyethylene stopper		300	
Weighing bottle	30 x 30 mm 40 x 40 mm	30 30	* (3)
Burette, plain plain amber amber	10 ml 50 ml 10 ml 50 ml	6 6 6 6	* (2)

Glassware		Number	Remarks
Measuring pipette	1 ml	30	* (3)
	2 ml	30	* (3)
	5 ml	30	* (3)
	10 ml	30	* (3)
Volumetric pipette	0.5 ml	6	
	1 ml	60	* (6)
	2 ml	60	* (6)
	3 ml	30	* (3)
	4 ml	4	
	5 ml	30	* (3)
	10 ml	30	* (3)
	15 ml	6	
	20 ml	30	* (3)
	25 ml	30	* (3)
	30 ml	9	
	50 ml	15	* (1)
	100 ml	15	
Komagome pipette	1 ml	12	
	2 ml	12	
	5 ml	12	* (2)
	10 ml	12	
Desiccator, plain	150 mm	12	* (2)
plain	300 mm	3	
amber	300 mm	3	
Desiccator plate	150 mm	12	* (2)
	300 mm	6	
Developing vessel (large size)		3	For TLC (thin-layer chromatography)* (2)
Developing vessel (small size : sample bottle)		5	For TLC* (2)
Capillary, disposable		3,000	For TLC* (100)
Nebulizer, amber		30	For TLC* (3)
Developing vessel		3	For PC (paper chromatography)
Filtering funnel, long stem			
	45 mm	15	
	75 mm	30	
Filtering funnel	30 mm	30	
	75 mm	90	* (10)
	120 mm	30	
	180 mm	15	

Glassware		Number	Remarks
Glass funnel, cylindrical type, fused-in fritted glass disc (30 ml)	G0 G1 G2 G3 G4 G5	6 6 15 6 6 6	
Separating funnel	50 ml 100 ml 300 ml 500 ml 1000 ml	15 30 15 10 6	* (5)
Aspirator		9	* (2)
Condenser (Liebig)		5	* (2)
Glass tubing	1.2 m	60	
Glassware for soxhlet extraction		6	* (2)
Glassware for distillation		6	* (2)
Quartz cuvette (6 pairs)		12	For UV-VIS (Ultraviolet and visible) spectrophotometer** (1)
Vessel for quartz cuvette		2	For UV-VIS spectro-photometer** (1)
Chromatographic column with stopcock		10	For column chromatography* (3)
Glass wool		1	For GC (gas chromatography)** (1)
Glass insert		12	For GC** (3)
Glass column	50 cm 1 m 2 m 4 m	10 10 20 10	For GC ** (6)
Glass syringe	1 ml 2 ml 5 ml 10 ml	6 6 6 6	** (2)
Stopcock		60	
Glass stirring rod		20	* (2)
Cover glass		3,000	For microscopes** (300)
Slide glass		1,500	For microscopes** (100)
Spherical ampoule		10	

1.3. Rubber and plastic ware

Laboratory ware	Number	Remarks
Beaker, polypropylene 50 ml 300 ml 1000 ml	15 15 6	* (2)
Rubber tube : Presser, endurable type Presser, endurable type (wire reinforced for hydraulic type) Silicone rubber type For gas Black or yellow rubber for laboratory use	 30 m 10 m 30 m 10 m 50 m	 For evaporators** (3) For analytical equipment For H_2, N_2, O_2, C_2H_2, Ar(He) Diameters should be suitable for glass tubes
Rubber bulb for measuring pipette 1 ml 2 ml 5 ml 10 ml	30 30 30 30	* (3)
Rubber stopper : For test tube, small Erlenmeyer flasks and reagent bottle (narrow mouth) For large Erlenmeyer flasks and reagent bottle (wide mouth)	 600 24	* (6)
Silicone rubber stopper, small	180	
Centrifuge vessel	10	** (3)
Reagent bottle (polyethylene) 50 ml 500 ml	60 90	
Specimen bottle	300	
Washing bottle 500 ml	30	* (2)
Graduated cylinders, polypropylene 10 ml 50 ml	6 6	
Measuring pipettes, polyethylene 1 ml 5 ml	30 30	
Pipette washer	3	
Disposable plastic syringe 5 ml 10 ml 50 ml	250 250 250	Per year Per year Per year
Spoon, plastic	12	* (3)
Rubber bulb	12	
Disposable gloves	500	Per year* (50)

Laboratory ware	Number	Remarks
Gloves, anti-acid	12	
Protection gloves	5	
Desiccator of plastics, box-type	3	For TLC* (1)
Septum	26	For GC** (6)
Column case	2	For GC** (1)
Pipette case	3	* (1)
Pipette controller	5	* (2)
Teflon seal tape	10	* (2)
Chemical structures (model)	1 set	
Trough	12	

1.4. Other laboratory ware and utensils

Laboratory ware	Number	Remarks
Evaporating dish, porcelain 20 ml 50 ml 120 ml 400 ml	 30 30 30 6	 * (3)
Porcelain crucible and lid 10 ml	600	
Disposable aluminium weighing/evaporating dish	126	Per year
Zirconium, vitreous carbon or alkali-resistant sintered alumina crucible 30 ml	5	
Porcelain funnel, Büchner type 55 mm 90 mm 150 mm	 6 6 3	 * (2)
Cork stopper : For test tube, small Erlenmeyer flask and reagent bottle (narrow mouth) For large Erlenmeyer flask and reagent bottle (wide mouth)	 600 50	 * (50)
Cork borer	3	* (1)
Cork press	3	* (1)
Cork ring 100 ml 200 ml 300 ml 500 ml	 10 10 10 10	 * (1)
Filter paper, for quantitative test 55 mm 90 mm 150 mm	 1,500 6,000 3,000	 * (100)
Filter paper, for quantitative test Quick filtering 90 mm Ordinary use 90 mm Fine particles 90 mm Ordinary use 90 mm	 300 1,500 3,000 1,500	Ash content 0.09 mg/piece 0.09 mg/piece 0.09 mg/piece 0.09 mg/piece
Thimble filter paper	750	For soxhlet extractors* (70)
Litmus paper	10	* (2)
Micro-dispenser	100	
Powder paper, small large	30,000 30,000	
Section paper	5	* (1)
Semilogarithmic graph paper	2	

Laboratory ware	Number	Remarks
Log-log graph paper	2	
Burette clamp, double	5	* (1)
Clamp, small large	12 24	
Clamp holder	20	
Clamp for glassware joint	120	* (10)
Clamp for test tube	60	* (6)
Mohr clamp	60	* (6)
Pinchcock clamp	10	
Rod or pipe connector	48	
Clay triangle	10	
Open ring support 50 mm 90 mm 150 mm	6 6 3	* (2)
Test tube holder	10	* (1)
Tripod	15	* (1)
Wire gauge, with asbestos	10	* (1)
Rack for test tube, small large	12 3	
Support, small large	12 12	* (1)
Support for funnel	12	
Support, jack, with precise vertical adjustment	5	* (1)
Support for burette	6	* (1)
Support for colour comparison	3	
Support for pipette	6	* (1)
Copper wire	3	* (1)
Nichrome wire	3	
Platinum wire	1	* (1)
Burner, Bunsen	12	LPG (Liquid petroleum gas)* (2)
Deep-blue cobalt glass	3	
File	6	
Label	10	* (1)
Level gauge	50	

Laboratory ware	Number	Remarks
Manometer	5	
Test sieve	12	
Test tube brush	50	* (5)
Thermometer 0 - 360 20 - 100 Precision 0 - 100	6 6 6	* (1) * (2)
Hygrometer	3	
Mortar and pestle, porcelain 60 mm 120 mm 210 mm	 6 9 3	
Mortar and pestle, agate 50 mm	3	
Mortar and pestle, alumina 120 mm	 3	
Filter paper, for chromatography 2 x 40 cm 40 x 40 cm	 300 150	For PC
TLC plate, silica gel 10 x 20 cm 20 x 20 cm cellulose 20 x 20 cm	12 p. 30 p. 6 p.	For TLC "p." = pack * (3)
Capillary column	3	For GC** (2)
Stainless-steel column	6	For GC
Column accessories for glass column for capillary column for stainless-steel column	 2 set 2 set 2 set	For GC ** (1) ** (1)
Column packing kit	1	For GC** (1)
Capillary column holder	3	For GC** (1)
Micro-syringe 1 µl 2 µl 5 µl	9 6 6	For GC** (2)
Press and evacuable die to prepare KBr pellets	1 set	For IR ** (1)
Crystal-polishing kit	1 set	For IR ** (1)
Blower, foot-powered	3	
Clip	10	
Forceps, stainless-steel	120	* (5)
Micro-spatula	60	* (6)
Needle for glass syringe	15	

Laboratory ware	Number	Remarks
Spoon, stainless steel	60 15	* (6)
Magnetic spin bar Tongs for crucible 210 mm 450 mm	36 15 3	** (3) * (3)
Carrier for laboratory bottles	5	
Safety goggles	15	* (2)
Magnetic stirrer	12	** (2)
Safety ware	25	* (2)
Driver set	1	* (1)
Glass cutter	1	* (1)
Knife	3	
Pliers	2	
Saw	3	
Micrometer, vernier, etc.	1	To determine thickness
Tool kit	1	
Tube cutter	1	
Vice	2	

1.5. Basic instruments and apparatus

Instruments	Main uses
Fourier transform infra-red (FT-IR) spectrophotometer *	Organic, inorganic chemicals, polymers, narcotics
Gas chromatograph *	Organic chemicals, petroleum, food, perfumes
Air compressor **	For GC
Vibrator for column packing **	For GC
UV & VIS spectrophotometer *	Quantitative measurement for food, chemicals
Emission spectrophotometer	Qualitative analysis of inorganic materials
X-ray diffractometer *	Inorganic chemicals, minerals
Atomic absorption spectrophotometer *	Measurement for content of metals
Accumulator	Quantitative analysis of metal, etc. (electrolytic method)
Hydrometer (1 set)	General purpose
pH meter *	General purpose
Photometer	Quantitative analysis
Polarograph	Quantitative analysis of metal, etc.
Potentiometer	Quantitative analysis of metal, etc.
Tester	General purpose
Melting-point apparatus *	Organic chemicals
Petroleum-oil distillator	Petroleum oil
Nitrogen analyser system *	Foods
Refractometer	General purpose
Surface-tension balance *	Surface-active agents
Automatic water distillation or Water purify *	Purifying water
Electric muffle furnace *	Food, inorganic chemicals, minerals
Electric drying oven *	General purpose
Vacuum drying oven *	Food, plastics or paint
Direct-reading chemical balance *	General purpose
Electronic even balance *	General purpose
Constant-temperature water bath *	For physical measurements at 15 °C or 20 °C
Oil bath	General purpose
Sand bath *	General purpose

Instruments	Main uses
Optical (polarised light) microscope *	Food, minerals
Stereo-microscope *	Textiles, minerals
Various kinds of electrodes	Quantitative analysis of metal, etc.
Centrifuge *	Food, paint, other separation
Homogenizer	Foods
Mill	General purpose
Oxygen burner	General purpose
Rotary evaporator *	For concentration or evaporation of organic solvents
Infra-red lamp	Evaporation of solvents
Mixer	General purpose
Resistance box	General purpose
Stopwatch	General purpose
Timer	General purpose
UV light **	For TLC
Calculator	General purpose
Heavy ion eliminator	Elimination of heavy metals from effluent

2. Special instruments and apparatus required for Harmonized System classification

The following instruments and apparatus are necessary for specific methods of analyses for determining criteria prescribed by the legal texts or the Explanatory Notes of the Harmonized System. The kinds of instruments and apparatus which need to be installed may vary from country to country, depending on the volume of trade in the relevant goods, etc.

It should be noted that many Customs laboratories are not equipped with all of these instruments and apparatus, because most of them are expensive and may not be frequently used. Attention should therefore be paid to the cost and benefits when deciding which kinds of instruments and apparatus are to be procured.

Special instrument and apparatus	HS provision
Polarimeter for sugar analysis (saccharimeter)	Note 2 (A)(a) to Chapter 11 and Subheading Note 1 to Chapter 17
Woven metal wire cloth sieves with apertures of 315 micrometres, 500 micrometres, 1.25 mm and 2 mm	Notes 2 and 3 to Chapter 11
Thermometer, fixed in a stopper, for Bellier reaction	ENs to headings 15.09 and 15.10
Pressure gauge and thermostat, for determining excess pressure of sparkling wines	Subheading Note 1 to Chapter 22
Distillation apparatus and hydrometer for alcoholic measurement	Note 6 to Chapter 20, Note 1 (f) to Chapter 21, Note 3 to Chapter 22, headings 22.07 and 22.08
Special steam-distillation apparatus for determining fluoride content	Subheadings 2529.21 and 2529.22
Special muffle furnace, silica crucible and silica lid for coal analysis	Subheading Notes 1 and 2 to Chapter 27
Adiabatic bomb calorimeter	Subheading Note 2 to Chapter 27
Vacuum distillation apparatus for petroleum products	Note 2 to Chapter 27 and Note 3 (a) to Chapter 39
Distillation apparatus for petroleum products	Note 2 to Chapter 27, Note 3 (a) to Chapter 39, Subheading Notes 3 and 4 to Chapter 27, subheading 2707.50 and ENs to headings 29.02 and 29.33
Hydrometer for petroleum	EN to heading 27.12
Penetrometer, standard cone, etc.	EN to heading 27.12
Apparatus for determining oil content in paraffin wax	Subheading 2712.20
Geiger-counter	Note 6 (d) to Chapter 28

Special instrument and apparatus	HS provision
Rotational viscometer and dropping point measuring apparatus	EN to heading 34.04
Devices for measuring minimum burst pressure of plastic tubes, pipes and hoses	Subheading 3917.31
Rubber testing apparatus consisting of - - vulcanizing apparatus - - test-piece press - - tensile test machine, and - - straining device	Note 4 to Chapter 40
Elrepho apparatus or other equivalent brightness tester	Note 5 to Chapter 48
Mullen-type bursting tester	Note 5 and Subheading Notes 1, 2, 5 and 6 to Chapter 48
Tear-testing machine and tensile-testing machine for paper	Subheading Note 2 to Chapter 48
CMT 30 crush-resistance tester	Subheading Notes 3 and 4 to Chapter 48
Thermohydrostat for producing the standard atmospheres for the conditioning and testing of textiles	General EN to Section XI
Tensile-testing machine	Note 6 and Subheading Note 1 (a) to Section XI, and subheadings 5607.21 and 5607.41
Straining device for textiles	Subheading Note 1 (a) to Section XI
Devices for determining mass per unit length of yarn	Section XI
Devices for determining mass per unit area of textiles	Section XI
Carbon analyser	Subheading 6903.10
Equipment for determining the linear coefficient of expansion of glass	Subheadings 7002.32, 7013.32 and 7017.20
Test sieves having mesh apertures of 63 micrometres, 0.5 mm, 1 mm and 5 mm	Subheading Note 1 to Chapter 71, Note 8 (b) to Section XV, Note 1 (h) to Chapter 72 and Subheading Note 1 (c) to Chapter 79
Carbon and sulphur analyser	Chapter 72
Electrolysis equipment	Notes 1 (a) and (b) to Chapter 74
Oxygen analyser	Subheading Note 1 to Chapter 75

3. Other equipment, instruments and apparatus which may be needed in a standard and advanced Customs laboratory

3.1. Equipment

Equipment	Number	Remarks
Safe	1	For illicit drugs, etc.
Supervision camera	1 set	
Harmful-gas alarm detector	>10	For each room
Inflammable-gas alarm detector	>5	For H_2(GC), etc.
Air cleaner	2	
Water pure refinery	1	
Crusher	1	For sampling conditioning
Vibratory disk mill	1	For sampling conditioning
Driller	1	For sampling conditioning
Saw for metal	1	For sampling conditioning
Pressure regulator to be used with compressed gases	1	For sampling conditioning
Apparatus and devices for waste treatment	1	
Fume hoods	6	For GPC (gel-permeation chromatography), etc.
Clean air bench	1	
Air conditioner	>3	For IR (infra-red), MS (mass spectrometer), NMR (nuclear magnetic resonance) rooms, etc.
Freezer	2	For storage of samples, reagents, reference materials

3.2. Glassware

Glassware		Number	Remarks
Beaker	1 ml	15	
3-Neck round-bottom flask	500 ml 1000 ml	10 10	
Filtering flask	500 ml 1000 ml	5 5	
Flat-bottom flask	100 ml 200 ml 300 ml 500 ml 1000 ml	20 10 10 5 3	
Short-neck pear flask	10 ml 20 ml 50 ml 100 ml 200 ml	15 15 30 20 10	
Distillation flask		30	For automatic (vacuum) distillation apparatus
Automatic burette		2	
Micropipette		10	
Pasteur pipette		25	
Evaporating dish	50 ml 100 ml 200 ml	10 25 15	
Mini petri dish for culture		50	
Test tube with stopper		100	
Syringe vial		100	
Crimp seal vial		100	
Desiccator, with socket on the cover		5	
Adapter	(reducing) (enlarging) (distilling) (straight type), etc.	10 10 10 10	
Condenser	(tube) (Alihn) (Graham) (Dimroth)	10 10 10 10	
Distilling column		5	
Bulb (Kjeldahl), long delivery tube, bent		5	

Glassware	Number	Remarks
Bulb (Kjeldahl)	10	
Sample pipette	10	For MS
Tube for auto-collector	100	For GPC
Sample tube	50	For NMR, per year

3.3. Other laboratory ware and utensils

Laboratory ware	Number	Remarks
Safe pipette controller	5	
Automatic burette (complete set)	5	
High-precision tweezers	3	
Colony counter	1	
Wire basket	5	For autoclave
Dewar container or vessel	5	
Flexible heating ribbon	3	
Laboratory shelf	5	
Diamond scriber	1	
Sealon film	5	Per year
Screw-cap vial 1 ml 2 ml 5 ml 10 ml	300 100 100 50	Per year Per year Per year Per year
Air-tight syringe	5	For GC
Foil for pyrolyser	10	For GC
Column for GC-MS (gas chromatograph/mass spectrometer)	5	
Column accessories for GC-MS	5	
Vacuum vial	50	
Rubber stopper	100	For seal vial
Crimp cap (aluminium)	100	For seal vial
Plier-type decapper	2	For seal vial
Hand-operated aluminium cap crimper	2	For seal vial
Seal vial 1 ml 2 ml 5 ml 10 ml	100 50 30 30	For GC and HPLC (high performance liquid chromatography), etc.
Column accessories for HPLC	10	
Column for HPLC	10	
Guard-column for HPLC	10	

Laboratory ware		Number	Remarks
Micro-syringe	2 µl	2	For HPLC
	5 µl	5	
	10 µl	2	
	25 µl	2	
Disposable plastic syringe			For HPLC, etc.
	10 ml	100	Per year
	25 ml	100	Per year
	50 ml	100	Per year
Micro-filter for micro-syringe			For HPLC
	0.10 µm	1000	Per year
	0.20 µm	1000	
	0.45 µm	1000	
(water and anti-water types)			
Micro-filter	0.10 µm	100	For HPLC
	0.20 µm	100	Per year
	0.45 µm	100	
(water and anti-water types)			
Filter holder		2	For HPLC
Sample table		30	For SEM (scanning electron microscope)
Film		50	For SEM
Printing photographic paper		100	For SEM
Aluminium pan, disposable		100	For thermal analyser
Platinum pan		5	For thermal analyser
Cathode lamp		1 set	For AAS (atomic absorption spectrometer)
NMR tube rack		3	For NMR
Cap for sample tube		20	For NMR
Diskette		100	For computer

3.4. Instruments and apparatus

Instruments	Main uses
Auto-sampler system for GC	Organic chemicals, drugs, essential oils, etc.
Pyrolyser system for GC	Polymers
Gas chromatograph/mass spectrometer (GC-MS)	Organic chemicals, essential oils, drugs, etc.
Secondary ion mass spectrometer (SIMS, etc.)	Organic chemicals, polymers, etc.
High-resonance inductively-coupled-plasma/mass spectrometer (ICP-MS)	Inorganic materials
Liquid chromatograph/mass spectrometer (LC-MS)	Organic chemicals, drugs, etc.
Gas chromatograph/Fourier transform infra-red spectrophotometer (GC-FT-IR)	Organic chemicals, drugs, essential oils, etc.
Super-critical fluid chromatograph	Organic chemicals
High-performance liquid chromatograph (HPLC)	Organic chemicals, drugs, oils, saccharides, etc.
Ion chromatograph	Inorganic chemicals
Fully-automated amino acid analyser	Amino acid, foods
Gel-permeation chromatography (GPC)	Organic chemicals, polymers
Fourier transform infra-red spectrophotometer	General purpose
Raman spectrophotometer	Organic and inorganic chemicals
Spectropolarimeter	Organic materials
Inductively coupled plasma atomic emission spectrometer	Inorganic materials
Nuclear magnetic resonance spectrometer (NMR)	Organic chemicals, polymers, drugs, etc.
X-ray fluorescence spectrometer	Inorganic materials
Scanning or transmission electron microscope (SEM or TEM)	Inorganic chemicals, starch, ore, etc.
Vacuum evaporator	For SEM or TEM (transmission electron microscope)
Ion-spattering device	For SEM or TEM
Freeze-dry device	For SEM or TEM, etc.
Energy dispersive X-ray spectrometer	Inorganic materials
Auto-tensiometer	Surface-active agents
Automatic vacuum distillation apparatus	Oils, polyolefins
Density/specific gravity measuring instrument	Oils, organic chemicals, etc.

Instruments	Main uses
Thermal analyser	Ore, inorganic chemicals
Capillary electrophoresis apparatus	Proteins, etc.
Electrophoresis apparatus	Proteins, etc.
Automatic molecular weight apparatus	Organic chemicals, polymers
Karl Fisher titrating apparatus	Quantitative analysis of water
Kinetic viscosity monitoring system	General purpose
Micro-organic element and gas analyser	Organic chemicals, polymers
Semi-micro amino nitrogen apparatus (Van Slyke method)	Foods
Surface-area apparatus	Clays, etc.
Viscosity measuring system	Liquid materials
Densitometer	For TLC
Autoclave	Enzymes, etc.
Incubator	Enzymes, etc.
Automatic titrating apparatus	Quantitative analysis
Automatic pipetting machine	Quantitative analysis
Chromatography system	Chemicals, drugs, lubricating oils, etc.
Coulometric analyser	Quantitative analysis of metal, etc.
Electrolytic analyser	Quantitative analysis of metal, etc.
Flame spectrophotometer	Quantitative analysis of metal, etc.
Circulating aspirator	General purpose
Electronic shaker	General purpose
High-vacuum pump	General purpose
Magnetic stirrer with heater	General purpose
Hot plate	General purpose
Personal computer	General purpose
Ultrasonic cleaner	For HPLC, etc.
Ultrasonic pipette cleaner	Washing of pipettes, etc.

4. Instrument maintenance and attention

4.1. Introduction

An inventory record and a service record (which covers information on performance problems, repairs and cost of repairs, date of service, etc.) and a checklist on each instrument should be kept by the person in charge, who is assigned by the Director General.

It is, of course, desirable to ask the suppliers to offer routine maintenance training as part of the purchase contract. The specially-trained staff should prepare diagrams, charts or a simple manual for other staff, and should explain the operating techniques to them. Before using the following instruments, staff should read the manual on each instrument and thoroughly understand its operation. If an accident occurs, the person in charge or specially-trained staff should be informed immediately. If the instrument can not be repaired by staff, a qualified service representative could be asked to repair the instrument, but this would entail considerable expense after the end of the guarantee period. To save costs, it is important to identify parts which are likely to need periodic replacement and hence to maintain a stock of these parts.

It is desirable that the periodic servicing (e.g., an annual check) be carried out on each instrument by a qualified service representative.

It may sometimes be advisable to operate certain complex and expensive instruments as a kind of "in-house service" (e.g., saccharide analysis by HPLC, fatty-acid analysis by GC) on a regular, (e.g., weekly) basis. In this way, the number of people operating expensive and complex instruments can be limited to a small number of experienced people. Furthermore, similar samples requiring similar analytical conditions can be grouped, thus avoiding frequent changes of instrument parameters and parts such as columns. Both measures will reduce the risk of mishandling and expensive repairs.

4.2. Instrument maintenance, etc.

Instrument	Parameter	Maintenance, etc.
Infra-red spectrophotometer	Resolution, wave-number accuracy and reproducibility	Resolution, wave-number accuracy and reproducibility should be checked with a polystyrene film on a monthly basis.
	Beam balance	The beam balance should be checked in air on a monthly basis.
	Solvents (CS_2, CCl_4, etc.)	Solvents (CS_2, CCl_4, etc.) should be handled in a fume chamber.
	KBr/NaCl plate, etc.	A KBr/NaCl plate should not be used for samples including moisture. The frosted plate should be polished on, for example, a deerskin dampened with alcohol. After use, the plates, assembly cells, fixed pass cells, agate mortars and pestles, etc., must be cleaned and stored in a dry desiccator.
	Humidity of IR room	An infra-red room has to be air-conditioned to ensure less than 50 % humidity.
Refrigerator	Sample	Samples, reagents, etc., should be put away and unnecessary materials should be disposed of by a person in charge. Valuable samples and reagents should be stored carefully, e.g., these materials should not be placed near the door.
	Temperature	Check the temperature of the refrigerator every day.
Gas chromatograph	Gas leak	When in use, check for a leak of H_2 gas, etc., with a soap solution. If the check is positive, immediately close the gas supply valve.
	Changing the column	After cooling the oven to room temperature, change the column carefully using gloves. Before use, sufficient carrier gas must flow in the column. The maximum temperature of each column should be checked in advance.

Instrument	Parameter	Maintenance, etc.
	Septum	Replace dirty septum with a new one before use.
	Glass insert	Clean or replace the glass insert every week, when in use.
	Washing of detector	Before use and after cooling a detector to room temperature, wash the detector, if necessary (acetone is employed, for example, if silicones and TMS(trimethyl silyl) - derivatives are injected).
	Ignition of H_2 gas	When in use, confirm ignition of H2 gas.
	Temperature of column oven	On a quarterly basis, the temperature of the oven should be checked using a portable indicating reference pyrometer. When in use, check the temperature control system and the overheating protector. After use, allow carrier gas to flow in the column for cleaning purposes until it has been cooled to room temperature for more than 30 minutes and all switches are finally turned off.
Gas chromatograph	Valve check for gas supply	After use, close each gas supply valve.
X-ray diffractometer	Angle check	An angle check should be carried out every month.
	X-ray leak	Checks for X-ray leaks should be carried out when in use.
	Cooling water	Before use, check the water pressure and flow rate. Clean the water tank every quarter.
	X-ray shutter	Before use, close the X-ray shutter.
	X-ray tube	When changing an X-ray tube, handle it carefully. Turn up the switch of the X-ray tube gradually.
	Changing of test samples	When the sample is changed, close the X-ray shutter. The body must not be exposed to X-ray radiation.
	X-ray detector	X-ray detector is used to judge whether the body is exposed to X-rays. When an X-ray diffractometer is in use, the analyst must always have an X-ray detector.

Instrument	Parameter	Maintenance, etc.
	Use	The Director General must designate staff to be in charge of the use of the X-ray diffractometer; only the designated staff can use this instrument. During operation, the X-ray protection box should be completely closed and the operator should inform other staff that the X-ray diffractometer is in use.
Mill	Crushing	During crushing, do not insert fingers into the body of the mill. The sample should be taken out after the electronic power has been switched off.
	Washing	After use, the cutter and sample vessel should be cleaned. Be careful not to drench the motor.
UV & VIS spectrophotometer	Wave-length accuracy and re-producibility	Twice a month, the wave-length accuracy and reproducibility should be checked over the entire UV-visible range by using standard/reference materials.
	Absorbance accuracy and re-producibility	Every month, the absorbance accuracy and reproducibility should be checked by using standard /reference materials scanning from 210 to 450 nm.
	Cell	Handle cells carefully ; after use, clean and store them in alcohol.
	Solvent	Care should be taken to ensure adequate ventilation; do not leave organic solvents on the benches.
	Light source	After stabilization, the measurement should be started. Eyes should not be exposed to the light source.
Balance	Accuracy	Reference weights should be calibrated. If necessary, ask a qualified service operator or a specially-trained staff member to check or repair.
	Cleaning	Keep the analytical balance clean, to protect all parts from dust or contamination.
Emission spectro-photometer	Mirror balance	Mirror balance should be performed by a qualified service representative.
	Ventilation system	Check the ventilation system to ensure that combustion gas does not leak into the room.

Instrument	Parameter	Maintenance, etc.
	Electrode support	Do not touch the electrode support during measurement.
	Safety system for high-voltage circuit	Inspect the safety system for the high-voltage circuit at regular intervals (quarterly).
	Diffraction grating and mirror surface	The door of the body of the instrument should always be closed. Do not touch the diffraction grating and mirror surface.
	Photographic film or plate	Photographic film and plates should be handled in a dark room.
	Cooling water	Cooling water is made to flow through this instrument when in use.
	Light source	Eyes should not be exposed to the light source.
Saccharimeter	Calibration and linearity	Calibrate and determine linearity with standard sucrose solutions.
Heating mantle	Electrical resistance box	An electrical resistance box should be employed for temperature control.
	Use	Do not use a heating mantle if the heating coil is exposed. If water and solvents are spilt, the heating mantle must immediately be switched off. Once adequately dried, the heating mantle can be used again.
Atomic absorption spectrophotometer	Gas leak	Check for any leakage of the gas to be used.
	Burner head	The burner head should be cleaned before use or after cooling to room temperature.
	Drain	Check that the drain bottle does not overflow.
	Position of burner, knob, etc.	Before ignition, check the position of the burner, the pressure control knob, the switching lever for the gas supply, etc.
	Cathode lamp	When changing a cathode lamp, it should be handled carefully.
	Leakage of liquids	If leakage of the sample solution is detected, the packing, connector, etc., must be changed after cleaning with water.

Instrument	Parameter	Maintenance, etc.
	Sensitivity	When used, the standard solution is aspirated into the flame and absorption should be determined. Sensitivity should be compared with previous results.
	Detection limit	When in use, the standard solution is aspirated with the flame six times consecutively. The solution, which gives a minimum of twice the baseline for every aspiration, represents the detection limit concentration. The detection limit should be compared with previous results.
	Gas valve	After use, each gas supply valve must be closed.
Centrifuge	Horizontal	The centrifuge is placed horizontally in the room.
	Cleaning	Keep the inside of the centrifuge clean.
	Balance	Equal weights of two sample vessels including muddy samples/suspension must be placed opposite one another.
	Abnormal sound	Revolutions should be gradually increased. If an abnormal sound is heard, the centrifuge should immediately be switched off and everyone should keep away from the centrifuge until it has stopped.
Oven	Temperature control	The temperature in the oven should be checked with an indicating reference pyrometer. When in use, check whether the temperature regulator is working.
	In the case of a sample including large amounts of organic solvent or water	If the sample includes a large quantity of organic solvent or water, then the solvent or water is initially evaporated to dryness on/in a water bath and the dried sample is then placed in the oven and thoroughly dried at an appropriate temperature.
	Vacuum drying oven	Between the drying oven and the vacuum pump, a vacuum trap should be employed. The air-supply valve should gradually be opened to bring the pressure in the oven up to the level of atmospheric pressure.

Instrument	Parameter	Maintenance, etc.
Rotary evaporator	Motor	The motor should be firmly fixed on a support.
	Quantity of sample	A sample solution corresponding to half the quantity of the flask to be used is added to it and the solvent in the sample solution is then evaporated.
	Sample vessel	The sample flask should be fixed to the evaporator with a clip, so as not to fall into the water bath.
	Water bath	Ensure that there is always a certain level of water in the bath to avoid heating an empty bath.
	Evaporated solvents	Evaporated solvents should be collected into the prescribed disposal bottle.
Homogenizer	Cutter	Before changing or washing the cutter, turn off the power.
	Vessel	A plastic vessel is very convenient to use.
	Use	The sample should be cut to a few mm in size in advance. After use, the cutter and vessel should be cleaned.
Surface tension balance	Plate or ring	The ring or plate of platinum or glass should be handled carefully with forceps and thoroughly washed before/after use.
	Sample dish	Sample dishes should be cleaned before/after use.
	Surface- tension accuracy	The surface tension of water/reference materials should be initially determined and adjusted.
Nitrogen analyser system	Decomposition procedure	The decomposition procedure should be carried out in a fume chamber.
	Sampling	Less than 1 g of a sample is used for nitrogen analysis.
	Heating	The sample, which includes a lot of hydrocarbons, should be carefully heated to avoid bubbling over. It is better to wait overnight after adding concentrated sulphuric acid and to start heating it the following morning.

Instrument	Parameter	Maintenance, etc.
	Diluting	After cooling, the decomposition solution should be transferred to a volumetric flask to which water has been added in advance. The flask should be shaken frequently to ensure thorough mixing.
	Distillation device	First check whether the connections are damaged. If so, they should be repaired/exchanged. Check that the distillation line is not tightly closed before heating.
	Alkali solution	The alkali solution should be carefully added to the sample solution.
	Washing	After distillation, this device should be washed thoroughly with water and subsequently with de-ionized water.
Water bath	Temperature check	When in use, check the temperature of the water bath by using a thermometer.
	Adding water	Ensure that there is always a certain level of water in the bath to avoid heating an empty bath.
Burner	Extinguisher	An extinguisher should be fitted beside the burner.
	Rubber tube	The rubber tube should be firmly settled. Check carefully for gas leaks.
	Volatile organic solvents	Before use, check that there is no volatile organic solvent around the burner.
	Ignition	Before igniting, check that the needle valve for adjusting the gas supply is closed. The gas supply valves are opened and the needle valve for adjusting the gas supply is then opened gradually and the burner is ignited. After ignition, the air and gas supply should be adjusted.
	Valve	After use, make sure that all gas supply valves are closed.
pH meter	Accuracy and linearity	When in use, pH value should be calibrated as closely as possible with commercially prepared buffers.

Instrument	Parameter	Maintenance, etc.
	Electrode	After use, the electrode should be carefully washed with de-ionized water and capped with de-ionized water to prevent it from drying out. Check the quantity of electrode solution every quarter and add more solution or change the solution if necessary.
Melting point apparatus	Calibration of thermometer	Take the melting points of standard materials.
	Heating bath oil or heating plate	Do not touch the heating bath oil or the heating plate directly.
Soxhlet extraction system	Extinguisher	An extinguisher should be fitted beside the fume chamber.
	Water bath	For long-term use, ensure that there is always a certain level of water in the bath to avoid heating an empty bath.
	Fume chamber	Extraction procedures should be carried out in the fume chamber.
	Temperature of water bath	The temperature of the water in the bath is set according to the boiling point of the solvent to be used, and there should be a sufficient flow of cooling water.
	Anti-bumper	Anti-bumper must be added in a collecting flask to prevent sudden bumping.
	Cooling and evaporation of solvent	After extraction, the pieces of equipment should be placed on an appropriate stand and cooled. The collecting flask is again placed on/in the water bath. The solvent in the flask is almost entirely evaporated. The flask is then placed in an oven, set at an appropriate temperature, and thoroughly dried.
	After use	After use, the extraction system should be switched off and the flow of cooling water should be stopped. Extraction tools must be cleaned and dried.

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x x

Chapter 3 : Chemical reagents, reference chemicals and materials

CONTENTS

3. **Chemicals for illicit drug analysis***

4. **Chemicals for titration analysis***

5. **Other organic reagents and materials***

5.1 Organic reagents for inorganic analysis

5.2 Hydrocarbons and their derivatives

5.3 Alcohols, phenols, phenol-alcohols and their derivatives

5.4 Ethers, ether-alcohols, ether-phenols and their derivatives

5.5 Aldehyde-function compounds

5.6 Ketone-function compounds

5.7 Carboxylic acids and their anhydrides, halides and their derivatives

5.8 Nitrogen-function compounds

5.9 Heterocyclic compounds

5.10 Vitamins

5.11 Other sugars

5.12 Starches

5.13 Vegetable oils

5.14 Waxes

5.15 Dyes

5.16 Essential oils

5.17 Textiles

5.18 Other

6. **Other inorganic reagents and materials***

6.1 Mineral products

6.2 Other inorganic acids of non-metals

6.3 Other salts of inorganic acids and metals, and inorganic compounds of precious metals

6.4 Other inorganic oxygen compounds

6.5 Base metals

6.6 Other

Notes 1 : Lists, which are marked by "*", would not include chemicals already listed in "1. Basic chemical reagents".

2 : "RM" means "Reference material". In order to save costs, known and well documented samples should be stored as reference materials.

3 : "RM(LH25.28)" and "RMs(LSXI)" mean Reference chemical (Legal text, heading 25.28) and Reference chemicals (Legal text, Section XI), respectively. "RM(ENC17N1b)" and "RM(ENC16G) mean Reference material (Explanatory Note, Chapter 17, Note 1-(b)) and Reference material (Explanatory Note, Chapter 16, General), respectively.

4 : Most goods classified in Chapters 28 and 29 are not listed as reference chemicals. If necessary, these reference chemicals should be prepared beforehand for use in experiments to determine, for example, whether unknown samples are separate chemical elements, separate chemically-defined compounds or separate chemically-defined organic compounds.

5 : Since the purchase of chemical reagents and reference materials is dependent on the resources available, it is desirable for a Customs laboratory to purchase the basic reagents and materials to establish a minimum capability, and expand as workflow increases or experience shows necessary.

List of chemical reagents, reference chemicals and materials for Customs laboratories

Reagent	Use	No.
Chloric acid		1.1
Hydrogen fluoride (Hydrofluoric acid)		1.1
Hydrogen chloride (Hydrochloric acid)		1.1
Nitric acid, fuming (Sp. Gr. 1.50)		1.1
Nitric acid (SG. 1.42)		1.1
Phosphoric acid		1.1
Phosphorous acid		1.1
Sulphuric acid, fuming (60 %)		1.1
Sulphuric acid		1.1
Ammonium hydroxide		1.2
Barium hydroxide	Deproteinising, etc.	1.2
Potassium hydroxide		1.2
Sodium hydroxide		1.2
Ammonium chloride	Buffer, etc.	1.3
Ammonium molybdate tetrahydrate		1.3
Ammonium nitrate		1.3
Barium chloride dihydrate	SO_4^-, etc.	1.3
Calcium carbonate		1.3
Cupric chloride dihydrate		1.3
Cupric sulphate pentahydrate	Fehling's solution A, crude protein, etc.	1.3
Cuprous chloride		1.3
Dipotassium carbonate anhydrous		1.3
Dipotassium sulphate	Crude protein, etc.	1.3
Disodium carbonate monohydrate	Alkaline solution, etc.	1.3
Disodium sulphate anhydrous		1.3
Ferric chloride	Drugs, etc	1.3
Ferrous chloride tetrahydrate		1.3
Ferrous sulphate heptahydrate		1.3
Platinic chloride	Drugs, etc.	1.3

Reagent	Use	No.
Potassium iodide	Illicit drugs, Hanes A, dextrose, etc.	1.3
Potassium permanganate		1.3
Potassium dichromate		1.3
Silver nitrate	Drugs, Cl^-, etc.	1.3
Sodium chloride		1.3
Sodium hydrogencarbonate		1.3
Sodium nitrate		1.3
Sodium sulphite		1.3
Sodium thiosulphate		1.3
Tetrapotassium hexacyanoferrate trihydrate		1.3
Tripotassium hexacyanoferrate	Hanes A, etc.	1.3
Trisodium orthophosphate		1.3
1,1,1-Trichloroethane		1.4
1,2-Dichloroethane		1.4
Acetone	Oils, etc.	1.4
Acetonitrile		1.4
Benzene	Oils, etc.	1.4
Butanone (Methyl ethyl ketone)		1.4
Carbon tetrachloride		1.4
Chloroform		1.4
Cyclohexane		1.4
Dichloromethane	Solvent	1.4
Diethyl ether		1.4
Ethanol (Conc. 99.5 % or more by weight)		1.4
Ethyl acetate	Alcoholic beverages	1.4
Glycerine		1.4
Methanol		1.4
n-Hexane	Squalene, etc.	1.4
n-Pentane		1.4
Petroleum ether	Lubricant oils, etc.	1.4
Phenol	RM(EN29.07)	1.9

Reagent	Use	No.
Propan-1-ol (n-Propanol)	Rum, etc.	1.4
Propan-2-ol (2-Propanol)	Alcoholic beverages	1.4
Pyridine		1.4
Tetrahydrofuran		1.4
Trichloroethylene		1.4
Toluene	RM(EN29.02)	1.4
Xylene	RM(EN29.02)	1.4
2,6-Dichloroindophenol sodium		1.5
Alizarin Red		1.5
Alizarin Yellow		1.5
Bromocresol Purple		1.5
Bromocresol Green		1.5
Bromophenol Blue		1.5
Bromothymol Blue		1.5
C.I. acid green 1 (Naphthol Green B)		1.5
C.I. direct red 28 (Congo Red)		1.5
Calmagite		1.5
Chloramine T		1.5
Chlorophenol Red		1.5
Cresol Red		1.5
EBT (Eriochrome Black T)		1.5
Eosin Y		1.5
Epichlorohydrin		1.5
Indigo carmine		1.5
Indole		1.5
Litmus		1.5
Metacresol Purple		1.5
Methyl Orange		1.5
Methyl Red		1.5
Methyl thymol Blue		1.5
Methyl Violet		1.5
Methyl Yellow		1.5

Reagent	Use	No.
Nessler's reagent		1.5
Neutral Red		1.5
Ninhydrin	Ninhydrin spray	1.5
o-Cresolphthalein		1.5
PAN (pyridylazonaphthol)		1.5
Phenol Red (Phenolsulphonephthalein)		1.5
Phenolphthalein		1.5
Sodium nitroprusside	Simon's reagent, nitrogen, etc.	1.5
Starch, soluble	Sucrose, etc.	1.5
Thymol Blue		1.5
Thymolphthalein		1.5
Xylenol Orange		1.5
Calcium chloride ($CaCl_2$)		1.6
Calcium oxide (CaO)		1.6
Calcium sulphate ($CaSO_4$)		1.6
Disodium sulphate (Na_2SO_4)		1.6
Silicone dioxide (SiO_2)		1.6
Sodium (Na)		1.6
Fructose	RM(LC17N1b)	1.7
Glucose	RM(LC17N1b)	1.7
Lactose, monohydrate	RM(LC17N1b)	1.7
Maltose, monohydrate	RM(LC17N1b)	1.7
Sucrose	RM(LC17N1b)	1.7
β-Galactosidase		1.8
Glucoamylase (α-1,4-Glucan glucohydrase)	Starch, etc.	1.8
Invertase	Sucrose	1.8
Acetic acid	Buffer, etc.	1.9
Boiling tips		1.9
Copper wire	Halogens, etc.	1.9
Dimethylsulfoxide		1.9
Dimethyl formamide	Textiles, etc.	1.9

Reagent	Use	No.
Hydrogen peroxide (Conc. approx. 30 %)	Fibres, etc.	1.9
Trifluoro acetic acid		1.9
Aluminium oxide (calcined alumina),(100-200 mesh)	Lubricant oils, preparations, etc.	2.1
Aluminium oxide (calcined alumina), acidic (100-200 mesh)	Lubricant oils, preparations, etc.	2.1
Aluminium oxide (calcined alumina), basic (100-200 mesh)	Lubricant oils, preparations, etc.	2.1
Celite	Vitamin E, etc.	2.1
Ion-exchangers	Mixtures, etc.	2.1
Silicon dioxide (activated silica gel) (100-200 mesh)	Lubricant oils, organic preparations, etc.	2.1
Tanning extracts of vegetable origin	RMs(LH32.01)	2.2
Anthracene	RM(EN29.02)	2.3
Benzene (purity 99 % (m/m) minimum)	RM(EN29.02)	2.3
m-Cresol	RM(EN29.07)	2.3
o-Cresol	RM(EN29.07)	2.3
p-Cresol	RM(EN29.07)	2.3
Xylenol	RM(EN29.07)	2.3
2-Ethyl-2-picoline	RM(EN29.33)	2.3
2-Vinylpyridine	RM(EN29.33)	2.3
Methylpyridine	RM(EN29.33)	2.3
Pyridine	RM(EN29.33)	2.3
Linoleic acid	RM(ENH29.16)	2.3
Ethanol	RM(LC20N6)	2.3
1,2,4-Trimethylbenzene	RM(LC27N2)	2.3
Cumene (Isopropylbenzene)	RM(LC27N2)	2.3
Ethylbenzene (purity 99 % (m/m) minimum)	RM(LC27N2)	2.3
n-Decane (purity 99 % (m/m) minimum)	RM(LC27N2)	2.3
Naphthalene	RM(LC27N2)	2.3
Squalane	RM(LH15.04)	2.3
Squalene	RM(LH15.04)	2.3
n-Dotriacontane	RM(LH15.10)	2.3
D-Glucitol (Sorbitol)	RM(LH29.05)	2.3

Reagent	Use	No.
Mannitol	RM(LH29.05)	2.3
Palmitic acid	RM(LH29.15)	2.3
Oleic acid	RM(LH29.16)	2.3
Stearic acid	RM(LH29.16)	2.3
Alcohol kit	RMs	2.3
Fatty acid kit	RMs	2.3
Hydrocarbon kit	RMs	2.3
Petroleum gases	RMs	2.3
Butter	RM(LH04.05)	2.3
Lard	RM(LH15.01)	2.3
10 % Sucrose diacetate hexabutylate (Chromosorb GAW DMCS, 80-100 mesh)		2.3
2 % Silicone OV-101 (Chromosorb GAW DMCS, 80-100 mesh)		2.3
2 % Silicone OV-1 (Gaschrom Q, 100-200 mesh)		2.3
2 % Silicone OV-17 (Chromosorb GAW DMCS, 80-100 mesh)		2.3
20 % DEGS (Chromosorb GAW DMCS, 80-100 mesh)		2.3
3 % Silicone SE-30 (Chromosorb GAW DMCS, 80-100 mesh)		2.3
3 % Dexil 300 GC (Chromosorb GAW DMCS, 80-100 mesh)		2.3
5 % PEG (polyethylene glycol) (Chromosorb GAW DMCS, 80-100 mesh)		2.3
PEG 20M (capillary column)		2.3
Silicone OV-101 (capillary column)		2.3
BSA (N,O-bis(trimethylsilyl)acetamide)	Narcotic drugs, etc.	2.3
Esterification reagents (BF_3-methanol (12 %, W/W), etc.)	Vegetable oils, fatty acids, etc.	2.3
Trifluoroacetic anhydride	Acetylation	2.3
Trimethylchlorosilane (TMCS)	Trimethylsilicification	2.3
Trimethylsilicification (TMS) reagent	Trimethylsilicification	2.3
1,4-Dioxane (HPLC grade)		2.4
Acetone (HPLC grade)		2.4
Acetonitrile (HPLC grade)		2.4
Benzene (HPLC grade)		2.4
Dichloromethane (HPLC grade)		2.4

Reagent	Use	No.
Diethyl ether (HPLC grade)		2.4
Ethyl acetate (HPLC grade)		2.4
Hexane (HPLC grade)		2.4
Methanol (HPLC grade)		2.4
N,N-Dimethylformamide (HPLC grade)		2.4
Water (HPLC grade)		2.4
Piperine	RM(ENC16G)	2.4
Caffeine	RM(LC19N3)	2.4
Theobromine	RM(LC19N3)	2.4
Polyethylene glycol (the average molecular weight : 200, 300, 500, 1,000, 2,000 etc.)	RM	2.5
Polystyrene (the average molecular weight : 200, 300, 500, 1,000, 2,000 etc.)	RM	2.5
Carbon bars	Support	2.6
Iron bar (Standard)	RM	2.6
Atomic absorption (AAS) standards (Ag, Al, As, Au, B, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ir, K, Mg, Mn, Mo, Na, Nb, Ni, P, Pa, Pb, Pt, Rh, Ru, Se, Sn, Te, Ti, V, W, Zn, Zr) 1000 ppm	RMs(LSXI)	2.7
Inductively coupled plasma (ICP) standards (Ag, Al, As, Au, B, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ir, K, Mg, Mn, Mo, Na, Nb, Ni, P, Pa, Pb, Pt, Rh, Ru, Se, Sn, Te, Ti, V, W, Zn, Zr) 10,000μg/ml	RMs(LSXI)	2.7
KRS-5 cell		2.8
Liquid paraffin		2.8
Potassium bromide (KBr) powder or die (spectrophotometric grade)		2.8
Potassium bromide (KBr) cell		2.8
Potassium bromide (KBr) salt plate		2.8
Sodium chloride (NaCl) cell		2.8
Sodium chloride (NaCl) salt plate		2.8
AgBr plates for wet or aqueous samples (1 set)		2.8
AgCl plates		2.8
Polyethylene	RM(LH39.01)	2.8
Polyisobutylene	RM(LH39.02)	2.8
Polypropylene	RM(LH39.02)	2.8
Poly(vinyl chloride)	RM(LH39.04)	2.8

Reagent	Use	No.
Poly(vinyl acetate)	RM(LH39.05)	2.8
Poly(vinyl alcohol)	RM(LH39.05)	2.8
Poly(methyl methacrylate)	RM(LH39.06)	2.8
Poly(ethylene terephthalate)	RM(LH39.07)	2.8
Polyacetals	RMs(LH39.07)	2.8
Silicones	RMs(LH39.10)	2.8
Standard substances for mass calibration	RM	2.9
Acetone-d_6		2.10
Benzene-d_6		2.10
Chloroform-d		2.10
Deuterium oxide		2.10
Dimethyl-d_6 sulphoxide		2.10
Methyl-d_3 alcohol-d		2.10
Tetramethylsilane (TMS)	RM	2.10
Buffer solution with pH 6.88 at 20 °C	pH meter	2.11
Buffer solution with pH 3.57 at 20 °C	pH meter	2.11
Buffer solution with pH 4.00 at 20 °C	pH meter	2.11
Buffer solution with pH 5.00 at 20 °C	pH meter	2.11
2,4-Dinitrophenylhydrazine		3
2,6-Dibromoquinone-4-chloroimide		3
2,6-Dichloroquinone-4-chloroimide		3
4-Dimethylaminobenzaldehyde	Psychotropic substances, etc.	3
Ammonium vanadate	Mandelin's reagent	3
Aurous chloride	Psychotropic substances, etc.	3
Bismuth subnitrate	Dragendorff spray	3
Cadmium potassium iodide	Marme reagent	3
Cadmium iodide	Marme reagent	3
Cobaltous thiocyanate	Scott reagent	3
Cobaltous acetate tetrahydrate	Dille-Koppanyi reagent	3
Ethanal (acetoaldehyde)	Dragendorff spray	3
Fast Blue B (di-6-anisidinetetrazolium chloride)	Fast Blue B solution	3

Reagent	Use	No.
Fast Blue BB salt (4-Benzamide-2,5-diethoxybenzene diazonium, zinc chloride salt)	Marijuana, etc.	3
Isopropylamine	Dille-Koppanyi reagent	3
Lithium sulphate		3
Magenta I (Fuchsine, C.I. basic violet 2)		3
Mercuric chloride	Mayer's reagent	3
Methanal (formaldehyde conc. approx. 37 %)	Marquis reagent	3
Methylrosaniline chloride	Narcotic drugs	3
Molybdic acid (or sodium molybdate as an alternative)	Frohde's reagent	3
p-Dimethylaminobenzaldehyde	Ghamrawy reagent	3
p-Dinitrobenzene	Zimmermann reagent	3
p-Nitroaniline	Psychotropic substances, etc.	3
Picric acid	Psychotropic substances, etc.	3
Platinum chloride (chloroplatinic acid)	Narcotic drugs	3
Potassium 1,2-naphthoquinone-4-sulphonate	Psychotropic substances, etc.	3
Reinecke salt monohydrate (Ammonium tetrathiocyanodiammonochromate)		3
Selenious acid	Mecke's reagent	3
Tetraethylammonium hydroxide		3
Trisodium hexacyanoferrate	Psychotropic substances, etc.	3
Vanillin	Duquenois reagent	3
Standards narcotic drugs and psychotropic substances (morphine sulphate, morphine hydrochloride, codeine phosphate, amfetamine hydrochloride, metamfetamine hydrochloride, etc.)	RMs	3
Cannabis	RM	3
Opium	RM	3
Ammonium thiocyanate (standard solution)		4
Ammonium ferrous sulphate (Mohr's salt) (standard solution)		4
Hydrogen chloride (Hydrochloric acid) (standard solution)		4
Iodine (standard solution)		4
Karl Fischer reagents		4
Nitric acid (standard solution)		4

Reagent	Use	No.
Oxalic acid (standard solution)		4
Potassium cyanide (standard solution)		4
Potassium dichromate (standard solution)		4
Potassium hydroxide (standard solution)		4
Potassium iodate (standard solution)		4
Potassium permanganate (standard solution)		4
Silver nitrate (standard solution)		4
Sodium chloride (standard solution)		4
Sodium hydroxide (standard solution)		4
Sodium thiosulphate (standard solution)	Sucrose, etc.	4
Succinic acid (standard solution)		4
Sulphuric acid (standard solution)		4
Acetic acid	RM(LC22N1d), Hanes A, etc.	4
Copper phosphide	RM(LC28N7)	4
Boric acid	RM(LH25.28)	4
Calcium fluoride	RM(LH25.29)	4
5-[p-(Dimethylamino)benzylidene]rhodanine	Ag, Au	5.1
Alizarin (1,2-Dihydroxyanthraquinone)	Al, In, Th, Zr, F	5.1
Aluminon	Al	5.1
Curcumin (C.I. natural yellow)	B, $B_2O_4^{2-}$, Be	5.1
3,5,7,2',4'-Pentahydroxyflavone (Morin)	Be, Ga, In	5.1
Thiourea	Bi, Se	5.1
Fluorescein	Br^-,BrO^{3-}	5.1
2,7-Dihydroxynaphthalene	$C_2O_4^{2-}$	5.1
Glyoxal bis(2-hydroxyanil)	Ca	5.1
2,2'-Dipyridyl	Cd, Fe(II)	5.1
Aniline	Cl^-, ClO_3^-	5.1
Diphenylcarbazide	Cl^-	5.1
Nitroso-R-salt	Co	5.1
Methylene Blue	Cr(III), Mo, Ta, CrO_4^{2-} Cl^-, ClO_4^-	5.1
Disodium chromotropate (Chromotropic acid, disodium salt)	Cr(VI), Ti	5.1

Reagent	Use	No.
Naphthol Yellow S	Cs, Rb, K	5.1
Cupron (α-Benzoinoxime)	Cu	5.1
Rubeanic acid (Dithiooxamide)	Cu, Ru	5.1
1,10-Phenanthroline	Fe(II)	5.1
Mannitol	Ge	5.1
Quinalizarin	Ge	5.1
Diphenylcarbazone	Hg(I), Hg(II)	5.1
Leucomalachite Green	Ir	5.1
1-Amino-4-hydroxyanthraquinone	Li	5.1
Magneson (4-(4-nitrophenylazo)resorcinol)	Mg	5.1
Hydroxylamine hydrochloride	Mn, drugs, etc.	5.1
Tannic acid	Nb, Ta	5.1
Diethylglyoxime	Ni, Pa, Re	5.1
α-Naphthylamine	NO^{3-}	5.1
Sulphanilic acid	NO^{3-}	5.1
4-Nitrosodiphenylamine	Pa	5.1
Diphenylthiocarbazone	Pb, Zn	5.1
4-Nitrosodimethylaniline	Pt, Rh	5.1
Resorcinol	Pt	5.1
Rhodamine B	Sb, Ga	5.1
Cochineal	Sc	5.1
N,N'-Diphenylbenzidine	Se	5.1
Disodium rhodizonate (Rhodizonic acid, disodium salt)	Sr	5.1
Thymol	Ti, NH_4^+	5.1
Oxine (8-hydroxyquinoline)	U, V	5.1
Malachite Green	W, Ce	5.1
2,6-Diethylaniline	Zn	5.1
Benzyl chloride		5.2
Ligroin	SEM	5.2
Nitrobenzene	Textiles, etc.	5.2
Paraffin		5.2
Styrene	Solvent	5.2

Reagent	Use	No.
α-Naphthol		5.3
β-Naphthol		5.3
2-Chloroethanol		5.3
2,4-Dinitrophenol		5.3
Benzyl alcohol	Perfume	5.3
Butan-1-ol (n-Butanol)	Ethanol (IS)	5.3
Butan-2-ol (2-butanol)	Alcoholic beverages	5.3
Catechol		5.3
Chloral hydrate	Microscope	5.3
Cyclohexanol	Solvent	5.3
Ethylene glycol	Solvent	5.3
Hydroquinone (p-Dihydroxybenzene)		5.3
Isoamyl alcohol	Rum, etc.	5.3
Isobutanol	Alcoholic beverages	5.3
Pentanol (Amyl alcohol)	Rum, etc.	5.3
Pyrocatechol	Tannins	5.3
Pyrogallol	Vitamin E, etc.	5.3
Resorcinol	Tannins	5.3
2,2'-Oxydiethanol (diethylene glycol)	Solvent	5.4
Anisole	Perfume	5.4
Benzaldehyde	Perfume	5.5
Anthraquinone	Oils, etc.	5.6
Anthrone	Colouring agent	5.6
Cyclohexanone	Solvent	5.6
Hexan-2-on (Butyl methyl ketone)	Solvent	5.6
Adipic acid	RM	5.7
Citric acid, monohydrate	RM, drugs, etc.	5.7
Lactic acid	RM	5.7
Maleic acid	RM	5.7
Malic acid	RM	5.7
Oxalic acid, dihydrate	RM	5.7
Succinic acid	RM	5.7

Reagent	Use	No.
Tartaric acid	RM	5.7
Acetic anhydride	Acetylation	5.7
Cinnamic acid	Perfume	5.7
Ethyl formate	Solvent	5.7
Formic acid (Conc. 99 % or more by weight)		5.7
Mercurous acetate	Iodine value, etc.	5.7
Methyl salicylate	Perfume	5.7
Potassium sodium tartrate	Fehling's solution B	5.7
Trichloroacetic acid	Iodine value, etc.	5.7
Trifluoroacetic acid	Acetylation	5.7
Glycine	RM (amino acid)	5.8
L-Alanine	RM (amino acid)	5.8
L-Arginine, HCl	RM (amino acid)	5.8
L-Aspartic acid	RM (amino acid)	5.8
L-Cysteine, HCl	RM (amino acid)	5.8
L-Glutamic acid	RM (amino acid)	5.8
L-Histidine, HCl	RM (amino acid)	5.8
L-Isoleucine	RM (amino acid)	5.8
L-Leucine	RM (amino acid)	5.8
L-Lysine, HCl	RM (amino acid)	5.8
L-Methionine	RM (amino acid)	5.8
L-Ornithine, HCl	RM (amino acid)	5.8
L-Phenylalanine	RM (amino acid)	5.8
L-Proline	RM (amino acid)	5.8
L-Serine	RM (amino acid)	5.8
L-Threonine	RM (amino acid)	5.8
L-Tryptophan	RM (amino acid)	5.8
L-Tyrosine	RM (amino acid)	5.8
L-Valine	RM (amino acid)	5.8
Dinitrophenylhydrazine	Drugs, etc.	5.8
Ethylenediaminetetraacetic acid (EDTA)	Standard	5.8
L- or DL-Ascorbic acid (Vitamin C)	RM	5.10

Reagent	Use	No.
Niacin (Nicotinic acid)	RM	5.10
Nicotinamide	RM	5.10
L- or DL- Panththenic acid	RM	5.10
Pyridoxine (Vitamin B_6)	RM	5.10
Riboflavin (Vitamin B_2)	RM	5.10
Thiamine (Vitamin B_1)	RM	5.10
Vitamin A	RM	5.10
Vitamin D_2	RM	5.10
Vitamin E	RM	5.10
Galactose	RM(LH29.40)	5.11
Mannose	RM(LH29.40)	5.11
Xylose	RM(LH29.40)	5.11
Starch (cassava)	RM(LH11.08)	5.12
Starch (corn)	RM(LH11.08)	5.12
Starch (potato)	RM(LH11.08)	5.12
Starch (wheat)	RM(LH11.08)	5.12
Soya-bean oil	RM(LH15.07)	5.13
Peanut oil (arachis, groundnut)	RM(LH15.08)	5.13
Olive oil	RM(LH15.09)	5.13
Palm kernel oil	RM(LH15.13)	5.13
Cotton-seed oil	RM(LH15.12)	5.13
Safflower oil	RM(LH15.12)	5.13
Sunflower-seed oil	RM(LH15.12)	5.13
Babassu oil	RM(LH15.13)	5.13
Coconut oil	RM(LH15.13)	5.13
Palm oil	RM(LH15.11)	5.13
Colza oil	RM(LH15.14)	5.13
Mustard oil	RM(LH15.14)	5.13
Rape oil	RM(LH15.14)	5.13
Castor oil	RM(LH15.15)	5.13
Corn oil	RM(LH15.15)	5.13
Jojoba oil	RM(LH15.15)	5.13

Reagent	Use	No.
Linseed oil	RM(LH15.15)	5.13
Sesame oil	RM(LH15.15)	5.13
Tung oil	RM(LH15.15)	5.13
Vegetable waxes	RMs(LH15.21)	5.14
Mineral waxes	RMs(LH27.12)	5.14
Acid dyes	RMs(LH32.04)	5.15
Basic dyes	RMs(LH32.04)	5.15
Direct dyes	RMs(LH32.04)	5.15
Disperse dyes	RMs(LH32.04)	5.15
Reactive dyes	RMs(LH32.04)	5.15
Vat dyes	RMs(LH32.04)	5.15
Bergamot oil	RM(LH33.01)	5.16
Geranium oil	RM(LH33.01)	5.16
Ground-nut oil	RM(LH33.01)	5.16
Jasmine oil	RM(LH33.01)	5.16
Lavender oil	RM(LH33.01)	5.16
Lemon oil	RM(LH33.01)	5.16
Lime oil	RM(LH33.01)	5.16
Orange oil	RM(LH33.01)	5.16
Peppermint oil	RM(LH33.01)	5.16
Cellulose acetate (yarn)	RM(ENC54)	5.17
Nylon (yarn)	RM(ENC54)	5.17
Polyester (yarn)	RM(ENC54)	5.17
Polyethylene (yarn)	RM(ENC54)	5.17
Polypropylene (yarn)	RM(ENC54)	5.17
Silk	RM(LC50)	5.17
Wool	RM(LC51)	5.17
Cotton	RM(LC52)	5.17
Flax	RM(LH53.01)	5.17
Hemp	RM(LH53.02)	5.17
Jute	RM(LH53.03)	5.17
Sisal	RM(LH53.04)	5.17

Reagent	Use	No.
Rayon	RM(LH54.03)	5.17
Acrylic fibres	RMs(ENC54)	5.17
Modacrylic fibres	RMs(ENC54)	5.17
Urea	General use	5.18
Soaps (sodium stearate, etc.)	RMs(LH34.01, LH34.02)	5.18
Anionic surface-active agents (sodium alkylbenzene sulphonate, etc.)	RMs(LH34.01, LH34.02), HPLC, etc.	5.18
Cationic surface-active agents (cetyl trimethylammonium chloride, etc.)	RMs(LH34.01, LH34.02), HPLC, etc.	5.18
Non-ionic surface-active agents (polyoxyethylene lauryl alcohol ether)	RMs(LH34.01, LH34.02)	5.18
Agar-agar	RM(LH13.02)	5.18
Pine oil	RM(LH38.05)	5.18
Bovine leather	RM(LH41.01)	5.18
Turpentine oils	RMs(LH38.05)	5.18
Rosin and resin acids	RMs(LH38.06)	5.18
Sublimed sulphur	RM(LH25.03)	6.1
Quartz	RM(LH25.06)	6.1
Quartzite	RM(LH25.06)	6.1
Kaolin	RM(LH25.07)	6.1
Andalusite	RM(LH25.08)	6.1
Bentonite	RM(LH25.08)	6.1
Chamotte	RM(LH25.08)	6.1
Decolourising earths	RM(LH25.08)	6.1
Dinas earth	RM(LH25.08)	6.1
Fire-clay	RM(LH25.08)	6.1
Fuller's earth	RM(LH25.08)	6.1
Kyanite	RM(LH25.08)	6.1
Mullite	RM(LH25.08)	6.1
Silimanite	RM(LH25.08)	6.1
Chalk	RM(LH25.09)	6.1
Barytes	RM(LH25.11)	6.1
Witherite	RM(LH25.11)	6.1

Reagent	Use	No.
Siliceous fossil meals	RM(LH25.12)	6.1
Corundum	RM(LH25.13)	6.1
Emery	RM(LH25.13)	6.1
Pumice stone	RM(LH25.13)	6.1
Slate	RM(LH25.14)	6.1
Ecaussine	RM(LH25.15)	6.1
Marble	RM(LH25.15)	6.1
Travertine	RM(LH25.15)	6.1
Basalt	RM(LH25.16)	6.1
Granite	RM(LH25.16)	6.1
Porphyry	RM(LH25.16)	6.1
Sandstone	RM(LH25.16)	6.1
Dolomite	RM(LH25.18)	6.1
Gypsum	RM(LH25.20)	6.1
Limestone	RM(LH25.21)	6.1
Hydraulic lime	RM(LH25.22)	6.1
Quicklime	RM(LH25.22)	6.1
Slaked lime	RM(LH25.22)	6.1
Soda asbestos	RM(LH25.24)	6.1
Mica	RM(LH25.25)	6.1
Steatite	RM(LH25.26)	6.1
Chiolite	RM(ENH25.30)	6.1
Cryolite	RM(ENH25.30)	6.1
Felspar	RM(LH25.29)	6.1
Fluorspar	RM(LH25.29)	6.1
Leucite	RM(LH25.29)	6.1
Nepheline syenite	RM(LH25.29)	6.1
Nepheline	RM(LH25.29)	6.1
Chlorite	RM(LH25.30)	6.1
Kieserite	RM(LH25.30)	6.1
Perlite	RM(LH25.30)	6.1
Vermiculite	RM(LH25.30)	6.1

Reagent	Use	No.
Earth colours	RM(LC25N2b)	6.1
Ores	RMs(LC26)	6.1
Hydrogen bromide	Bromination	6.2
Hydrogen iodide	Iodination	6.2
Ammonium cobaltous thiocyanate	General use	6.3
Barium carbonate	General use	6.3
Calcium chloride dihydrate	General use	6.3
Chloroauric acid	Drugs, etc.	6.3
Cupric sulphate, anhydrous	General use	6.3
Disodium hydrogenorthophosphate	Drugs, etc.	6.3
Disodium sulphate decahydrate	General use	6.3
Disodium tetraborate decahydrate (Borax)	General use	6.3
Magnesium chloride hexahydrate	General use	6.3
Mercuric chloride	General use	6.3
Potassium bromate	General use	6.3
Potassium bromide	General use	6.3
Potassium chloride	General use	6.3
Potassium chromate	As an indicator, etc.	6.3
Potassium cyanide	General use	6.3
Potassium fluoride	As an indicator, etc.	6.3
Potassium hydrogencarbonate	General use	6.3
Potassium iodate	General use	6.3
Potassium nitrate	General use	6.3
Potassium nitrite	General use	6.3
Sodium bromide	General use	6.3
Sodium cyanide	General use	6.3
Sodium hypochlorite	Fibres, etc.	6.3
Sodium nitrite	General use	6.3
Sodium phosphotungstate	Starch, etc.	6.3
Stannic chloride	General use	6.3
Zinc sulphate	Starch, Hanes A, deproteinizing, etc.	6.3
Reagent	Use	No.

Zinc chloride	Fibre, etc.	6.3
Calcium hydroxide	General use	6.4
Cupric oxide	General use	6.4
Cadmium (high purity)	RM	6.5
Iron, reduced	RM	6.5
Magnesium powder (high purity)	RM	6.5
Manganese (high purity)	RM	6.5
Iron powder (high purity)	RM(LC72)	6.5
Copper (high purity)	RM(LC74)	6.5
Nickel (high purity)	RM(LC75)	6.5
Aluminium (high purity)	RM(LC76)	6.5
Lead (high purity)	RM(LC78)	6.5
Zinc powder (high purity)	RM(LC79)	6.5
Tin (high purity)	RM(LC80)	6.5
Bromine	Drugs, etc.	6.6
Carbon disulphide	Solvent	6.6
Charcoal, activated, powder	General use	6.6
Glass wool	General use	6.6
Iodine	Iodine, etc.	6.6
Platinum wire	General use	6.6

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Chapter 4 : Technical literature and reference books

CONTENTS

	Title	Author	Publisher
1	Analytical Chemistry, 4th Edition	Gary D. Christian	John Wiley & Sons, Inc.
1	ASTM (American Society for Testing and Materials) standards		ASTM
1	BSI (British Standards Institute) standards		BSI
1	Chemical and Process Technology Encyclopedia	D.M. Considine	McGraw-Hill
1	Colour Index		The Society of Dyes and Colour Colourists
1	Concise Etymological Dictionary of Chemistry	S.C. Bevan	Applied Science Publishers
1	Concise Encyclopedia of Biochemistry	T. Scott	de Gruyter
1	Dictionary of Scientific and Technical Terms	S.P. Parker	McGraw-Hill
1	Dictionary of Organic Compounds		Chapman and Halls
1	Encyclopedia of Industrial Chemical Analysis	F.D. Snell	Interscience
1	Encyclopedia of Chemical Technology	Kirk-Othmer	John Wiley & Sons, Inc.
1	Encyclopedia of Science and Technology		McGraw-Hill
1	General Chemistry in the Laboratory, 2nd Edition	J.L. Roberts Jr., et al.	Freeman
1	Handbook of Chemistry	Adolph Lange	McGraw-Hill
1	Hawley's Condensed Chemical Dictionary	G.G. Hawley	Van Nostrand Reinhold Company
1	ISO (International Organization for Standardization) standards		ISO
1	IUPAC (International Union of Pure and Applied Chemistry) standards		Butterworths
1	IUPAC Nomenclature of Organic Chemistry		Butterworths
1	IUPAC Nomenclature of Inorganic Chemistry		Butterworths
1	Molecular Biology and Biotechnology 2nd Edition	J.M. Walker & E.B. Gingold	Royal Society of Chemistry 1988
1	Official Method of Analysis, Association of Official Analytical Chemists (AOAC)		Association of Official Analytical Chemists, Inc.
1	Official Methods of Chemical Analysis		Association of Official Analytical Chemists
1	Quality Assurance Principles for Analytical Laboratory	Frederick M. Garfield	AOAC

	Title	Author	Publisher
1	Standard Methods of Chemical Analysis	Frank J. Welcher	Robert E. Kringer Publishing Co.
1	The Merck Index, 11th Edition		Merck & Co., Inc.
2	Encyclopedia of Polymer Science and Technology	H.F. Mark	John Wiley & Sons, Inc.
2	Encyclopedia of Polymer Science and Engineering, 2nd Edition (1987)		John Wiley & Sons, Inc.
2	Handbook of Common Polymers		Rolf and Scott
2	Identification and Analysis of Plastics		Illife Books
2	Modern Plastics Encyclopedia		McGraw-Hill
2	Plastic Additives & Modifiers Handbook	Jesse Edenbaum	Van Nostrand Reinhold Company
2	Plastics Analysis Guide, Chemicals and Instrumental Methods	A. Krause, etc.	Hanser Publishers
3	Fairchild's Dictionary of Textiles, 6th Edition	I.B. Wingate	Fairchild Publications
3	Guide to The Identification of Animal Fibres, 2nd Edition, 1978	H.M. Appleyard	WIRA
3	Identification of Textile Materials, 9th Edition		The Textile Institute
3	Identification of Vegetable Fibres, 1982	D. Catling & J. Grayson	Chapman and Hall
3	Introductory to Textile Science	Marjory L. Joseph	Holt, Rinehart and Winston
3	Textile Terms and Definitions, 1981		The Textile Institute
4	Analysis of Paper, 2nd Edition	B.L. Browning	Mercel Dekker, Inc.
4	Handbook of Pulp and Paper Technology, 2nd Edition	Kenneth W. Britt	Van Nostrand Reinhold Company
4	The Dictionary of Paper, 4th Edition, 1980		American Paper Institute
5	The Practical Identification of Wood Pulp Fibres	R.A. Parham, R.L. Gray	TAPPI Press
5	Wood Structure and Identification, 2nd Edition, Syracuse Wood Science Series 6, 1979	H.A. Core, W.A. Cote, A.C. Day	Syracuse University Press
6	Bailey's Industrial Fat and Oil Products		John Wiley & Sons, Inc.
6	Chocolate, Cocoa and Confectionery Science & Technology 3rd Edition (1989)	Bernard W. Minifie	Van Nostrand Reinold
6	Encyclopedia of Food Science		Food Technology and Nutrition

	Title	Author	Publisher
6	Food Composition and Nutrition Tables (in three languages)		Wissenschaftliche Verlagsgsesellschaft GmbH
7	Analysis of Drug Manual		Drug Enforcement Administration in USA
7	Basic Tests for Pharmaceutical Dosage Forms		WHO
7	Basic Tests for Pharmaceutical Substances		WHO
7	Clarke's Isolation and Identification of Drugs		The Pharmaceutical Press
7	Clandestine Manufacture of Substances under International Control		UNDCP
7	Cosmetic Bench Reference		Allured Publishing Corp.
7	CTFA International Cosmetic Ingredients Dictionary	Nikitakis, J.M.MC.Ewen, G.N. Eds	The Cosmetic Toiletry and Fragrance Association
7	Dictionary of Drugs		Chapman and Halls
7	European Pharmacopoeia		Maisonneuve S.A.
7	Flavour and Fragrance Materials		Allured Publishging Corp.
7	Flavour Science and Technology, 1990	Y. Bessière and A.F. Thomas	John Wiley & Sons, Inc.
7	Glossary of Terms for Quality Assurance and Good Laboratory Practices		UNDCP
7	Instrumental Data for Drug Analysis		Elsevier
7	Multilingual Dictionary of Narcotic Drugs and Psychotropic Substances under International Control		The United Nations
7	Perfume and Flavor Chemicals	S. Arctander	Allured Publishing
7	Perfume and Flavor materials of Natural Origin	S. Arctander	Allured Publishing
7	US Pharmacopoeia		United States Pharmaceutical Convention
7	USP Dictionary of USAN and International Drugs Names		United States Pharmaceutical Convention
7	Good laboratories practices in governmental drug control laboratories and Training programme in drug analysis		WHO
7	Validation of analyical procedures used in the examination of pharmaceutical materials		WHO
8	A Concise Introduction to Ceramics (1991)	George C. Phillips	Van Nostrand Reinhold

	Title	Author	Publisher
8	Industrial Minerals and Rocks, 6th Edition		Society of Mining, Metallurgy and Exploration, Inc.
8	Metals Handbook		American Society for Metals
9	An Infra-red Spectroscopy Atlas for the Coatings Industry, 2nd Edition		Infra-red Spectroscopy Committee of the Chicago Society for Coating Technology, pub.
9	Atlas of Polymer and Plastic Analysis	Deiter O. Hummel	Hanser Publishers
9	Infra-red Spectra Handbook of Common Organic Solvents		Sadtler Research Laboratories
9	The Infra-red Spectra Atlas of Monomer and Polymer		Sadtler Research Laboratories
9	The Infra-red Spectra Atlas of Surface Active Agents		Sadtler Research Laboratories
9	The Infra-red Spectra Handbook of Mineral and Clays		Sadtler Research Laboratories
9	The Infra-red Spectra Handbook of Inorganic Compounds		Sadtler Research Laboratories
9	UV and IR Spectra of Pharmaceutical Compounds		Association of Official Analytical Chemists
10	Chromatographic Science Series (Vol.II)	K.H. Altgelt and T.H. Gouw	Marcel Dekker, Inc.
10	Chromatography : Concept and Contrasts	James M. Miller	John Wiley & Sons, Inc.
10	Handbook of Analytical Derivatization Reactions	Daniel R. Knapp	John Wiley & Sons, Inc.
10	Handbook of Chromatography General Data and Principles	G. Zweig and J. Sherma	CRM Press
10	High Performance Liquid Chromatography	Csaba Horvath	Academic Press
10	Modern Practice of Gas Chromatography, 2nd Edition	Robert L. Grob	John Wiley & Sons, Inc.
10	Practice of High-Performance Liquid Chromatography, 1986	H. Engelhardt	Springer-Verlag
10	Practice of Thin-Layer Chromatography	J.C. Touchstone	John Wiley & Sons, Inc.
10	Quantitative Analysis Using Chromatographic Techniques, 1987	E. Katz	John Wiley & Sons, Inc.
10	The Analysis of Gases by Chromatography	C.J. Cowper and A.J. DeRose	Pergamon Press
10	Thin-Layer Chromatography, 2nd Edition	Bernard Fried and Joseph Sherma	Marcel Dekker, Inc.
11	Enzyme Handbook, Supplement I, 1974	T.E. Barman	Springer-Verlag

	Title	Author	Publisher
11	Handbook of Adhesives	Irving Shiest	Van Nostrand Reinhold Company
11	Handbook of Industrial Chemical Additives, 1991	Michael Ash	VCH Publishers
11	Industrial Chemical Thesaurus, 1992	Michael Ash	VCH Publishers
11	MC Cutcheon's Functional Materials, 1994	MC Cutcheon's Division	MC Publishing Co.
11	MC Cutcheon's Emulsifiers and Detergents, 1994	MC Cutcheon's Division	MC Publishing Co.
11	Paint Handbook	Guy E. Weismantel	McGraw-Hill
11	Refractory Materials Chemical Technology Review No. 76 (1976)	G.B. Rothenberg	Noyes Data Corporation
11	Rubber Worl Magazine's 1995 Blue book		Lippincot & Peto Inc.
11	The Pesticide Manual 9th Edition	CH R. Worthing	The British Crop Protection Council

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