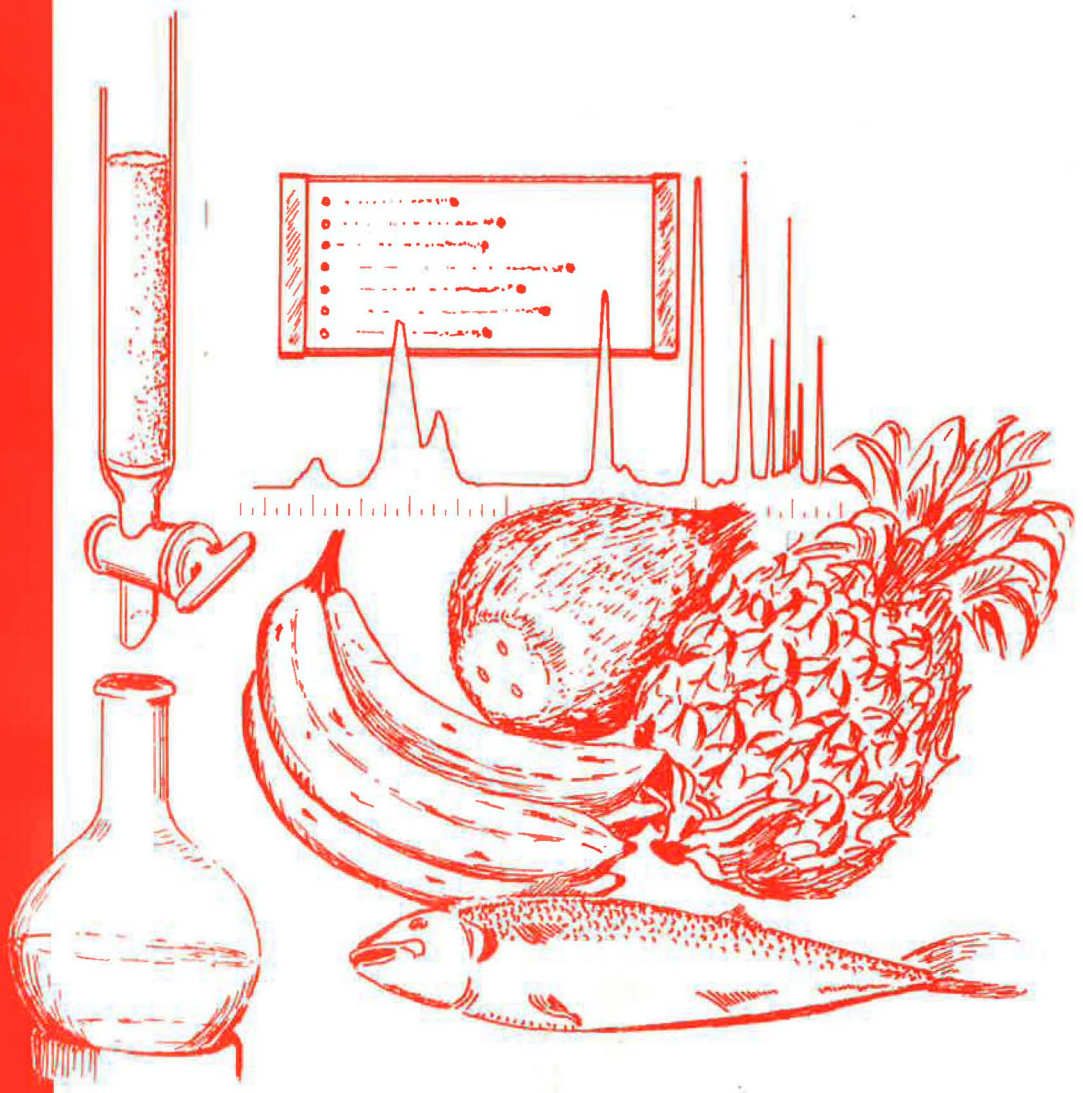
A guide to the establishment and maintenance of pesticide laboratories in developing countries (NRI Bulletin No. 28)

Natural Resources Institute

A GUIDE TO THE ESTABLISHMENT AND MAINTENANCE OF PESTICIDE LABORATORIES IN DEVELOPING COUNTRIES

Bulletin No. 28



Overseas Development Administration

NATURAL RESOURCES INSTITUTE

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A GUIDE TO THE ESTABLISHMENT AND MAINTENANCE OF PESTICIDE LABORATORIES IN DEVELOPING COUNTRIES

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The Natural Resources Institute (NRI) is the scientific arm of Britain's Overseas Development Administration. NRI's principal aim is to increase the productivity of renewable natural resources in developing countries through the application of science and technology. Its areas of expertise are resource assessment and farming systems, integrated pest management, and food science and crop utilization.

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SUMMARY

The difficulties in establishing and maintaining pesticide laboratories, particularly residue laboratories, in developing countries are addressed in an attempt to arouse a greater awareness of the commitment and resources required for successful operation. Accommodation, staffing, laboratory services, equipment and materials are all considered in detail and model specifications, including full equipment lists, are provided for both formulation and residue laboratories.

RÉSUMÉ

Les problèmes soulevés par la création et l'entretien de laboratoires de pesticides et surtout de laboratoires de pesticides rémanents dans les pays en développement sont envisagés dans le but d'être plus conscients de la résolution et des ressources nécessaires pour assurer le succès des opérations. Locaux, personnel, services de laboratoires, équipement et matériaux: tout est envisagé en détail et des spécifications modèles sont fournies pour les laboratoires chargés tant de composition de produits que de leurs résidus.

RESUMEN

El artículo estudia las dificultades encontradas en la creación y mantenimiento de laboratorios de pesticidas — particularmente, laboratorios de residuos — en países en desarrollo, en un esfuerzo por despertar una mayor toma de conciencia sobre la dedicación y recursos necesarios para que su funcionamiento sea satisfactorio. El autor examina detalladamente los requisitos de espacio, personal, servicios de laboratorio, equipo y materiales. También se incluyen especificaciones modelo, tales como listas de equipo, tanto para laboratorios de formulación como de residuos.

A guide to the establishment and maintenance of pesticide laboratories in developing countries

INTRODUCTION

Pesticide analysis is accepted as being one of the most demanding forms of chemical analysis in terms of both labour and materials. The capital cost of establishing a pesticide laboratory is high (*see* Appendices A, B1, B2 and B3) as are the recurrent costs for consumable materials and instrument maintenance, a consideration often overlooked in the provision of funds. Similarly, careful thought needs to be given to the manpower requirements in terms of qualifications, experience and training as well as consideration of the staffing levels required for the projected throughput and nature of the work.

Laboratories in developing countries often experience particular difficulties and, regrettably, there are numerous examples of laboratories which have been established in good faith, often with external assistance, functioning inefficiently or ceasing to function at all. There are many reasons for this, but in general, laboratory 'failure' can be attributed to one or more of the following:

- poor long-term planning;
- inadequate appreciation of the complexity of the laboratory operation;
- inadequate commitment of resources including finance and manpower;
- inadequate laboratory facilities and services.

It is the intention of this guide to define the requirements for the establishment of a pesticide laboratory with particular reference to developing country conditions and to give the reader a greater appreciation of the difficulties involved with and of the commitment required for the establishment and maintenance of such a laboratory.

In the text and in the appendices, specifications are given for laboratory accommodation, services, equipment, general apparatus, glassware and reagents required. Full laboratory specifications for formulation (*see* Appendix A) and residue laboratories at three levels of operation (*see* Appendices B1, B2 and B3) are provided. These model specifications are designed to 'stand alone' and repetition of format and content is inevitable although reflecting a different level and nature of operation.

It is beyond the scope of this guide to detail laboratory working practices, analytical methodology, or to consider the implications of analytical quality assurance, although some general points are made (*see Analytical Methodology*) and some useful references are provided in Appendix C.

PESTICIDE ANALYSIS

The size and nature of the laboratory will depend upon the projected work programme in terms of the types of sample that it is expected to analyse,

together with an assessment of the number of samples involved. The analytical programme can be broken down into two main categories:

- the analysis of pesticide residues;
- the analysis of pesticide formulations.

Each of these requires a particular expertise and makes separate demands on the laboratory facilities available. These facilities will be considered in detail later in this guide. It is important to recognize at an early stage the differences between these forms of analysis and the necessity to separate, physically, the work areas and laboratory equipment used.

Pesticide residue analysis is the analysis of trace levels of a chemical in a material arising from the application, directly or indirectly, of a pesticide. Residue levels are in the μg range and require sensitive and selective methods of determination.

Formulation analysis is at the opposite end of the spectrum, being the examination of a product as supplied for use in pest control for its physical and chemical properties. As such, examination of the product can be required in its concentrated form or in a diluted form as directed for application purposes. In either case the pesticide is present in a quantity greater by several orders of magnitude than that present in a sample for residue analysis.

The requirement to undertake either of these analytical functions can be associated with most laboratories in the fields of agriculture, veterinary science and public health, but residue analysis and formulation control should never be undertaken in a multi-purpose laboratory. A separate laboratory arrangement is necessary in each case.

SEPARATION OF FUNCTIONS

The problem of accidental contamination of residue samples from a sample of a concentrated formulation is self-evident and must be avoided. The consequences of the contamination of a sample, besides the waste of a considerable amount of time and materials (assuming that the samples can be replaced, which is not always the case) will result in the production of grossly misleading results which could have commercial implications, could result in the misdirection of research or agricultural programmes or even in unjustified legal action. Complete physical separation of the two forms of analysis is the only way to avoid the possibility of such contamination.

Circumstances are rarely ideal and unless a purpose-built laboratory suite has been provided so that the analyses can be separated, the analyst will invariably be called upon to carry out the operations in parallel in the same room and/or with the same equipment. In such circumstances contamination **cannot** be avoided. It will not be the fault of the analyst, but of the organization and allocation of work. The responsibility must lie with the parent organization in consultation with specialist advisers to recognize the problem and to act accordingly—**either** by providing separate facilities, as described later, **or** to undertake one operation only. Priority should always be given to the quality control of pesticide formulations.

In many cases, financial limitations become the overriding consideration and funds cannot be allocated for the provision of the separate facilities required. This problem is well recognized, but circumstances should not be allowed to dictate that the laboratory serve a dual function without the correct facilities.

THE ESTABLISHMENT OF A PESTICIDE LABORATORY

The role and overall function of the laboratory must be clearly defined at the planning stage. The planning must take into account the nature of the samples that it is proposed should be analysed, together with a realistic forecast of the

number of samples of each type. This basic information is required in order to determine:

- the size and design of the laboratory complex;
- the external services required;
- the nature and quantity of the laboratory equipment, including service and maintenance facilities;
- the consumable materials required for the initial commissioning of the laboratory and the anticipated annual cost of their replenishment;
- the level and size of staffing and expertise required for this level of operation;
- other support services (e.g., access to library facilities).

Financial planning must also make allowance for the routine maintenance of laboratory equipment and facilities, the replacement of outdated and unserviceable items of equipment and the purchase of new items to take into account changes in the work pattern and the development of new techniques.

Each of these considerations is examined in more detail below. Firstly however, it may be of benefit to examine briefly the procedures involved in pesticide analysis.

Pesticide residue analysis

As defined earlier, pesticide residue analysis is the analysis of trace amounts of active ingredients or their breakdown products in a commodity arising from the application, directly or indirectly, of a pesticide. The analytical sample can reflect a wide range of activities—agriculture, horticulture, veterinary science, public health, environmental protection or a 'market basket' survey to study residue levels in locally available or imported foodstuffs. The defined role of the laboratory will determine the areas from which samples can be expected.

To identify the pesticides(s) present and to determine the quantities present it is necessary to:

- ensure that the sample is homogeneous and representative;
- extract the sample by a suitable procedure after initial sample preparation
  - — washing/peeling if required
  - — chopping/grinding to suitable form
  - — sub-sampling;
- separate the pesticide(s) from other co-extractives which may interfere with the analysis and prepare the sample extract for the final analysis;
- identify the pesticide(s) present, using confirmatory techniques as necessary, and quantify the residues present.

These operations require the attention of skilled, well-trained analytical chemists and the provision of suitable equipment. The complexity of the individual operations will vary depending upon the nature of the substrate and the pesticide(s), and the degree of the removal of co-extractives from the sample extract ('clean-up') that is required. Sample extracts with little co-extractive interference will require less clean-up, with consequential savings of time and consumable materials, than extracts with high fat or lipid content, for example, oilseeds and dried/smoked fish. Laboratory capacity required and running costs are thus dependent on the nature of the programme.

Pesticide formulation analysis

Analysis of pesticide formulations covers both testing to ensure that the formulation meets the physical specifications required, and chemical examination to determine active ingredient content, presence of undesirable impurities and such factors as pH which affect stability of the active ingredient.

Physical testing involves the determination of a range of properties including:

- freezing point
- melting point

- insoluble material
- flowability
- particle size range
- suspensibility
- wettability
- foaming
- flashpoint
- low temperature stability
- emulsion stability
- density after compaction
- rate of release of active ingredient
- heat stability.

This list is not meant to be comprehensive and other tests may be required, although it does serve to illustrate the range of physical determinations. The number and nature of tests to be carried out on any formulation are determined by its type, for example whether it is a dust, a wettable powder, an emulsifiable concentrate, and so on.

Chemical testing of products is generally confined to the establishment of acidity or alkalinity and the determination of active ingredient content (including after testing for heat stability and storage stability).

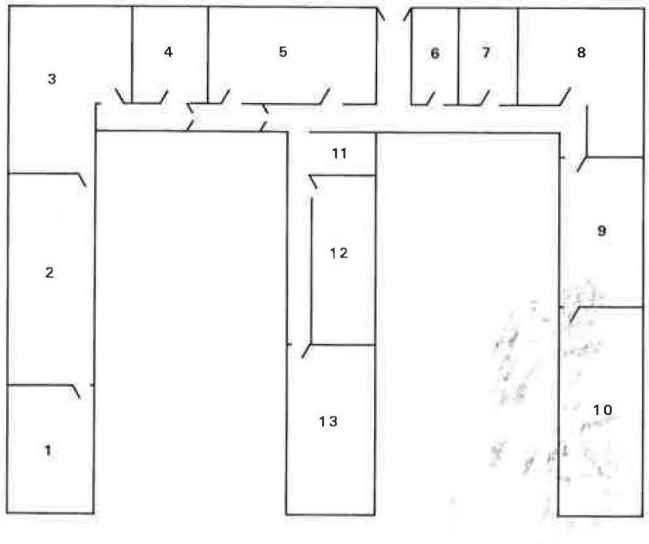
All of these tests require specialized equipment and the provision of good, well-ventilated laboratory facilities. Air conditioning will also be essential for most laboratories.

LABORATORY REQUIREMENTS

Accommodation

If it is intended that the laboratory complex be designed so that both formulation analysis and residue analysis can be carried out, then the initial consideration should be whether or not it is possible to provide facilities in separate buildings. This is obviously the most appropriate arrangement for the minimization of cross-contamination. If this is not possible then an alternative approach is to construct a suitable complex possibly of the pattern illustrated in Figure 1. This allows for the use of certain limited, common facilities in a 'clean' area with office facilities central to each activity.

Figure 1



Room schedule

1 Formulation laboratory, Room 1
2 Formulation laboratory, Room 2
3 Formulation laboratory, Room 3
4 Reference standards store
5 Instrument room (1)
6 Store room
7 Instrument room (2)
8 Residue laboratory, Room 1–sample store
9 Residue laboratory, Room 2–sample extraction
10 Residue laboratory, Room 3–sample clean-up
11 Lockers/washroom
12 Office
13 Office

(windows not shown)

It is one thing to design the laboratory complex under 'ideal' conditions, but often the laboratory manager is faced with the task of having to modify existing accommodation because of budgetary considerations. In such cases compromises in the design may have to be made, although the principle of separation of the formulation and residue activities must not be sacrificed.

In considering laboratory design it is important to ensure that the overall design matches the need and the nature of the department or organization of which it is an integral part. This may mean looking at activities outside the laboratory or the laboratory complex itself but which could have a direct impact upon it. For example, the situation of a laboratory located on an agricultural or veterinary research station where trials involving the application of pesticides are undertaken might be considered. Depending upon the nature and magnitude of the trials, fairly large quantities of pesticides may be being used or stored. These activities provide both a source of analytical samples and a source of contamination which can be spread by a variety of mechanisms:

- inadequate pesticide storage facilities;
- storage facilities too close to the laboratory;
- on the clothes of field workers bringing samples for analysis;
- on the clothes of laboratory workers moving between laboratories;
- by field workers passing through corridors/doorways adjacent to the laboratory.

The risk from all of these can be minimized with some thought and common sense. The storage facility should be made secure, well-ventilated and sited as far away from the residue analysis laboratory as space and other considerations permit. Contamination spread by individuals can be minimized by education—often workers do not realize that they can spread contamination in this way. Many research stations have strict codes of practice covering such personal hygiene and insist that all 'outdoor' clothing is changed before the worker enters the laboratory/office accommodation. Similarly, laboratory workers should change their laboratory coats or overalls before entering a different work area. A little forethought at the planning stage can facilitate these operations by allowing for a small changing room, with washing facilities, to be incorporated in the design and through which field workers must pass when entering the building. It is also worth providing a small sample reception point where field samples can be deposited—perhaps through a hatch accessible from the laboratory, without the worker having to enter the building.

Accommodation for the storage of chemicals must not be overlooked. Although a certain number of such materials can be kept in the laboratories, there will be the need for the storage of additional supplies, particularly where there are likely to be delays in restocking, and large quantities have to be obtained at any one time. There must be careful segregation of reactive chemicals to minimize hazards and, ideally, solvents should be kept in a separate, ventilated store. Depending on climate, precautions may need to be taken to ensure that reactive chemicals and solvents are not subjected to high ambient temperatures.

Laboratory layout

Internal laboratory design is often a matter of personal preference although consideration of the work pattern and of the workload will determine the facilities required and, to a certain extent, the layout of those facilities. The amount of space available and its shape will also affect the internal layout. An example of laboratory design and internal layout has been given in Figure 1 showing basically separated activities, but allowing for the use of certain shared facilities. This design can be expanded to take the work pattern into account, but the minimum requirement should allow for laboratory space as defined below.

Residue laboratory

The basic provision for a residue laboratory should be a suite of three rooms to separate the three main stages of analysis:

- sample preparation and extraction;
- sample clean-up;
- determination of residues (instrument room).

Further sub-division is desirable if conditions permit to allow for a separate weighing room with a vibration-proof balance table and a room with deep-freeze facilities for the reception and storage of samples. Partitioning of one of the main rooms is acceptable, with care, although deep-freeze units can occupy a great deal of space and ideally should be sited outside the laboratory. Where large numbers of samples are anticipated the provision of a walk-in cold store should be considered, although such stores can be expensive to construct.

Provision must also be made for the storage of the reference library of pesticide standard concentrates, which must be sited outside the residue laboratory. Storage could be associated with the formulation laboratory if one exists. Storage of the dilute working solutions does not constitute a problem and they can be kept within the main laboratory complex.

The need for chemical storage space outside the laboratory has already been described, but additionally there will be a need for the storage of in-laboratory supplies for immediate use. Lockable cupboards should be used wherever possible, with solvents being kept on trays in metal, lockable cupboards.

Formulation laboratory

Laboratory requirements for a pesticide formulation laboratory are for a minimum of one laboratory with a separate instrument room. Suggested spatial requirements are detailed in Appendix A. Further sub-division of the main laboratory can be useful, if space permits, and there should be a separate balance room.

Pesticide reference materials suitably contained (*see* p. 17) can be stored in the laboratory, although if this is to be a common stock for the formulation laboratory and for a residue laboratory then siting outside the laboratory is recommended. Fume cupboards will be required in the formulation laboratory as well as in the residue laboratory.

If design permits, a floor drain is recommended to allow for washing down in the case of accidental spillage of a pesticide concentrate. Care would need to be taken that the drain emptied into a trap or a soak-away sited so that no contamination of ground water or crops could occur.

Chemical storage space both within the laboratory and outside it must be provided as for the residue laboratory, although the nature of the materials stored will be slightly different, with smaller quantities of flammable solvent, but possibly greater quantities of acid being used in the formulation laboratory.

Laboratory services

External services can prove to be a major problem in some countries. Power supplies are prone to disruption, sometimes to the extent of becoming routine, and are subject to frequent voltage fluctuations. Water supplies, although generally not limited, are often at low pressure and at a supply temperature in the range of 20–30°C in tropical countries.

Electricity supplies

Modern laboratories use relatively large amounts of electrical equipment and the overall power consumption can be high. Some of the equipment is in

transient use, but a proportion of it, for example, laboratory ovens, refrigerators/freezers, balances and gas–liquid chromatographs, must be run continuously. With refrigerators/freezers and the need to preserve stored materials this is self-evident, but it is equally critical for the stability of modern, sophisticated analytical equipment that the power supply be stable, uninterrupted and maintained 24 hours a day. The power supply must:

- be capable of meeting the full demands of the laboratory, including air-conditioning where necessary;
- be stable and not subject to voltage fluctuations. If voltage fluctuations are likely then a voltage stabilizer must be installed for those circuits operating laboratory equipment, for example, gas-liquid chromatographs.

The provision of an auxiliary power supply is essential where cuts in the supply are of a regular or frequent nature; a modern laboratory cannot function without power and to eliminate costly down-time, the potential loss of analytical samples and instrument instability or damage, the cost of a stand-by generator together with fuel costs should be included in the commissioning budget for the laboratory. The output capacity of the generator will reflect the size of the laboratory but is likely to be in the range of 20–60 KVa.

Water supplies

Pesticide laboratories tend to use fairly large volumes of water and again it is important that the supply is not subject to interruption. Sample extraction is, with few exceptions, carried out using organic solvents, as are the clean-up and separatory stages. Most of these solvents are highly flammable and there are very obvious safety implications if the cooling water supply fails during solvent reflux or distillation – common operations in the course of an analytical determination.

For adequate solvent condensation during reflux or evaporation it is important that a reasonable flow (not less than 5 litres/min) of water at a temperature of not more than 18°C be maintained. As indicated earlier, the temperature of water supplied to laboratories in countries with a tropical climate can be in the range of 20–30°C, particularly where a roof head-tank is fitted. Flow rates may also be relatively low. This is recognized to cause problems with the condensation of solvent vapours and also often precludes the use of water-generated vacuum – a safer alternative to the use of electric vacuum pumps for vacuum solvent distillation. One approach is to install a pump in the supply line to enable a greater operating pressure and rate of flow. This will also have the effect of lowering, by a few degrees, the temperature of the water.

However, the preferred solution is to include a chiller unit in the pressurized supply line, and recirculating systems with a built-in cooler are commercially available for this purpose. The system used should be designed to include all outlets for use with condensing units on the recycling circuit with other pressurized outlets flowing to waste, and in certain situations there may be the need to vent solvent vapour from the water (for example, in the case of trapped vapour from a vacuum distillation using water-generated vacuum). Two approaches may be considered: cooling a continous flow through the laboratory, or cooling and recirculating the mains supply; the latter is the most practical and effective.

The main considerations are: the total flow and volume of water involved, the output pressure required from the circulator, and the degree of cooling required. The outlets to the washing-up sink or for the generation of vacuum should be left on the open mains supply, although if the mains pressure is low there may be the need for a line pump to supplement the pressure. The number of outlets operated from the recirculated supply can be readily

calculated and the overall maximum flow determined. Similarly the determination of the temperature of the 'normal' mains water supply can readily be made and the degree of cooling established. Manufacturers of such pumps, and there are many world-wide, will provide quotations of cost and ready advice if these data can be specified. A generous allowance should be made for future expansion of the system when preparing the specification, rather than tailoring it for the exact calculated need.

In addition, provision should be made, as in Appendices A, B1, B2 and B3, for a small number of portable individual cooling and recycling units for emergency use.

Gas supplies

A supply of compressed gases will be needed for the laboratory for use with analytical equipment and also for 'bench' operations. Bench requirements will normally be limited to compressed air although in certain cases nitrogen may be needed. Analytical equipment can require a wider range of gases, as listed below, although the exact needs cannot be established until the equipment is defined, and not all of those listed may then be required.

- Nitrogen – oxygen-free (< 4 p.p.m. oxygen)
- Air
- Oxygen
- Hydrogen
- Helium

} for gas chromatography and gas chromatography/mass spectrometry

It is important at the design stage to ensure that provision is made for the pipework for the supply of the desired gases. The nature of this will depend upon the range of gases, the flow rates and overall consumption, and the supply mechanism. Some, if not all, of the gases will be provided from cylinders and for safety reasons these should be located outside the laboratory, necessitating a 'feed-line' from the supply source to the internal network. The cylinders should be contained in a separate store outside the laboratory building and shielded from direct sunlight, rain and extremes of temperature. The store should be secure and accessible only to authorized personnel, but should be located conveniently for the delivery of cylinders when fresh stocks are required.

Some gases (air, hydrogen and nitrogen) can alternatively be supplied from generators and this approach may significantly affect the requirements for pipework. The generators/compressors, although a little bulky, will generally fit beneath or beside most laboratory bench units and can be contained within the laboratory complex. This system has a number of benefits particularly where supplies of these gases are limited or very expensive; some countries have to import gas in cylinders and when freight charges are included this is an extremely costly and uncertain means of supply. Recent developments in gas generation systems have made them of particular value to users of gas-liquid chromatographic systems and their use is recommended wherever possible. Gas supply system specifications are given below, both for conventional supplies from a cylinder source and for gas generation systems.

Gas purification

Irrespective of the source of supply, gas purifiers must be fitted in the lines between the supply and the gas chromatograph. Each gas feed must pass through a moisture filter and in addition the nitrogen supply should pass through an oxygen trap. These precautionary measures are essential to preserve the safe and effective working of the analytical equipment at high sensitivity. The purifiers must be changed regularly and frequently to prevent saturation and it is recommended that filtration systems incorporating visible indicators be used, which change colour when the purifier requires changing. One

disadvantage used to be that the indicating systems were not designed for recharging or repacking, but this is now possible with some models. There is a small recurrent cost but this is considered to be worth while.

Cylinder source supply

The gas cylinders should be sited outside the laboratory with feed pipes running into the laboratory and forming a network for distribution to the required points. The cylinders should be secured in racks and shielded from direct sunlight and rain. Ideally they should be in a locked store or compound to prevent tampering.

Where the gas flow requirements are large, a manifold enabling the simultaneous use of two or more cylinders should be used. For a laboratory using just two or three gas-liquid chromatographs this should not be necessary but the change-over rate will be fairly high – perhaps a fresh cylinder every ten or twelve days will be necessary. If the change-over from one cylinder to another causes disruption or other difficulties, a change-over manifold can be fitted. This manifold is connected to two or more cylinders of which only one is on-line at any given time, and as that cylinder empties the line supply is changed, manually or automatically, to a different cylinder. The change-over is thus effected without any line flow disruption. The use of such a system can prevent instrument down-time caused by the accidental failure to change a cylinder.

Each cylinder or manifold must be fitted with a pressure control gauge with which to set the line pressure. For safety reasons this gauge must be at the cylinder head and not lower down through the supply system. Gauges must be kept clean and grease-free. The pressure setting and hence the specification for the gauge itself will depend on the requirements of the system and, ultimately, the operating pressure of the gas chromatographs. This operating pressure varies from make to make and depends on the nature of the instrument pneumatics. In general the following supply pressures should be more than adequate:

nitrogen – 6.9 bar (100 psig);
hydrogen– 3.45 bar (50 psig);
oxygen – 2.1 bar (30 psig);
air – 3.45 bar (50 psig);
helium – 6.9 bar (100 psig).

It should be stressed that gas-liquid chromatographic systems will not require supplies of all of the gases listed above but the need will depend upon the makes of instrument in use and on the detection systems fitted:

(i) carrier gas will normally be **nitrogen** or **helium** (argon/methane mixtures are sometimes used with electron capture detectors (ECDs), but nitrogen is an adequate alternative and is to be preferred for overseas laboratories where supplies of argon/methane will be both difficult to obtain and expensive);
(ii) for electron capture detectors, **nitrogen** will be required;
(iii) for the flame photometric detector, **hydrogen** and **air** will be required. In addition, with some detector designs **oxygen** may be needed;
(iv) the flame ionization and nitrogen-phosphorus detectors will both require supplies of **hydrogen** and **air** only;
(v) the ion trap detector (ITD) requires **helium** only.

Detection systems (ii) – (iv) can use either nitrogen or helium as the carrier gas, although if helium is used with an ECD a make-up supply of nitrogen to the detector will be required. System (v) can only use helium as the carrier and care must be taken, if purchase of an ITD is considered, to ensure that helium supplies are available and that the cost is not prohibitive.

Gas supplies should normally be within the following specification:

	Oxygen	*Moisture*	*Hydrocarbons*
● nitrogen	< 4 p.p.m.	< 5 p.p.m.	< 5 p.p.m.
● helium 99.998% purity or better (for ITD)	< 1 p.p.m.	< 1 p.p.m.	< 1 p.p.m.
● hydrogen	< 2 p.p.m.	< 2 p.p.m.	< 5 p.p.m.
● air	–	< 5 p.p.m.	< 5 p.p.m.
● oxygen	–	< 5 p.p.m.	< 5 p.p.m.

All piped supplies should run through clean, high-purity tubing; copper is the material generally used. Joints must be carefully welded and solder must not be used, as with time this can produce contaminants which will particularly affect electron capture detectors. It is recommended that on/off valves be fitted where the supply pipes enter the laboratory so that supplies can be turned off in case of need or emergency. Pipe outlets should be capped for cleanliness whilst awaiting connection.

The cost of piping installation varies with the complexity of the distribution network and is difficult to estimate. These costs should generally be included in the construction or conversion costs.

Gas generation systems

Hydrogen generators Hydrogen generators have been commercially available for many years but suffered in popularity because of problems with unreliability, although this was more often due to insufficient service and maintenance rather than to an inherent fault in design. Improvements in design and associated ease of maintenance have now made them a viable proposition for use with gas chromatography and maintenance is minimal, imposing little demand on laboratory staff. Units are available giving a choice of output capacities with a maximum of about 300 ml/min and with operating pressures of up to 60 psig. The purity of the gas is comparable to that obtainable from cylinders, and in some cases is better.

Generation principles vary with design but the models employing a palladium membrane elecrolytic cell are particularly effective and the operation is quite clean.

This design is recommended.

Nitrogen generators Nitrogen generators are a fairly new concept and operate on the principle of passing compressed air, produced by an associated oil-free compressor, through a carbon molecular sieve which physically separates oxygen and other trace contaminants, allowing pure nitrogen to pass through and into a holding reservoir. When the carbon molecular sieve is saturated with oxyen and other materials it is vented to atmosphere and the contents discharged. It is then repressurized and the cyclic process continued. The technique is known as pressure swing adsorption.

Systems commercially available (in the United Kingdom, from Nitrox Ltd) come in a range of ouput capacities from 750-4000 ml/min and with an operating pressure of 4.5 bar. Other capacity units can be supplied to customer specification.

The manufacturer's technical specification for generated nitrogen claims less than 10 p.p.m. oxygen, moisture, hydrocarbons and carbon dioxide.

The maximum ambient operating temperature for the units is approximately 30-35°C. Air conditioning is advised to keep the equipment within these operating temperatures for most tropical countries.

A 2000 ml/min unit has been evaluated by NRI over a period of two years and very satisfactory results obtained. The nominal purity specification for the unit was not as good as for cylinder supplies. However, in practice no adverse effects were noticed and instrument stability and response actually improved;

consumption of in-line filters did not increase and these in fact were found to need changing less often than with cylinder supplies.

The operating pressure of the generator is less than the 6.9 bar recommended above, although for many instruments the ouput of 4.5 bar is still quite satisfactory. The major problem that will be encountered is that the gas-liquid chromatography (GLC) flow controllers will be calibrated for a given inlet pressure and at a lower pressure will require re-calibration to read the correct carrier gas flow rates. In general this does not prove to be a difficulty.

It is recommended that nitrogen generators be used wherever possible, although it is suggested that a back-up cylinder of gas be available in case of generator failure because of power supply difficulties. Automatic change-over units are available which operate as the pressure drops and one of these on-line and permanently coupled to both the generator and to a cylinder will meet most eventualities.

Maintenance is normally limited to the overhaul of the compressor, perhaps annually, or as directed by the manufacturer.

Air generators The term generator is misleading as the systems are basically compressing units with an associated air reservoir. Both oil and oil-free compressors are available, but the oil pumps do produce traces of oil in the air supply which must be removed on a carbon filter, and it has been suggested that the oil can impede the removal of moisture particularly where the primary removal is by refrigeration. There is a wide range of pumps commercially available with different output capacities and/or reservoir sizes. The unit of choice will depend upon ouput required but for two or three gas-liquid chromatographs using a typical range of detectors, a unit with a reservoir of 25 litres or an output capacity of 1 l/min should be adequate. Outlet pressures of 6.9 bar or more are normal and these are generally controllable from the pump outlet.

Laboratory studies at NRI have shown the major difficulty with these systems to lie with the purification of the gas, particularly with drying. The Nitrox system, which employs a pressure swing system and gas purification through carbon and alumina beds, has been found to be the most effective and this system is recommended.

The manufacturer's specification for this system claims that the air contains less than 10 p.p.m. of moisture and hydrocarbons.

Combined nitrogen/air generators Nitrox Ltd now supply combined units for nitrogen and air, operating from a single compressor, and these are particularly attractive for users of GLC. Standard units are available that produce:

Nitrogen (l/min)	Air (l/min)
0.75	1
2.0	4.0
4.0	8.0

Other combinations can be supplied to specification.

Laboratory piping and fittings

Whichever supply system is adopted there will be the need for a comprehensive internal network of piping to supply the instruments. The network should be carefully planned to minimize the run of piping and should be neat and tidy. The commonest material for this purpose is copper and high purity material in a range of internal diameters is available. It is suggested that the main network be in one-quarter inch (6.4 mm) tubing, with final connection from junction points being in one-eigth inch (3.2 mm) tubing. Swagelock fittings are recommended for joints and distribution points, with all threaded joints being bound with Teflon tape to reduce the likelihood of leaks. On completion of the plumbing, including connection to instruments, all joints should be

tested under pressure for leaks using either a dilute soap solution or commercially available leak-testing solutions. Care should be taken to prevent these solutions entering the system and they should be carefully wiped off after use.

The costs of tubing and fittings is difficult to estimate as it will depend upon the layout adopted which in turn may be governed by space considerations.

The following is a check list of fittings and supplies likely to be needed.

- Copper tubing – ¼ inch (6.4 mm)
  – ⅛ inch (3.2 mm)
- Assorted fittings – T pieces
  4-way crosses
  unions
  ¼-⅛ inch (6.4-3.2 mm) reducers
  ¼ and ⅛ inch nuts (6.4,3.2 mm)
  ¼ and ⅛ inch (6.4,3.2 mm) ferrules

Gas purifying systems

The necessity for gas purifying systems was mentioned earlier in the text (*see* p.9) and the removal of oil and moisture will be essential when using certain air compressors. Irrespective of the supply system each supply line must contain in-line filters as follows:

- nitrogen – moisture and oxygen filters;
- helium – moisture filter;
- hydrogen – moisture filter;
- oxygen – moisture filters;
- air – moisture filter.

The use of self-indicating filters, rechargeable preferably to reduce costs, is recommended.

Filters can be either wall or bench mounted. Whichever system is chosen, it is essential that the filters be both visible to detect indicator changes immediately and accessible to allow for ease of changeover. The appropriate mounting brackets must be ordered with each filter holder.

Tools

A selection of tools including spanners, screwdrivers and allen keys will be required. In addition there will be the need for a hacksaw with spare blades, a tubing cutter, a selection of small files for smoothing the ends of cut tubing and a good vice, preferably for permanent bench mounting.

Recurrent costs

Recurrent costs will depend upon the method of gas supply. Cylinders will need to be recharged regularly and these incur either rental charges or purchase charges (which can be expensive) together with the cost of the gases and any freight or delivery charges. Generators may require maintenance and there will be the need to renew any associated filters. The hydrogen generator should have provision for the replacement of the palladium catalytic cell after a period (say 3-6 years) depending upon its rate of use and proper handling.

The on-line filters need to be changed or recharged at a rate defined by the indicator; moisture filters may need to be changed more frequently in a humid climate.

Provision should also be made for the purchase of a small amount of tubing and some fittings to take into account any moving of equipment or replumbing.

Vacuum supplies

The provision of a vacuum supply is also recommended. Some laboratories use a central pump to generate vacuum with a number of outlets fed by a network of piping. This type of system does have some advantages but is not recommended for use in developing countries and 'local' generation within

the laboratory is to be preferred. Water-generated vacuum is favoured on safety grounds if the water pressure and supply are adequate. In cases where vacuum can only be provided through the use of electrically operated pumps, particular care must be taken to prevent liquids or vapours, especially those of an inflammable nature, passing through the pump. The use of well-maintained cold traps to condense and retain volatile solvent vapours is essential and it is also beneficial to ensure that the exhaust of the pump is vented externally from the laboratory.

LABORATORY EQUIPMENT, GLASSWARE AND REAGENTS

General equipment requirements for residue laboratories and formulation laboratories are discussed separately.

Residue laboratory

Equipment needs for a residue laboratory can be broken down by activity as described below.

Sample storage

There must be sufficient storage capacity for the anticipated sampling programme and this must reflect storage of both the fresh sample as submitted and of extracts at all stages of clean-up. Most fresh samples will require deep-freeze storage conditions (−20°C to −30°C) to minimize pesticide loss by degradation before extraction. Certain commodities, for example, fruits and vegetables, will deteriorate if stored in this way and be rendered unsuitable for analysis. Storage at 5-10°C will be required in such cases.

Sample extracts must be stored in a refrigerator whilst awaiting clean-up and analysis; similarly, dilute pesticide reference solutions must be kept in a refrigerator when not in use. Samples and standards must not be kept in the same refrigerator and therefore separate provision must be made for each.

Sample extraction

The process of sample extraction also includes basic sample preparation including mixing, sub-sampling and chopping/grinding to a condition suitable for extraction.

Equipment needs for sample mixing are minimal although if large samples of, for example, cereals, are to be mixed before sub-sampling then some large-scale tumbling facility is required. Depending on scale this can be accomplished by mixing streams of grain being poured from large beakers and repeating the process until adequate mixing has been effected.

Sample chopping and/or grinding can require a range of apparatus depending on the nature of the samples and the scale of operation. The first requirement is for a range of good-quality knives and chopping boards; these are commonly used for chopping tissue samples and fruit and vegetables. It is normally necessary to complement chopping by the use of a high-speed blender and this is also essential for the fine grinding of dry materials such as cereals and oilseeds. There is a wide range of blenders/macerators available, from domestic coffee grinders to heavy-duty commercial systems. Light, cheap coffee grinders are good to use with cereals and oilseeds but the capacity is limited and the blades are easily damaged, necessitating a good supply of spares. Similarly, domestic liquidizers can be used for soft tissues or fruit. These are normally made of plastic and care must be taken not to allow organic solvents to come into contact with them. Specially designed laboratory blenders, with glass or stainless steel maceration containers, offer a wide range of sizes and applications. They can generally be used for dry or wet grinding and for sample extraction by maceration with solvent, although care must be

taken to ensure that only spark-proof grinders are used for maceration with organic solvent. These systems are expensive but their lifespan is greater. However, some problems with excessive wear have been noted on the drive shafts of some models and a good supply of spare parts is necessary to keep them in service. A further system finding favour with many laboratories is the use of heavy-duty food processors as used by commercial kitchens. These are available with a wide range of attachments for different grinding and cutting operations and have the advantage of large capacity. They also effectively mix as they divide the sample, aiding the removal of sub-samples for extraction. Their use depends on the nature of the samples but for laboratories dealing with large tissue or fruit and vegetable samples they are *strongly* recommended; they have no advantage for laboratories dealing solely, for example, with cereals.

Extraction systems are varied but to cover the full range and omitting glassware requirements (considered later in Appendices B1, B2 and B3, pp. 35, 46 and 61) and systems for maceration with solvent as considered above, equipment needs can be summarized as:

- wrist-action shaker, 6- or 8-place;
- ultrasonic bath;
- Soxhlet extraction electrothermal mantle, 6-place and 250-500 ml flask size capacity.

Required sample throughput will determine the number of each item required.

Sample processing (before analysis)

Sample processing, extraction and clean-up before analysis have high requirements for glassware and equipment. Glassware needs are considered separately (*see* Appendices B1, B2 and B3, pp. 35, 46 and 61) and equipment requirements are discussed below under their application headings.

Weighing facilities

There is a requirement to weigh, accurately, both small and large samples. Earlier the point was made that pure reference standards should be kept outside the residue suite and similarly the weighing-out of quantities of these materials for the preparation of stock and working solutions should also be carried out outside the residue laboratory using separate weighing facilities. There is, however, the need to weigh out small quantities of reagents, and for this a three-place analytical balance will be adequate. For larger quantities of reagents and for weighing out samples or sample aliquots, a top-pan balance will be required. A wide range of such balances is available and a dual analytical range system is most appropriate. These will commonly weigh 0-2000 or 3000 grammes to one decimal place on one scale, and 0-1000 grammes to two or three decimal places.

The balances must be mounted on a vibration-free table and shielded from draughts, in a room separate from the laboratory. The top-pan balance(s) is better placed in the laboratory in a partitioned-off area or quiet corner.

Laboratory ovens

There is the conventional requirement for a laboratory oven with an upper temperature limit of about 200-250°C. In addition, access to a muffle furnace capable of temperatures up to about 750°C is important for the purification of certain reagents where contamination has been observed and for the activation of column adsorbents.

Solvent distillation and sample evaporation

Solvent distillation and sample evaporation require the use of steam baths, electric heating mantles or rotary vacuum evaporation. In practice a combination of these is often in use. Solvent distillation should be employed as a

means of purifying solvents such as acetone and petroleum ether from local sources which often contain impurities that interfere with residue analysis. This latter operation will be on a larger scale and will need to cope with a wide range of boiling points. An electric heating mantle is normally employed for this operation of a size compatible with the distillation flask (commonly 2-5 litres for laboratory operations).

Sample evaporation carried out using a rotary vacuum evaporator is the most popular technique nowadays. Care must be taken with this method to prevent the loss of particularly volatile compounds.

A hot plate (electric) is also advantageous for reagent preparation or for reactions where temperature control is not critical.

Miscellaneous

Access to other 'common' items of laboratory equipment such as electric stirrers, magnetic stirrers, laboratory centrifuges, etc. is also required.

Equipment specifications are given in Appendix B2.

Analysis of samples

The equipment required for qualitative and quantitative analysis will often constitute the major costs in the establishment of a residue laboratory. The analytical techniques can be summarized under the headings of:

- gas-liquid chromatography (GLC)
- high-performance liquid chromatography (HPLC)
- spectroscopy
- thin-layer chromatography (TLC).

Each of these techniques has a particular application but its use can often be dependent upon local considerations and limitations on the equipment available or serviceable. An overview of these techniques may be of some help before the requirements are discussed in detail. **Gas-liquid chromatography** is the most commonly used analytical tool in residue analysis because of its ability to separate complex mixtures, and because of the range of sensitive detectors available for the measurement of extremely low quantities of residues. **High-performance liquid chromatography** is also used, particularly for the analysis of residues of fungicides and of the synthetic pyrethroids, but its use has been restricted by the limitations on the detection systems available and, in general, its lower sensitivity when compared to GLC. Another difficulty to date has been with the introduction of a suitable interface for HPLC to be coupled to a mass spectrometer as commonly happens with GLC. **Spectroscopic** techniques are generally less commonly used, although they still find favour in the analysis of certain carbamate and dithiocarbamate compounds.

Thin-layer chromatography is still used extensively in residue analysis for the estimation of residues, but it is also used as a clean-up aid using modified TLC plates. Opinions vary on the technique and on the extent to which residues can be defined. Modern refinements such as the use of a densitometer to aid the 'reading' of a plate have made the technique more consistent but these have moved it away from its position as a low-cost semi-quantitative method. There is no doubt that in the hands of a skilled operator low-cost reliable results can be obtained. Both spectroscopic and TLC techniques are used extensively in developing countries.

Formulation laboratory

Sample storage

Pesticide formulations are normally stored at room temperature. In some climates it may be necessary to lower the ambient temperature with the aid of air conditioning but there will be a requirement to filter the air leaving an enclosed store. If the storage time is relatively short and the quantities of

material are fairly small and well packaged, then laboratory shelving may suffice.

If an external store is required for the storage of larger quantities of material, a running water supply should be provided together with a floor drain in case of spillage or accidents contaminating a worker.

Laboratory equipment and glassware

The techniques employed in the formulation laboratory require recourse to a mix of items of 'general' laboratory equipment and glassware and also to more specialized items for specific tests. This can be seen from the detailed list provided at Appendix A and will not be repeated here.

Reagents and consumables

The specification and nature of the laboratory and analytical reagents required for formulation and residue analysis and the quantities of each are difficult to define as these will vary with the nature of the analyses to be conducted. However there are a number of basic requirements and these are listed in the model laboratory specifications detailed in Appendices A, B1, B2 and B3. An approximation of the rate of usage is also given. Provision must be made for the purchase of additional reagents required for particular analyses after the definition of requirements.

Materials required for GLC, HPLC and TLC are listed with the appropriate technique in the model laboratory specifications.

Storage of pesticide reference materials (standards)

The proper storage of reference materials is crucial if analytical results are to be meaningful. The store should be outside the residue laboratory and could be associated with the formulation laboratory if one exists. Most pesticide standards are best kept in a refrigerator or deep freeze although there are certain exceptions; advice from the supplier is useful in this context.

Reference standards stored in a freezer will have a shelf life of several years, although note should be taken of the 'Use by . . .' information generally supplied by the manufacturer/supplier.

It is recommended that the reference materials be stored in a self de-frosting upright freezer of stainless steel construction, for ease of cleaning, and of a capacity of not less than 200 litres. The freezer should be fitted with stainless steel shelves forming discrete compartments to facilitate segregation of materials by class of compound.

A disadvantage is that freezers can be a source of moisture and whilst it is recommended that materials be kept in sealed vials this is not always possible. A useful solution is to keep the individual containers inside a larger screw-capped container to which a desiccant has been added; self-indicating silica gel is useful for this purpose. This material can be regularly changed and regenerated. The number of containers (polythene is preferred to glass) and the quantity of silica gel required depends on the quantities of reference material to be stored. Freezers function more efficiently when full and it is probably best initially to fill or nearly fill the freezer with containers holding single compounds, and to double up as the library of standards expands. With a good coding or labelling system location of individual materials can be quite rapid.

Recurrent costs

Recurrent costs for storage of reference materials are slight and unless there is a difficulty with the operation of the freezer itself, are confined to the replacement of silica gel and any damaged storage containers (assuming

sufficient were purchased initially). Silica gel requirements annually will be about 1 kg.

ANALYTICAL REQUIREMENTS

Gas-liquid chromatography

The provision for gas-liquid chromatography is essential in both the formulation and residue laboratories. The number of instruments and their detection systems must reflect the nature of the materials to be analysed and this can mean the purchase of several units.

For residue analysis, instruments may be fitted with an electron capture detector (ECD), a flame photometric detector (FPD), a thermionic detector (TID), also known as an alkali flame ionization detector (AFID), or a nitrogen phosphorus detector (NPD). These detection systems may be mounted singly or in multiples depending on the instrument design. The ECD is an ionization detector responding to the presence of materials with a high electron affinity and is of particular value in the analysis of chlorinated insecticides, natural and synthetic pyrethroids and some herbicides and carbamates following the formation of suitable derivatives. The FPD selectively allows for the analysis of compounds containing phosphorus or sulphur and the AFID/NPD is used for the analysis of compounds containing nitrogen or phosphorus. The NPD is extremely sensitive and in most cases will give a higher sensitivity to phosphorus-containing compounds than the FPD. However, some systems can be a little unstable and the FPD is to be preferred for routine use because its operation tends to be simpler and down-time is reduced.

The use of the selective detectors can assist with pesticide identification or confirmation of identity. Parallel operation of detectors using a column eluent splitter can provide for simultaneous examination from a single sample injection. This is one benefit of the use of a multiple detector instrument. One slight disadvantage is that if the detectors are being operated independently, they have to either operate simultaneously under common column oven conditions or be used one at a time as need be. Many laboratories operate on the basis of using 'dedicated' instruments, that is, with just one detection system, and although there is a greater cost implication with this method of operation, it does mean that both instruments can be operated simultaneously with different operating parameters. If funds are limited, there is an advantage in purchasing a multiple detector unit.

If GLC facilities are used in the formulation laboratory there will also be the need to equip at least one of the gas chromatographs with flame ionization detection to facilitate the analysis of concentrates without having to undertake the large dilutions necessary for the other types of detectors.

Column systems can also vary in design and application with the use of capillary columns of lengths up to 50 metres becoming much more popular following the development of the flexible fused silica columns. Conventional packed columns are still widely used and account for the greater proportion of published retention time data.

An important development has been the introduction of the ion trap detector (ITD), a simplified version of the mass spectrometer, and residue analysis with built-in confirmation of identity is becoming more common. Such a system is much more expensive than those considered earlier but eliminates the need for a range of detectors. The system does require the attention of skilled operators and should not be contemplated for use anywhere other than in laboratories where the staff are experienced. It should best be considered as complementary to conventional systems.

Gas supplies for gas-liquid chromatography (GLC)

Laboratory gas supplies including those for use with the analytical instruments were discussed, and detailed specifications for the purity of supply were given in *Gas supplies,* (pp.9–13), as were details of recommended gas generation systems. The advantages of these systems must be stressed; they are of particular benefit where cylinder supplies are expensive or difficult to obtain.

High-performance liquid chromatography

High-performance liquid chromatography has been considered as having limited application in a residue laboratory although there are specific uses for certain types of compounds, for example, the synthetic pyrethroids and many fungicides. Since these materials can be analysed by other techniques, it is considered that apart from larger residue laboratories with wide-ranging analytical programmes, that the purchase of an HPLC system is not a priority.

If a system is purchased, then it is worth while obtaining one that is comprehensive, allowing for gradient elution and fitted with a diode array detector and the necessary computer back-up. These detection systems allow for simultaneous multi-channel analysis which can be used for confirmation of identity or for assessing the purity of a sample component. Problems with reduced sensitivity have now been resolved and such systems now give sensitivity comparable to that of a conventional UV/visible range HPLC spectrophotometer.

HPLC is becoming increasingly used for active-ingredient determination in formulation analysis, where reduced sensitivity is not a disadvantage because of the nature of the sample. A UV/visible range detector should be adequate although the diode array system would be useful for identification purposes in the case of an unknown or wrongly labelled material.

Data handling

Data handling has traditionally been through the use of a potentiometric strip chart recorder and this system is still a prime recommendation. It has the advantage of cheapness, but requires manual interpretation. This may not be a disadvantage and may even benefit a newly established laboratory where the staff are still receiving training.

A range of integrators of varying degrees of sophistication are also available but cannot take the place of experienced laboratory staff. These integrators can take the form of the most basic unit, merely measuring the areas of peaks on a chromatographic trace (as programmed by the operator) and perhaps including a few calculation functions, through to systems that will allow multiple peak measurement and subsequent storage and re-analysis of data.

A further and more exciting development has been the use of computer systems for the on-line measurement and manipulation of data. Such systems are a development from integrators but allow a greater flexibility of use. The computer can be used for other applications, such as laboratory data storage, with data generated from an analytical system being stored in a suitable interface until the computer is free for data discharge. Other uses will of course be limited by the degree of data input.

The degree of sophistication to which the laboratory will be equipped will have to be judged taking into account its role, workload and the availability of trained staff and back-up services.

Spectrophotometric requirements

A UV/visible-region spectrophotometer is a useful piece of back-up equipment for a residue laboratory and for certain compounds can substitute for GLC or HPLC. Colorimetric procedures are available for organophosphorus and carbamate compounds, dithiocarbamates and for certain herbicides and fungi-

cides. These procedures are used extensively in developing countries where other facilities are limited, but they can be prone to interference and in some cases the sensitivity can be poor. A laboratory dealing with a range of compounds may find it useful to have access to a spectrophotometer, but for many laboratories it will find little use and funds can be better spent on other equipment.

Many current methods for the determination of active-ingredient content in pesticide formulations employ spectrophotometric methods although these are gradually being replaced by GLC and HPLC procedures. There is therefore still a certain requirement for a spectrophotometer for this purpose.

Thin-layer chromatography

Thin-layer chromatography for pesticide residue analysis is a valuable technique which finds favour with many laboratories. It can be made as sophisticated or as simple as the operator wishes and in its basic form is more than adequate as a screening technique. Thin-layer plates of high quality can be purchased ready made and are available with a wide range of adsorbents and surface treatments. They can be expensive however and may be a budgetary constraint for developing countries. Plates can also, with practice, be prepared in the laboratory. This latter option is cheaper once the materials and spreading equipment have been purchased although the prepared plates will be found to vary in quality until suitable experience has been gained by the staff. This latter option is nonetheless recommended for laboratories in developing countries. Used as a screening procedure on partly cleaned-up extracts, TLC can save time, and more importantly, reagents, in certain circumstances.

ANALYTICAL METHODOLOGY

Analytical methodology will reflect the nature of the samples under analysis and the range of pesticides involved. It will also need to take into account the reasons why the analyses are being performed. For example, routine screening will often involve the use of well-evaluated, locally developed or simplified procedures, whereas samples analysed for legislative or other legal purposes, or those showing residues on or about national or international tolerance levels, may need to be analysed or re-analysed using established or referee procedures.

The initial choice of procedures is often governed by experience, although there are well-established patterns of approach. The choice of methodoloy is also affected by whether the analyst is looking for a known compound(s) or if the sample needs to be screened for unknown residues.

There is little merit in using a detailed referee method for the analysis of a group of samples from, for example, a field study where a number of compounds are being screened for biological activity and only an indication of the likely pesticide residue levels is required. A simplified method, with labour and material savings, can be used for this or similar situations. The key is to be able to identify where such procedures can be used and where they cannot. Routine screening of residues is commonly undertaken by laboratories using simplified procedures with 'borderline' samples being reserved for more rigorous analysis. This is quite acceptable, but **it must not become a licence for corner cutting**. The essence is to use the procedure, fully evaluated and shown to be both efficient and reproducible, most suited to the analysis in hand.

An approach used by some laboratories is to use only a partial clean-up before GLC analysis on the basis that a number of analyses can be completed before the GLC column becomes contaminated and unusable. The cost of repacking or replacing the column is less than the cost in labour and materials and the analysis is speeded up. This practice does work well in some cases but is not to be recommended where the operator is relatively inexperienced;

unreliable results can be obtained through the co-injection of quantities of oil or fat and the inexperienced operator may miss the early tell-tale signs. Instrument contamination problems can also occur, particularly with GLC.

Some recommended references for analytical methodology, sampling procedures and good analytical practice are given in Appendix C.

MANPOWER REQUIREMENTS AND THE TRAINING OF LABORATORY STAFF

Manpower levels will reflect the scope of the work programme and the grading structure of the organization. The analysts should be graduates, assisted by junior scientific staff and other support personnel. In practice, a ratio of two or three support staff to each analyst provides a reasonable working structure. The graduates should have a degree in chemistry supplemented by a qualification in analytical techniques, or with suitable analytical experience, preferably in pesticide chemistry and instrumental techniques.

It was stressed earlier that pesticide residue analysis is particularly demanding and that there was a requirement for thorough training of the personnel involved. The training must be of a practical nature, supplemented by lecture and discussion, and conducted under the supervision of a suitably qualified tutor. The minimum training period to provide for a basic understanding of the subject must be at least three months. Depending upon the nature of the duties of the individual, this may need to be supplemented by a further more advanced period of training. More often than not the training will be conducted in an external laboratory and there will then be the need for a period of familiarization on return and for the practice of routines. Time and materials must be reserved for this. The general practice is for the senior members of the laboratory to receive external training and then, on return, train the more junior members of staff and the support staff.

A problem that is commonly encountered however, is for the trained analyst to be transferred or promoted on return and for the expertise to be lost. There needs to be a degree of commitment from the analyst if the benefits of training are not to be wasted. Similarly, there is a commitment for senior management to ensure that the analyst does not have limited career prospects and that his career progression and his personal motivation are not depressed. The management structure needs to take all these factors into account to ensure that talent is not wasted and that there is maximum benefit to the laboratory.

A training period of about two months should be adequate for familiarization with the techniques involved in the physical examination of formulations. Although complicated, they reflect a number of repetitive techniques common for most pesticides and the period of training is correspondingly shorter. The analysis of active-ingredient content requires a range of analytical skills and experience in GLC, HPLC and spectroscopy. If the chemist is not trained in analytical techniques, then a period of a further two months' training will be required.

Appendices

APPENDIX A
PESTICIDE FORMULATION LABORATORY MODEL SPECIFICATION

Laboratory capacity

Laboratory capacity for a pesticide formulation laboratory cannot be defined using the same criteria as for the pesticide residue laboratory (*see* Appendices B1, B2 and B3). Sample throughput will be relatively low unless the laboratory is part of a manufacturer's quality control line, or the organization is concerned with the quality control of marketed or stored materials. Pesticide formulations can be subjected to a wide range of tests for physical properties in addition to the determination of active ingredient. There is a basic need for a range of apparatus which reflects more the nature of the analysis rather than the overall sample throughput.

Staffing requirements

There is a requirement for one chemist and one technician. If the workload is particularly high then this requirement may need to be increased in the light of experience. Much of the work can be undertaken at the technician level but there is a requirement for adequate training and supervision.

Laboratory accommodation

Space requirements

A minimum of 90 m^2 of floor area with 28 m^2 of available bench surface area is required. In the instrument room the benching should be movable to facilitate access to instruments and not fixed. Laboratory design should optimize the use of available space and should have due regard to the need for water, electricity and gases for both the analytical equipment and for bench supplies. A suggested design has been given in Figure 1. The accommodation should be subdivided as suggested below (minimum areas are given):

	Floor area, m^2	Bench surface area, m^2
Main laboratory	37	13
Glassware washing room	15	6
Instrument room	15	6
Balance room	8	3
Equipment/glassware store room	15	–

The balance room can be partitioned off from the main laboratory if so required, although the balances must be mounted on a separate vibration-free table rather than on a continuous run of standard benching.

The whole laboratory area should be air-conditioned.

The equipment store should have full shelving on two walls and cupboard and drawer enclosed storage facilities.

A lockable, well-ventilated sample reception/store room of approximately 20 m^2, ideally sited outside the laboratory block, will also be required. This room should have full shelving on two walls, cupboard storage and adequate floor-standing area for large containers. Laboratory benching will not be required.

Service requirements and fittings

Water

There is a requirement for good supplies of running water with multiple laboratory outlets – a minimum of 12 is suggested for the main laboratory, with two outlets associated with each of the sinks detailed below and with a further six spaced out along the benching, mounted on a service spine, and with attendant tun dishes.

In the main laboratory there should be one large bench-mounted sink (60 x 45 x 20 cm) and two smaller sinks (40 x 30 x 20 cm or similar), one in the fume cupboard and the other bench mounted. The washing-up room should contain two large, (60 x 45 x 20 cm) sinks each with a draining board and with ample wall-mounted drying racks for glassware. In addition a heated drying cupboard (150 litres capacity or similar) will be required.

Some laboratories use scientific glassware-washing machines for the bulk of their washing requirements; such provision, however, does not obviate the need for a washing-up sink for larger items of glassware, nor for the drying facilities detailed above.

Both hot and cold water supplies are required although the hot water can be confined to one outlet in the large sink in the main laboratory and to one each in the sinks in the washing-up room.

Electricity

Power will be required for a range of operations, some intermittent in function, but others, e.g. freezers/refrigerators and certain items of analytical equipment, will need a constant supply. It is suggested that the minimum number of power outlets required is:

Main laboratory	15
Instrument room	10
Balance room	3

The instrument room will require a 'clean' supply, i.e. free from interference, and should also have a voltage stabilizer if power fluctuations are likely.

Gas supplies

Supplies of nitrogen, hydrogen and compressed air will be required for the operation of the gas–liquid chromatograph in the instrument room, with additional outlets (1) for nitrogen and for compressed air (2) in the main laboratory. Consideration will need to be given to the plumbing for these supplies and as to whether most of the requirement should be met by the use of generators or from cylinder supplies (*see Gas supplies*, p.9). It should be noted that bench supply is practicable from a generated source, but the pressure drop can affect the gas–liquid chromatographs supplied from this source. The use of a line tank with suitable pressure controls can virtually eliminate this effect if attention is given in the design specification to overall demand, GLC pressure requirements, etc.

Fume cupboard

Two fume cupboards (1.5 x 0.75 m) will be required. They should each have outlets for cold running water (2) and compressed air (1). A small sink (approximately 20 x 50 x 15 cm) is a minimum requirement, although preferably the fume cupboard should be designed so that the sink occupies the full

available working area and is covered with removable acid/solvent resistant inserts enabling the fume cupboard to be used 'wet' or 'dry' or as a combination as the need arises.

Office accommodation

Office area requirements are not specified although these should not be overlooked. Because of the nature of the work, only a minimal amount of written work should be undertaken in the laboratory and office accommodation is essential for the preparation of analytical reports, maintenance of records, etc. Similarly, no provision is made for a library or common room although these may be considered desirable.

Equipment

Gas liquid chromatography

Two GLCs will be required, consisting of:

(i) twin-column oven system with linear, multi-ramp temperature programming and with dual-heated packed column injectors. To be fitted with twin, heated flame ionization detectors and be complete with all associated pneumatic and electrical control systems;
(ii) as for (i) but with dual capillary column injectors.

Instrument voltage requirements will need to be specified when ordering the instruments.

Spares, accessories and consumables

(i) A service kit for each instrument as recommended by the manufacturer.
(ii) Injection septa 200
(iii) Injection liners (if required) 6 for each of the injectors
(iv) Glass GLC columns, empty:

0.5 m	3
1.0 m	5
2.0 m	3

(v) Column packing materials, commercially prepared, 100 g quantities of each:

10% DC 200 *or* OV 101 on Chromosorb HP (80-100 mesh)
1:1 mixture of 10% DC 200 and 15% QF_1 each coated on Chromosorb W HP (80-100 mesh)
2% DEGS (stabilized) on Chromosorb W HP (80-100 mesh)
3% OV 225 on Chromosorb W HP (80-100 mesh)
Silianized glass wool, 100 g

Alternatively the individual stationary phases and support materials can be purchased within the cost of the commercially prepared materials and suitable mixtures prepared in the laboratory. This has the advantage, as long as the mixtures can be prepared properly, that a wider range of mixtures can be prepared and in greater quantities for the same initial outlay. Such preparation should only be undertaken by experienced personnel, and if doubts exist then the commercially prepared mixtures should be obtained.

(vi) Capillary columns for GLC:

2	25 m	0.2 mm i.d. fused silica DB 1
2	25 m	0.2 mm i.d. fused silica OV 225
2	25 m	0.2 mm i.d. fused silica BP 1

(vii) Syringes, hypodermic, needle length will be dependent upon the make and model of the GLC.

10 µl	5
2 µl	3
1 µl	2

(viii) A selection of tools including screwdrivers, spanners and allen keys will be required. Note that a limited number of tools are provided with the GLC; check to eliminate unnecessary duplication.

Recurrent costs

Recurrent costs will be the continual replacement of materials used from the initial stock. An approximation for the early years, unless the wastage rate or instrument usage is particularly high, would be a cost of about one-fifth of the cost of the initial stock of consumables and accessories.

As the throughput of the laboratory increases with experience then greater recurrent charges can be expected. This is difficult to estimate, but the costs could be about 25% more than in the earlier years; if trouble-free operation is maintained then the costs will be substantially lower.

Provision should also be made if possible for regular servicing of the equipment, perhaps by means of a service contract. This can reduce in the long term costly 'call-out' charges.

Gas supplies

Gas supply and purification systems are discussed in *Gas supplies*, p.9. Those details are not repeated in this model specification.

If a generated supply is used, it is recommended as a precaution against instrument 'down-time' should a fault develop, that a reserve supply of one cylinder of each gas be kept.

Gas generators

(i) Combined nitrogen and compressed air generator, output capacity 0.75 l/min (or similar) of nitrogen and 1 l/min of compressed air.
(ii) Hydrogen, output 300 ml/min or similar.

Miscellaneous

(i) Cylinder head gauges, one per gas for back-up cylinders and for distribution of the laboratory supply where feed from generator system is considered impractical.
(ii) Moisture filters for each gas and an additional oxygen filter for the nitrogen (excluding the laboratory supply). Two spare moisture filters and one spare oxygen filter. Mounting brackets and fittings.
(iii) Copper tubing, ⅛ inch (3.2 mm), 10 m.
¼ inch (6.4 mm), 10 m.
(iv) Assorted fittings, ⅛ and ¼ inch (3.2 and 6.4 mm) adequate for the proposed gas distribution network. The following will give a guide to quantities required:

T-pieces, ¼ inch (6.4 mm)	5
T-pieces, ⅛ inch (3.2 mm)	5
4-way crosses, ¼ inch (6.4 mm)	3
4-way crosses, ⅛ inch (3.2 mm)	3
Unions, ¼ inch (6.4 mm)	5
Unions, ⅛ inch (3.2 mm)	5
¼-⅛ inch reducers (6.4-3.2 mm)	80
¼ inch (6.4 mm) nuts	20
⅛ inch (3.2 mm) nuts	20
¼ inch (6.4 mm) front and back ferrules	20
⅛ inch (3.2 mm) front and back ferrules	20

(v) Tools for cutting tubing, etc. as listed on p.13.

Recurrent costs

The generators may require maintenance and there will be the need to renew any associated filters. The hydrogen generator should have provision for the replacement of the palladium catalytic cell after a period depending upon its rate of use (3-6 years).

The on-line gas filters need to be changed or recharged at a rate defined by the indicator; moisture filters may need to be changed more frequently in a humid climate if used with generated air or nitrogen.

Provision should also be made for the purchase of a small amount of tubing and fittings to take into account any movement of equipment or replumbing.

High-performance liquid chromatography

An HPLC system incorporating:

programmed solvent gradient elution;
pumping system with programmable flow rate in the range of 0-10 ml/min and an operating pressure range of 0-420 bar;
sample loop injection valve;
UV/visible fixed wavelength detector with flow through cell.

Consumables and accessories

(i) An instrument service kit as recommended by the manufacturer.

(ii) Columns, cartridge system such as Chrompack Chromsep system or similar.

10 cm hardware kit	1
20 cm hardware kit	1
Spare column compressor	1
Spare column housing for 10 cm columns	1
Spare column housing for 20 cm columns	1
Spare column couplings	5
Teflon rings and screens	10
Valco nuts, 1/16 inch (1.6 mm)	10
Valco ferrules, 1/16 inch (1.6 mm)	20

6 each of the following ChromSep-type columns, 10 cm length:
Lichrosorb Si 60, 7 µm
Spherisorb ODS, C18, 5 µm
Lichrosorb RP, 7 µm

ChromSep guard columns for reversed phase columns	12
ChromSep guard columns for silica columns	12

(iii) Syringes,

25 µl	2
50 µl	2
200 µl	2
1000 µl	2
Gas-tight syringes, 50 ml	2

Needle length and point design will depend on the valve system fitted to the instrument.

(iv) Injection valve loops:

20 µl	2
50 µl	2
200 µl	1
500 µl	1
1000 µl	1

Recurrent costs

Recurrent costs will depend upon the rate of use and the wear and tear on system components. There will be a need to replace elements of the service

kit as they are used, together with syringes and columns. Used properly the columns will have a life-span of several hundred injections but poor technique can dramatically reduce this. The guard columns will have a relatively short life. An approximation for recurrent costs would be something of the order of 20-25% of the initial cost of the consumable materials for the first 2 or 3 years with a rise to 30-40% after that. As the system ages, provision must also be made for instrument maintenance charges.

Data handling

Data handling can be accomplished manually using a standard chart recorder or through an automatic or semi-automatic system. It is recommended that the outputs from both the GLC and HPLC systems should be linked into an integrator, preferably a model with the capability to print the full chromatogram. Theoretically there is a requirement for the measurement of five data channels, four from the GLC and one from the HPLC, although it is unlikely that all will be in use at any one time. Provision should be made for the higher number however and this will give a limited degree of back-up. In such a case there will be a requirement for one single and two double channel integrators.

A supply of chart paper, 10 rolls per integrator will also be required.

General laboratory equipment

Equipment	No./quantity required
(i) Analytical balance, single pan, electronic, weighing range 0-200 g, readable to four decimal places (or similar)	
(ii) Top-loading balance, dual range, weighing range a) 0-300 g readable to two decimal places and weighing range b) 0-3000 g readable to one decimal place (or similar)	
(iii) Laboratory oven, 0°-250°C, fan convection, capacity 150 litres or similar	
(iv) Muffle furnace, 1100°C, capacity 7.5-10 litres	
(v) Laboratory hotplate, rectangular, approximate size 23 x 30 cm	
(vi) Water deionizer, wall mounted, maximum flow rate 10 litres/hour	
(vii) Refrigerator/freezers	
(a) Refrigerator, spark proof, approximately 200 litres capacity, for storage of reference standard solutions	
(b) Freezer, upright, −10° to −20 °C, approximately 250 litres capacity, for storage of pure analytical reference materials	
(viii) UV/visible spectrophotometer, double beam	
1 cm and 4 cm matched absorption cells, set of four of each	
(ix) Infrared recording spectrophotometer, double beam, grating monochromator, transmission repeatability 0.5% or better	
Instrument spares kit as recommended by the manufacturer	
Cells with sodium chloride windows, 0.2 mm and 0.4 mm path length	1 of each
Chart paper	5 packs
Pens for recorder	20
(x) pH meter with combination electrode, electrode stand and holder	

	No./quantity required
(xi) Flask shaker, six-or-eight-place, with 60-minute timer	
(xii) Laboratory interval timer, 60 minute	2
(xiii) Timer, stop-watch, triple range to 30 minutes	
(xiv) Rotary vacuum evaporator with temperature-controlled water bath: Buchi Rotavapor RE 121 or similar, with:	
vacuum controller	2
spare receiving flasks	2
vapour ducts, B 24/29 cone	3
spare gasket seals	5
clips for securing receiver and distillation flasks	5
mesh-type material to cover condenser and receiving flask to reduce hazard from glass implosion under vacuum	
(xv) Vacuum pump, water, solvent and corrosion resistant (e.g. Genevac type)	
Option If water-generated vacum is not practicable, then a second vacuum pump will be required	
(xvi) Cooled water bath/circulator	2
(xvii) Laboratory bench-top centrifuge, capacity for 4 x 200 or 250 ml containers. To be supplied with rotor assembly and sealed cups and adaptor for use with tubes of 10 and 50 ml capacity. 6 of the 200/250 ml size containers and 12 of each of the 10 and 25 ml tubes to be included	

Recurrent costs

The water deionizer will require the purchase of fresh cartridges at intervals and provision should be made for this. None of the other items detailed above has a similar requirement, although funds should be available to meet the cost of replacement parts and servicing should faults develop. Longer-term provision must also be made for the replacement of equipment with age. It is difficult to forecast the working life of individual items, although for most a life of 10-15 years can be expected.

Specific equipment for the testing of pesticide formulations

(i) Thermostatically controlled constant-temperature water bath with vibration-free agitator or circulation unit
(ii) Flash-point apparatus
(iii) Sieves, mesh sizes: 75, 150, 250, 355, 420, 500, 710 and 850 µ., 8 inch (200 mm) diameter
(iv) Sieve test shaker, 8 inch (200 mm) platform
(v) Tap density apparatus

Glassware and miscellaneous items

A General laboratory glassware	No./quantity required
(i) Volumetric flasks, Grade B	
10 ml	50
20 ml	25
25 ml	25
50 ml	25
100 ml	40

	No./quantity required
250 ml	15
500 ml	10
1000 ml	5
(ii) Round-bottomed flasks, Quickfit	
250 ml, B24 neck, short	15
500 ml, B24 neck, short	24
1000 ml, B24 neck, short	5
(iii) Conical flasks, Quickfit	
100 ml, B19 neck	10
150 ml, B24 neck	15
250 ml, B24 neck	25
500 ml, B24 neck	10
(iv) Measuring cylinders, lipped	
10 ml	10
25 ml	10
50 ml	15
100 ml	20
250 ml	15
500 ml	6
1000 ml	4
(v) Measuring cylinders, stoppered	
10 ml	10
25 ml	25
50 ml	25
100 ml	30
250 ml	10
1000 ml	4
(vi) Beakers, squat form, lipped	
50 ml	20
150 ml	20
250 ml	25
500 ml	10
1000 ml	6
(vii) Pipettes, Class B	
1 ml	10
2 ml	10
5 ml	15
10 ml	15
25 ml	5
50 ml	3
Graduated pipettes	
10 ml	10
Piston-type pipettes	
10 ml	10
Pasteur pipettes	
14.5 cm, boxes of 1000	1
(viii) Burettes, Class A	
25 ml, 0.05 ml divisions	3
50 ml, 0.1 ml divisions	3
(ix) Separating funnels, pear shape, glass stoppered	
100 ml	5
250 ml	10
500 ml	10
1000 ml	10

		No./quantity required
(x)	Glass stoppers	
	size C10 (10/13)	40
	C12 (12/14)	40
	C14 (14/15)	20
	B19 (19/17)	50
	B24 (24/20)	50
	B29 (29/32)	10
(xi)	Condensers	
	Allihn condenser	
	40/38 cone	8
	50/42/cone	8
(xii)	Soxhlet extractors	
	100 ml capacity, 40/38 socket	8
	200 ml capacity, 50/42 socket	8
(xiii)	Filtration equipment	
	Filter funnels, Pyrex – 55 mm diameter	4
	125 mm diameter	4
	200 mm diameter	6
	Buchner flasks, Quickfit	
	B24 socket 250 ml	6
	B24 socket 500 ml	6
	Funnels, Sintaglass, Buchner, sinter porosity 3, 24/29 cone	
	140 ml	3
	400 ml	3
	Crucibles with sintered glass disc, and ground glass cone 34/35, grade P16	5
	Expansion adapters, 34/35 socket	
	24/29 cone	5
(xiv)	Thermometers, total immersion type,	
	25°C: type IP 30C	3
	Thermometers, partial immersion type	
	−20.5°C to +20.5°C	2
	−5°C to +105°C	2
(xv)	Weighing bottles, glass	
	15 g	4
	30 g	4
	Weighing funnels, glass	
	5 ml	4
	15 ml	4
	(or similiar sizes)	
(xvi)	Glass jars, plastic screw cap	
	100ml	30
	200 ml	30
	500 ml	30
(xvii)	Glass rod,7 mm	3 m
(xviii)	Glass tubing, normal wall, borosilicate glass	
	7 mm	3 m
	9 mm	3 m
(xix)	Reagent bottles, glass stoppered	
	labelled for organic solvents	2 sets
	labelled for acids	2 sets

B Special glassware and accessories	No./quantity required
(i) Measuring cylinders, glass stoppered, 1 ml graduation. Distances between 0 ml and 250 ml graduations must be between 20 cm and 21.5 cm and between the 250 ml mark and the stopper, 4 cm to 6 cm	10
(ii) Glass measuring cylinders for tap density apparatus	4
Rubber base pad for the above	2
(iii) Glass beakers, 12–13 cm internal diameter (2.5–3 litres)	4
(iv) Piston or discs to fit loosely into above beakers	4
(v) Dean and Stark apparatus, BS 756:1952 or equivalent	3
(vi) Viscometer, BS 188: 1957 or equivalent	1

C Storage of pesticide stock solutions	No./quantity required
(i) Hypo vials, capacity 125 ml	72 (pack size)
(ii) Caps and liners	144 of each
(iii) Crimper and decapitator	1 of each

D Miscellaneous glassware	No./quantity required
(i) Desiccator, vacuum, 0–ring seal, 200 mm with disc	3
(ii) Crystallizing basins	
121×65 mm	4
150×75 mm	4
(iii) Basins, porcelain	
105 mm	3
148 mm	4
(iv) Watch glasses	
90 mm	5
150 mm	5
(v) Test tubes, glass stoppered, graduated 0-25 ml	10
(vi) Aspirator with stopcock, 10 litres	3
(vii) Weighing funnels	
85 mm	5
100 mm	5

E Miscellaneous items	No./quantity required
(i) Retort stand bases	
250×160 mm	10
Retort stand bases	
160×100 mm	10
Retort stand rods	
600×10 mm	10
Retort stand rods	
1000×12 mm	10
(ii) Boss heads	25
Boss heads, single jaw	10
Clamps	20
Burette clamps	2

		No./quantity required
(iii)	Rings, retort stand 70×125 mm	10
	Rings, retort stand 100×100 mm	10
	Rings, retort stand 130× 90 mm	5
(iv)	Scaffolding rods, aluminium alloy 1800 mm×13 mm	8
(v)	Spatulas, mixed sizes	15
	Palette knives, assorted sizes	7
	Scoops, 25 ml	5
	50 ml	5
	Forceps, blunt ended	4
	Forceps, pointed ends	4
	Scissors	4
(vi)	Tongs, beaker	2
	Tongs, crucible	2
	Tongs, furnace	1
(vii)	Tubing, vacuum	
	6 mm i.d.	15 m
	7 mm i.d.	15 m
	Tubing, PVC, colourless	
	6.5 mm i.d.	10 m
	8 mm i.d.	10 m
(viii)	Pipette filler bulbs	8
	Blow balls	8
	Rubber teats, assorted sizes	
	(1.5,2.0 and 2.5 ml	15 of each size
	Soft brushes, 1 and 2.5 cm	2 of each size
(ix)	Test tube rack	
	6–place	2
	10–place	2
(x)	Weighing boats, disposable	
	100 ml capacity	1000
	250 ml capacity	1000
(xi)	Aluminium foil	
	1 metre width	100 m
	0.5 m width	100 m
	Cotton wool, absorbent	2.5 kg
	Glass wool	1 kg
	Tissues, boxes of 200	20
(xii)	Gloves, heat resistant	1
	Gloves, acid resistant	1
	Gloves, disposable	5 boxes
	Safety spectacles	6
	Goggles	2
(xiii)	Burner, butane gas	1
	spare gas cartridges	2
(xiv)	Brushes, soft hair, 15 mm	3
	Brushes, beaker	assorted sizes
	Brushes, bottle	
	Brushes, burette	3 of each type
(xv)	Bowls, washing up, polythene	3
	Buckets, polythene	4

Option– If water-generated vacuum is selected there will be a requirement for three metal-bodied water jet filter pumps giving a vacuum of approximately 20 mbar. *Note* that this system will only operate if the water pressure is adequate.

		No./quantity required
(xvi)	Plastic containers with lids, capacity approximately 350 ml	25
(xvii)	Tubing clips,	
	10–14 mm	20
	13–17 mm	20
(xviii)	Cork rings,	
	75 cm o.d., 45 cm i.d.	10
	115 cm o.d., 85 cm i.d.	30
	150 cm o.d., 120 cm i.d.	10
(xix)	Safety screen, 3-panelled, flexible with feet	2
(xx)	Flammable solvent store, 710×915×483 mm	1
(xxi)	Acid store, 710×915×483 mm	1

Recurrent charges

The rate of consumption of glassware, even with due care, can be quite high and provision should be made for annual replacement at a level of about 15% of the cost of the initial stock. Similarly a number of the items detailed in section E are consumables and will need replacement at a rate dependent upon use and work patterns.

Reagents and consumables

Filter papers and extraction thimbles		No./quantity required
(i) Filter papers		
number 1*	7 cm	2 boxes
	15 cm	2 boxes
	24 cm	2 boxes
number 4*	7 cm	2 boxes
	15 cm	2 boxes
	24 cm	1 boxes
number 41*	7 cm	2 boxes
	15 cm	2 boxes
	24 cm	2 boxes
(ii) Extraction thimbles, cellulose, single thickness – Whatman or equivalent		
33×100 mm		8 boxes
41×123 mm		8 boxes

*Or equivalents

General reagents

Reagent requirements and the rate of replenishment are particularly difficult to predict as these will reflect the nature of the analyses or tests to be carried out on the pesticides in question. The reagents listed below form a good starting point and will cover a range of analyses. However the list is not meant to be comprehensive, nor in the circumstances could it be in the absence of full details of the work programme. It is important that the materials required for particular analyses are identified from standard procedures (for example from the *CIPAC Handbook*) and purchased accordingly.

	No./quantity required
Anti-bumping granules	250 g
Ammonium ferric sulphate, AR	250 g
Ammonium thiocyanate, AR	250 g
Buffer powders or tablets, pH 4	sufficient to make 5 litres of each
7	
9	

	No./quantity required
Calcium carbonate, AR	100 g
Di-n-butyl phthalate, AR	250 ml
Diaminoethanetetra-acetic acid, disodium salt, AR	50 g
Diethylamine, AR	250 ml
Florisil, 100–200 US mesh	3 kg
Iodine, AR	100 g
Magnesium chloride, AR	250 g
Magnesium oxide, AR	100 g
Potassium bromate, AR	100 g
Potassium bromide, AR	100 g
Potassium hydroxide, AR	2 kg
Potassium iodate, AR	100 g
Potassium iodide, AR	100 g
Potassium metal	100 g
Silica gel, self-indicating	1 kg
Silver nitrate, AR	25 g
Sodium hydroxide, AR	2 kg
Sodium hydrogen carbonate, AR	1 kg
Sodium sulphate, anhydrous, AR	2 kg
Sodium sulphate, AR	250 g
Sodium sulphite, AR	250 g
Sodium thiosulphate, AR	250 g
Acetic acid	2.5 l
Ammonia, s.g. 0.880	2.5 l
Hydrochloric acid	2.5 l
Nitric acid	2.5 l
Sulphuric acid	2.5 l

Indicators	No./quantity required
Bromocresol green	10 g
Bromothymol blue	10 g
Phenolphthalein	10 g
Methyl red	10 g
Solochrome black T	10 g
Solochrome dark blue	10 g
Thymol blue	10 g

Solvents	No./quantity required
Acetone	40 1
Acetonitrile	25 1
Chloroform	20 l
Diethyl ether	15 l
Ethanol	10 l
Ethyl acetate	15 l
Hexane	40 l
Methanol	25 l
4–methyl–2–pentanone	10 l
Solvent naphtha	2.5 l
2–Propanol	10 l
Toluene	10 l
Xylene	2.5 l

APPENDIX B1
PESTICIDE RESIDUE LABORATORY MODEL SPECIFICATION: LEVEL 1, 200/250 SAMPLES PER ANNUM

Laboratory capacity

A laboratory established with the equipment, facilities and staffing levels detailed below will have a capacity to analyse up to 200/250 samples per annum (approximately 300 analyses with replicates and recovery experiments). The size and facilities of the laboratory will restrict the range and complexity of the analyses that can be conducted, although some limited progression is possible with experience. A laboratory of this nature will commonly be associated with a small agricultural research unit where a limited range of pesticides is being studied.

Staffing requirements

The optimal staffing level for the laboratory would be one qualified analytical chemist supported by one or two technicians.

Laboratory accommodation

Space requirements

A minimum overall floor area of approximately 35 m^2 with 13 m^2 of available bench surface area is required. Separate rooms should be provided for the analytical operations and for the washing up of used glassware. The analytical laboratory should be sub-divided to separate the analytical equipment from the bench operations, with the analytical balance mounted on a draught-free, vibration-proof table or bench. If the GLC is installed in a separate instrument room then a floor area of about 6 m^2 with 2-3 m^2 of benching should be adequate. Air-conditioning is likely to be required.

Ample under-bench cupboard and drawer storage space must be provided together with shelving for reagents, etc. and storage for solvents and other reagents.

A fume cupboard will be required for certain laboratory operations.

Laboratory design should optimize the use of the available space and should have regard to the need for supplies of water, electricity and gases for the analytical instruments (*see* below).

A separate sample reception/storage room is difficult to justify at this level of operation although due consideration must be given to the type of sample expected and a separate area provided if cleanliness is likely to be a difficulty.

Office accommodation, not detailed here, will be required for the preparation of laboratory reports, record maintenance, etc. As a general principle, only a minimum of writing should be done in the laboratory.

Service requirements and fittings

Water

There is a requirement for good supplies of running water with perhaps 6 outlets in the analytical laboratory, two associated with bench mounted sinks (40×30×20 cm or similar) and with the other two spaced out along the benching, from a service spine, with associated tun dishes.

The washing-up room will require 4 outlets, two associated with the main sink (60×45×20 cm or similar) and the others wall mounted.

The washing-up room will require 4 outlets, two associated with the main sink (60×45×20 cm or similar) and the others wall mounted.

The washing-up sink will require supplies of both hot and cold water, with cold water only to the other outlets.

Provision should be made for the washing-up sink to have a draining board and ample wall-mounted drying racks for glassware. A heated drying cupboard for glassware (approximately 90 l capacity) should also be provided.

Electricity

A constant, stable power supply will be required. Some laboratory operations only require power intermittently but other items of equipment, e.g. the refrigerators/freezers and the GLC, will be switched on all the time. For a laboratory suite of this size, about 15 power outlets will be required. The GLC will require a 'clean' supply, i.e. free from interference, and should also have a voltage stabilizer if power fluctuations are likely.

Gas supplies

There are requirements for supplies of nitrogen, hydrogen and compressed air for the operation of the GLC, and one bench outlet each for nitrogen and compressed air.

Safety

Laboratory safety must be taken into full account when designing the laboratory and its internal layout; national safety regulations and, additionally, 'local' safety policies must be strictly followed. A minimum of safety equipment has been detailed on p.43, but this should be extended as necessary to comply with safety policy.

Laboratory overalls for members of staff are not included in the list.

Equipment

Gas-liquid chromatography

This level of operation suggests a requirement for just one GLC, but allows no margin for instrument breakdown, down time, etc. and there should be a minimum operational level of two GLCs. This is particularly important for laboratories in developing countries remote from service facilities and where enforced delays can cause particular difficulties. Additional establishment costs are necessarily incurred.

Instrument one: comprising basic twin-column oven system with linear, multi-ramp temperature programming and heated injectors for use with packed columns. To be fitted with one electron capture detector and one flame photometric detector and with all associated pneumatic and electrical control systems.

Instrument two: as above but with one electron capture dectector and one nitrogen – phosphorus detector.

Instrument voltage requirements will need to be specified.

Spares, accessories and consumables

(i)	One service kit for each GLC as recommended by the manufacturer		
(ii)	Injection septa		200
	Injection liners (if required)		5
(iii)	Glass GLC columns, empty:	0.5 m	3
		1.0 m	5
		1.5 m	4
	Each column to be complete with all necessary fittings		
	Spare ferrules, graphite, for column installation		25

(iv) Column packing materials, commercially prepared, 50 g quantities of each:

10% DC 200 *or* OV 101 on Chromosorb HP (80-100 mesh)
1:1 mixture of 10% DC 200 and 15% QF1 each coated on Chromosorb W HP (80-100 mesh)
2% DEGS (stabilized) on Chromosorb W HP (80-100 mesh)
3% OV 225 on Chromosorb W HP (80-100 mesh)
Silianized glass wool, 100g

(v) Syringes, hypodermic 10 μl capacity. Needle length will be dependent upon the make and model of GLC. 5

(vi) A selection of tools including screwdrivers, spanners and allen keys will be required. Note that a limited number of tools is provided with the GLC; check to eliminate unnecessary duplication.

Recurrent costs

Recurrent costs will be the continual replacement of materials used from the initial stock. An approximation for the early years, unless the wastage rate or instrument usage is particularly high, would be a cost of about one-fifth of the cost of the initial stock of consumables and accessories.

As the throughput of the laboratory increases, or the range of analyses is increased, together with wear and tear on the instruments and the need to purchase spare parts, then greater recurrent charges can be expected. This is difficult to estimate, but the costs could be about 50% more than in the earlier years; if trouble-free operation is maintained then the costs will be substantially lower.

Provision should also be made where practicable for regular servicing of the equipment perhaps by means of a service contract. This can reduce in the long-term costly 'call-out' charges.

Future requirements

The future GLC requirements must be carefully considered, costed and provision made. This must include a programme of replacement as equipment ages and although a life span of 8-10 years can be expected (more in some cases), analytical developments can make an instrument obsolete very quickly and there may be the need to replace a little earlier. The scope for expansion as experience is developed must also be borne in mind.

Gas supplies

Gas supply and purification systems are detailed on p.9 and those details are not repeated in this model specification.

It is likely that at this scale of operation the balance between cylinder supply or the use of generators is marginal and will need to take into account local factors such as the availability of gases, their purity and cost. However it is recommended that the use of generators should be adopted wherever possible. The retention of a single cylinder of each gas is also recommended as a precaution against instrument 'down time' should a fault develop with any of the generators.

The bench supply can be provided from the generators, with suitable modification and the provision of an in-line reservoir, or from a conventional cylinder supply.

For the GLC system detailed above, a supply of the following gases will be required:

- nitrogen
- hydrogen
- compressed air

Gas generators (Specifications are given on p.11)

(i) Combined nitrogen and compressed air generator, 0.75 l/min nitrogen and 1l/min compressed air or similar
(ii) Hydrogen, output capacity 300 ml/min or similar

Miscellaneous

(i) Cylinder head gauges, one per gas for the generator back up. If cylinder supply is to be used instead of generators then a spare gauge for hydrogen and one for nitrogen will be required. Gauges will also be required for each of the bench supply gas cylinders
(ii) Moisture filters for each gas and additionally an oxygen filter for the nitrogen supply.
Three spare moisture filters and one spare oxygen filter
Mounting brackets and fittings
The bench supply should not require filters
(iii) Copper tubing, ⅛ inch (3.2 mm) as in *Laboratory piping and fittings*, p.12.
(v) Tools for cutting tubing, etc. as listed on p.13.

Recurrent costs

Recurrent costs will depend upon the method of gas supply. Cylinders will need to be recharged regularly and these incur rental charges unless they need to be purchased (which is expensive, although preferable) together with the cost of the gases and any freight or delivery charges. Generators may require maintenance and there will be the need to renew any associated filters. The hydrogen generator should have provision for the replacement of the palladium catalytic cell after a period, depending upon its rate of use (3-6 years).

The on-line filters need to be changed or recharged at a rate defined by the indicator; moisture filters may need to be changed more frequently in a humid climate if used with generated air or nitrogen.

Provision should also be made for the purchase of a small amount of tubing and some fittings to take into account any movement of equipment or replumbing.

Data handling

This scale of operation, limited to the analysis of pesticide residues, requires only modest data handling facilities which can be met more than adequately by the use of two potentiometric chart recorders with an input signal range from 1 mv to 1 v. Initial purchase should include a minimum of 20 rolls of chart paper and a similar number of pens if the fibre-tipped type are chosen. Ink-fed pens can be used, although these can dry up quickly in tropical climates and are not recommended.

The replacement of chart paper and pens will be a recurrent charge at a rate dependent upon use or abuse.

General laboratory equipment

Equipment

(i) Analytical balance, single pan, electronic, weighing range 0-200 g, readable to four decimal places (or similar)
(ii) Toploading balance, dual range, weighing range a) 0-300 g readable to two decimal places and weighing range b) 0-3000 g readable to one or two decimal places (or similar)
(iii) Laboratory oven, 0°-250°C, fan convection, capacity 150 l (or similar)
(iv) Laboratory hotplate, rectangular, approximate size 23 × 30 cm
(v) Water deionizer or water distillation unit, wall mounted, maximum flow rate 10 l/h

(vi) Electrothermal heating mantle (for solvent redistillation/purification), 5-litre flask capacity with integral energy regulator

(vii) Refrigerator/freezers

(a) Refrigerator, spark proof, approximately 120 litres capacity for storage of reference standard solutions

(b) Refrigerator, spark proof, approximately 200 litres capacity for storage of sample extracts

(c) Freezer, $-10°$ to $-20°C$ approximately 300 litres capacity, for storage of analytical samples

(viii) Cooled water bath/circulator 1

(ix) *Option* If water-generated vacuum is not practicable then a vacuum pump will be required resistant to water, solvent and corrosive gases (e.g. Genevac CVP 50)

(x) Laboratory bench-top centrifuge, capacity for 4×200 or 250 ml containers.
To be supplied with rotor assembly and sealed cups and adaptor for use with tubes of 10 and 50 ml capacity. 6 of the 200/250 ml size containers and 12 of each of the 10 and 25 ml tubes to be included

Recurrent costs

A water deionizer will require the purchase of fresh cartridges at intervals and, if this option is chosen, provision should be made for this. None of the other items detailed above has a similar requirement, although funds should be available to meet the cost of replacement parts and servicing should faults develop. Longer term provision must also be made for the replacement of equipment with age. It is difficult to forecast the working life of individual items, although for most a life of 10-15 years can be expected barring accidents.

Equipment for sample grinding and extraction

Equipment

(i) Domestic coffee grinders, each with two spare blades 2

(ii) Waring blender, spark proof with maceration containers of 2 pint and 500 ml capacity with relevant adaptor. Spare drive assemblies and blades for each container

(iii) Electrothermal Soxhlet extraction heater, 6-place, 500 ml flask size

Recurrent charges

Recurrent costs will not be incurred as such although there will be the need for funds to be available for the purchase of spare parts and to meet service charges as necessary; the demands of high-speed sample grinding on equipment can be heavy.

Provision must be made for the replacement of the grinding equipment with time. The domestic coffee grinder will have a shorter life span than the Waring blender although it is felt that the advantages of the coffee grinder for the treatment of small samples of, e.g., a cereal, together with its relative cheapness, still makes this viable.

Equipment for sample processing

Equipment

No./quantity required

(i) Flask shaker, 6- or 8-place with 60 minute timer

(ii) Water bath, 1kW with power controller. Circular with concentric rings and centre cover. Fitted

	No./quantity required
with constant level device. Diameter 20 cm and depth 13 cm or similar	2
(iii) Rotary vacuum evaporator with temperature controlled water bath: Buchi Rotavapor RE 121 or similar	2
With:	
Vacuum controller	3
Spare receiving flasks	4
Vapour ducts B 24/29 cone	4
Spare gasket seals	8
Clips for securing receiver and distillation flasks	10
Mesh-type material to cover condenser and receiving flask to reduce risk from glass implosion under vacuum	

Recurrent charges

The above items will incur little in the way of recurrent charges, although the rotary evaporator will require periodic replacement of vacuum seals and there is the possibility of damage to the glass parts of the system necessitating repair or replacement. As noted for other items of equipment, there will need to be provision for replacement with time.

Glassware and miscellaneous items

General laboratory glassware

	No./quantity required
(i) Volumetric flasks, Grade B (glass stoppered – *see* (ix))	
5 ml	20
10 ml	50
15 ml	10
20 ml	10
25 ml	30
50 ml	25
100 ml	50
250 ml	10
500 ml	10
1000 ml	5
(ii) Round-bottomed flasks, Quickfit	
50 ml, B24 neck, short	15
250 ml, B24 neck, short	15
500 ml, B24 neck, short	25
1000 ml, B24 neck, short	5
(iii) Conical flasks, Quickfit	
150 ml, B24 neck	10
250 ml, B24 neck	12
500 ml, B24 neck	5
(iv) Measuring cylinders, lipped	
10 ml	3
25 ml	10
50 ml	10
100 ml	10
250 ml	6
500 ml	3
1000 ml	1

	No./quantity required
(v) Measuring cylinders, stoppered	
10 ml	5
25 ml	10
50 ml	10
100 ml	25
250 ml	5
1000 ml	1
(vi) Beakers, squat form, lipped	
50 ml	5
150 ml	10
250 ml	10
500 ml	5
1000 ml	2
(vii) Pipettes, Class B	
Graduated 0.5 ml	5
1 ml	10
2 ml	10
5 ml	10
10 ml	10
25 ml	2
Pasteur pipettes	
14.5 cm, boxes of 1000	2
23.0 cm, boxes of 1000	2
Pump pipettes, graduated, 10 ml	10
(viii) Separating funnels, pear shape, glass stoppered	
100 ml	10
250 ml	10
500 ml	6
1000 ml	5
(ix) Glass stoppers (do *not* use plastic stoppers for residue analysis)	
size C10 (10/13)	10
C12 (12/14)	20
C14 (14/15)	20
C16 (16/16)	10
B19 (19/17)	20
B24 (24/20)	20
B29 (29/32)	10
(x) Condensers	
Ether condenser, 19/26 socket, 24/29 cone	1
Allihn condenser, 40/38 cone	8
(xi) Soxhlet extractors	
100 ml capacity, 40/38 socket	8
200 ml capacity, 50/42 socket	8
Adapters, 40/38 socket to 50/42 cone	8
(xii) Filtration equipment	
Filter funnels, Pyrex – 55 mm diameter	4
125 mm diameter	6
200 mm diameter	6
Buchner flasks, Quickfit	
B24 socket 250 ml	3
B24 socket 500 ml	3

	No./quantity required
Funnels, Sintaglass, Buchner, sinter porosity 3, 24/29 cone:	
140 ml	2
400 ml	2
Filter funnels, Sintaglass, porosity 3:	
120 ml	10
(xiii) Chromatography columns	
2.2 cm i.d., 60 cm, length, B24 socket and with PTFE stopcock	10
1.5 cm i.d., 12.5 cm, length, fitted with 100 ml solvent reservoir, B24 socket and with PTFE stopcock	10

B Glassware for solvent re-distillation/recovery	No./quantity required
(i) 5-litre round-bottomed flask	1
(ii) Distillation columns, plain, 250 mm	3
(iii) Still head, plain with thermometer pocket	2
(iv) Distillation thermometer, −10 to 250°C	2
(v) Ether condenser, as defined under A (x)	1
(vi) Spiral supports	3
(vii) Fenske helices	1 l

C Glassware for sample distillation/evaporation	No./quantity required
(i) Splash heads, sloping, flask cone 24/29, condenser cone 19/26	2
(ii) Receiver adapters, straight, 24/29 socket	2
(iii) Receiver adapters, vertical with vacuum connection	2

D Storage of pesticide stock solutions	No./quantity required
(i) Hypo vials, capacity 125 ml	72 (pack size)
(ii) Caps and liners	144 of each
(iii) Crimper and decapitator	1 of each

E Miscellaneous glassware	No./quantity required
(i) Desiccator, vacuum, o-ring seal 200 mm with disc	2
(ii) Crystallizing basins:	
121 mm × 65 mm	3
150 mm × 75 mm	3
(iii) Watch glasses, 90 mm	5
(iv) Test-tubes, glass stoppered, graduated, 0-25 ml	25
(v) Aspirator with stopcock, 10 litres	2
(vi) Adapter, cone/rubber tubing, B24 cone	2
(vii) Weighing funnels:	
85 mm	3
100 mm	3
(viii) Glass jars, plastic screw cap, wide neck	
100 ml	40
250 ml	50
500 ml	50

		No./quantity required
(ix)	Glass rods, 7 mm	3 m
(x)	Glass tubing, normal wall, borosilicate glass	
	7 mm	3 m
	9 mm	3 m

F Miscellaneous items		No./quantity required
(i)	Retort stand bases 250×160 mm	8
	Retort stand bases 160×100 mm	6
	Retort stand rods 600× 10 mm	6
	Retort stand rods 1000× 12 mm	8
(ii)	Boss heads	20
	Boss heads, single jaw	12
	Clamps	20
(iii)	Rings, retort stand 70×125 mm	10
	Rings, retort stand 100×100 mm	10
	Rings, retort stand 130× 90 mm	6
(iv)	Scaffolding rods, aluminium alloy	
	1800 mm×13 mm	6
(v)	Spatulas, mixed sizes	8
	Palette knives, assorted sizes	3
	Scoop, 25 ml	1
	Scoop, 50 ml	1
	Forceps, blunt ended	2
	Forceps, pointed ends	2
	Scissors	2
	Tongs, beaker	2
	Tongs, crucible	2
(vi)	Tubing, vacuum:	
	6 mm i.d.	5 m
	7 mm i.d.	5 m
	Tubing, PVC, colourless:	
	6.5 mm i.d.	5 m
	8 mm i.d.	5 m
(vii)	Pipette filler bulbs	4
	Blow balls	4
	Rubber teats, assorted sizes	
	(1.5, 2.0 and 2.5 ml)	10 of each
(viii)	Test-tube rack:	
	6-place, 2.5-3.0 cm tube diameter	2
	12-place, 2.5-3.0 cm tube diameter	2
(ix)	Weighing boats, disposable:	
	100 ml capacity	500
	250 ml capacity	500
(x)	Aluminium foil:	
	1 m width	100 m
	0.5 m width	100 m
	Cotton wool, absorbent	1.5 g
	Glass wool	1 kg
	Tissues, boxes of 200	10
	Polythene bags with closures:	
	10×15 cm	100
	20×25 cm	100
	1000×500 cm	50
(xi)	Gloves, acid-resistant	1
	Gloves, disposable	2 boxes
	Safety spectacles	2
	Goggles	1

		No./quantity required
(xii)	Burner, butane gas	1
	spare gas cartridge	1
(xiii)	Brushes, soft hair, 15 mm	2
	Brushes, beaker, Brushes, bottle, Brushes, burette } assorted sizes	2 of each type
(xiv)	Bowls, washing-up, polythene	3
	Buckets, polythene	3
(xv)	Plastic containers with lids, capacity approximately 350 ml	20
(xvi)	Tubing clips,	
	10-14 mm	20
	13-17 mm	20
(xvii)	Cork rings,	
	75×45 cm	10
	115×85 cm	25
	150×120 cm	5
	240×200 cm	2
(xviii)	Safety screen, 3-panelled, flexible, on feet	1
(xix)	Flammable solvent store, 710×915×483 mm	1
(xx)	Acid store, 710×915×483 mm	1

Option If water generated vacuum is selected there will be a requirement for three metal-bodied water jet filter pumps giving a vacuum of approximately 20 mbar. *Note* that this system will only operate with an adequate water pressure.

Recurrent charges

The rate of consumption of glassware, even with due care, can be quite high and provision should be made for annual replacement at a level of about 10% of the cost of the initial stock. Similarly, a number of the items detailed in section F are consumables and will need replacement at a rate dependent upon use and work patterns.

Reagents and consumables

Filter papers and extraction thimbles

		No./quantity required
(i)	Filter papers,	
	Whatman, number 1* 7 cm	1 box
	15 cm	1 box
	24 cm	1 box
	Whatman, number 4* 7 cm	1 box
	15 cm	1 box
	24 cm	1 box
	Whatman, number 41* 7 cm	1 box
	15 cm	1 box
	24 cm	1 box

* Or equivalents

(ii)	Extraction thimbles, cellulose, single thickness – Whatman or equivalent		
		33×100 mm	10 boxes
		41×123 mm	10 boxes

General reagents	No./quantity required
(i) Sodium sulphate, granular, anhydrous	10 kg
Sodium sulphate, powder, anhydrous	10 kg
(ii) Sodium hydroxide	1 kg
(iii) Sodium chloride	1 kg
(iv) Sodium dichromate	500 g
(v) Anti-bumping granules	250 g
(vi) Self-indicating silica gel	1 kg
(vii) Hydrochloric acid	1.0 l
(viii) Sulphuric acid	2.5 l
All reagents must be Analar grade	
(ix) Laboratory glassware detergent	10 l

Adsorbents	No./quantity required
(i) Florisil, PR grade, 60-80 mesh	4 kg
(ii) Alumina, neutral, activity grade 1	1 kg
(iii) Silicic acid, 100 mesh	100 g
(iv) Bio-beads, S-X3, 200-400 mesh	100 g

Solvents	No./quantity required
All solvents must be glass distilled or pesticide residue grade	
(i) Acetone	50 l
(ii) Acetonitrile	20 l
(iii) Chloroform	10 l
(iv) Cyclohexane	15 l
(v) Dichloromethane	20 l
(vi) Diethyl ether	15 l
(vii) Ether acetate	15 l
(viii) Hexane	60 l

Rate of consumption

The rate of consumption will obviously depend upon the nature of the analyses performed and also on whether any in-laboratory staff training is being conducted. The quantities of materials recommended above should be sufficient for some 200-250 general analyses, although long runs on particular methods will cause an imbalance. The drain on some of the general reagents such as the acids will be slight.

APPENDIX B2
PESTICIDE RESIDUE LABORATORY MODEL SPECIFICATION: LEVEL 2, 600/700 SAMPLES PER ANNUM

Laboratory capacity

A laboratory established with the equipment, facilities and staffing levels detailed below will have a capacity to analyse up to 600/700 samples per annum (approximately 750-800 analyses including replicates and recovery experiments). The nature and range of the analyses that can be conducted will be marginally restricted by equipment limitations; the experience of the staff may also be an initial limiting factor. A laboratory of this nature will commonly be associated with a government department carrying out screening for legislative or public health purposes, or as a regional analytical laboratory in support of local research stations.

Staffing requirements

The optimal staffing level for the laboratory would be three qualified analytical chemists supported by three technicians.

Laboratory accommodation

Space requirements

A total minimum floor area of 75 m^2 with 30 m^2 of available bench surface area is required. Laboratory design should optimize the use of the available space and should have regard to the need for supplies of water, electricity and gases for the analytical instruments (*see Laboratory requirements*, p.5). At this scale of operation the accommodation should be sub-divided to separate each of the main activities as suggested below (minimum areas).

	floor area, m^2	available bench area, m^2
Washing-up room	10	4
Sample preparation and storage	19	7
Sample extraction and clean-up	36	13
Instrument room	10	6

A single fume cupboard will be required in the sample preparation room. Ample under-bench cupboard and drawer storage space must be provided together with shelving for reagents, etc. and storage for solvents and other reagents.

The sample preparation, extraction and clean-up and instrument rooms must all be air-conditioned.

Office accommodation, not detailed here, will also need to be provided for the preparation of analytical reports and the maintenance of laboratory records. Only a minimum of written work should be undertaken in the laboratory itself.

Service requirements and fittings

Water

There is a requirement for a good supply of running water with multiple laboratory outlets.

The washing-up room will require four outlets, two associated with a main sink (60 × 45 × 20 cm or similar) and two wall mounted.

The sample preparation room will require six water outlets, four bench-mounted with associated tun dishes and two outlets to a bench sink (40×30×20 cm or similar).

The main laboratory (sample clean-up) will require seven outlets, two to each of two bench mounted sinks (dimensions as for bench sink above), two bench mounted with tun dishes and one in the fume cupboard.

The instrument room should not have any water outlets.

A hot-water supply will be required for one outlet to the washing-up room sink, all other outlets being cold water.

The washing-up room sink should have a draining board with ample wall-mounted drying racks for glassware. In addition a heated drying cupboard (150 l capacity or similar) will be required.

Many laboratories rely on scientific glassware washing machines for the bulk of glassware cleaning and this is listed in *General laboratory equipment* (x). The use of such a machine is strongly recommended. It should be noted that such use does not obviate the need for the washing-up sink nor for the drying facilities detailed above.

Electricity

A constant, stable power supply is essential. Although some laboratory operations will only require an intermittent supply, some equipment, refrigerators/freezers and the GLCs will be switched on all of the time. It is suggested that the minimum number of power outlets required is:

Washing-up room	4
Sample preparation room	10
Sample-clean up room	15
Instrument room	8

The instrument room will require a 'clean' supply, i.e. free from interference, and should also have a voltage stabilizer if power fluctuations are likely.

Gas supplies

There is a requirement for supplies of nitrogen, hydrogen and compressed air to the instrument room for the operation of the GLCs (*see* p.9) and for one bench outlet each for nitrogen and compressed air in the sample clean-up room.

Consideration will need to be given to the plumbing for these supplies and as to whether most of the requirement should be met by the use of generators or from cylinder supplies (*see* p.9). It should be noted that bench supply is practicable from a generated source but the pressure drop can affect the GLCs supplied from this source. The use of a line tank with suitable pressure controls can virtually eliminate this effect, however, if attention is given in the design specification to overall demand, GLC pressure requirements, etc.

Safety

Laboratory safety must be taken into full account when designing the laboratory and its internal layout; national safety regulations and, additionally, 'local' safety policies must be strictly followed. A minimum of safety equipment has been detailed on p.58, G (xi), but this should be extended as necessary to comply with safety policy.

Laboratory overalls for members of staff are not included in the list.

Equipment

Gas-liquid chromatography

Instrument one: comprising basic twin-column oven system with linear multi-ramp temperature programming and with one heated packed column injector

and one capillary injector for split/splitless operation. To be fitted with one electron capture detector and one flame photometric detector and with all associated pneumatic and electrical control systems.

Instrument two: as for the above specification except for the detector configuration which should be for one electron capture detector and one nitrogen phosphorus detector.

Instrument voltage requirements will need to be specified.

Spares, accessories and consumables

		No./quantity required
(i)	One service kit for each of the GLCs as recommended by the manufacturer	
(ii)	Injection septa	200
	Injection liners (if required)	5 for packed column injectors 5 for capillary injectors
(iii)	Glass GLC columns, empty	
	0.5 m	3
	1.0 m	6
	1.5 m	3

(iv) Column packing materials, commercially prepared, 50 g quantities of each:

10% DC 200 *or* OV 101 on Chromosorb HP (80-100 mesh)
1:1 mixture of 10% DC 200 and 15% QF1 each coated on Chromosorb W HP (80-100 mesh)
2% DEGS (stabilized) on Chromosorb W HP (80-100 mesh)
3% OV 225 on Chromosorb W HP (80-100 mesh)
Silianized glass wool, 100 g

Alternatively the individual stationary phases and support materials can be purchased within the cost of the commercially prepared materials and suitable mixtures prepared in the laboratory. This has the advantage, as long as the mixtures can be prepared properly, that a wider range of mixtures can be prepared, and in greater quantities, for the same initial outlay. Such preparation should only be undertaken by experienced personnel, and if doubts exist then the commercially prepared mixtures should be obtained.

(v) Capillary columns for GLC:

25 m×0.2 mm i.d. fused silica DB 1	2
25 m×0.2 mm i.d. fused silica OV 17	2
25 m×0.2 mm i.d. fused silica OV 210	2

(vi) Syringes, hypodermic, needle length will be dependent upon the make and model of GLC.

10 µl	5
2 µl	3
1 µl	2

(vii) A selection of tools including screwdrivers, spanners and Allen keys will be required. Note that a limited number of tools is provided with the GLC; check to prevent unnecessary duplication.

Recurrent costs

Recurrent costs will be the continual replacement of materials used from the initial stock. An approximation for the early years, unless the wastage rate or instrument usage is particularly high, would be a cost of about one-fifth of the cost of the initial stock of consumables and accessories.

As the throughput of the laboratory increases or the range of analyses is increased, coupled with wear and tear on the instruments and the need to

purchase spare parts, then greater recurrent charges can be expected. This is difficult to estimate, but the costs could be about 50% more than in the earlier years; if trouble-free operation is maintained then the costs will be substantially lower.

Provision should also be made where practicable for regular servicing of the equipment perhaps by means of a service contract if this is possible. This can reduce in the long term costly 'call-out' charges.

Future requirements

The future GLC requirements must be carefully considered, costed and provision made. This must include a programme of replacement as equipment ages and although a life span of 10 years can be expected (more in some cases) analytical developments can make an instrument obsolete very quickly and there may be the need to replace a little earlier. The scope for expansion as experience is developed must also be borne in mind.

Gas supplies

Gas supply and purification systems are detailed on p. 9 and those details are not repeated here.

At this scale of operation the use of gas generators is recommended. The retention of a single cylinder of each gas is also recommended as a precaution against instrument 'down-time' should a fault develop with any of the generators. For the GLC systems detailed above, a supply of the following gases will be required:

- nitrogen
- hydrogen
- compressed air

Gas generators

(specifications are given on p. 11)

(i) Combined nitrogen and compressed air generator, output capacity 0.75 l/min (or similar) of nitrogen and 1 l/min of compressed air

(ii) Hydrogen, outlet capacity 300 ml/min or similar

Miscellaneous

(i) Cylinder head gauges, one per gas (for back up cylinders) plus one each for the bench supply if a cylinder source is used.

(ii) Moisture filters for each gas and additionally an oxygen filter for the nitrogen supply
Three spare moisture filters and one spare oxygen filter
Mounting brackets and fittings
Filters are not normally required on the bench supply

(iii) Copper tubing, ⅛ inch, (3.2 mm) 10 m
¼ inch, (6.4 mm) 10 m

(iv) Assorted fittings, ⅛ and ¼ inch (3.2 and 6.4 mm) as listed and calculated to be adequate for the proposed gas distribution network. The following will give a guide to the quantities required:

T-pieces, ¼ inch (6.4 mm)	5
T-pieces, ⅛ inch (3.2 mm)	5
4-way crosses, ¼ inch (6.4 mm)	3
4-way crosses, ⅛ inch (3.2 mm)	3
Unions, ¼ inch (6.4 mm)	5
Unions, ⅛ inch (3.2 mm)	5
¼ – ⅛ inch reducers (6.4–3.2 mm)	8
¼ inch (6.4 mm) nuts	20
⅛ inch (3.2 mm) nuts	20
¼ inch (6.4 mm) front and back ferrules	20
⅛ inch (3.2 mm) front and back ferrules	20

(v) Tools for cutting tubing, etc. as listed on p. 13.

Recurrent costs

The generators may require maintenance and there will be the need to renew any associated filters. The hydrogen generator should have provision for the replacement of the palladium catalytic cell after a period depending upon its rate of use (3-6 years).

The on-line filters need to be changed or recharged at a rate defined by the indicator; moisture filters may need to be changed more frequently in a humid climate.

Provision should also be made for the purchase of a small amount of tubing and some fittings to take into account any moving of equipment or replumbing.

Data handling

The following data handling facilities are recommended to complement the GLC systems defined earlier.

Chart recorders

(i) Potentiometric strip chart recorder, input range 1 mv to 1 v, variable chart speed. 3
(ii) Chart paper for recorders, 20 rolls
(iii) Pens for recorders: of the two pen systems available – ink or felt-tip, the felt-tip system is recommended. This incurs a recurrent cost but is felt to be the better system. Twenty pens will initially be required.

Integration system

It is recommended that if access to a more sophisticated form of data handling is required, integration systems, good as they are, should be rejected in favour of a computerized chromatography data system, of which a number are currently available. Laboratories in developing countries will need to take care with power supplies and ambient temperature control. It is not recommended for immediate installation into a new laboratory with relatively untrained staff, but more as of a later development.

Chromatography data system

A number of systems of varying degrees of sophistication are available commercially. Some are part of a total laboratory information system package, but can also be bought separately for use with chromatographic systems – GLC or HPLC. They are expensive as the requirement is for a computer with the necessary interface between the chromatograph, together with the relevant software. The system can be expanded with time and the capacity is large. Data manipulation is possible and with the right software can be a labour-saving device. An advantage is that the computer is available for other purposes whilst not being used for data capture.

Individual system specifications vary and the Nelson 3000 chromatography system is used here as an example:

(i) Computer (minimum requirement): IBM XT or equivalent, 640K RAM, 10Mb hard disk, single 360K diskette drive, monochrome display, parallel printer interface, keyboard, DOS 3.0 operating system, Hercules monochrome graphics card (Optional software: word processing and spread sheet packages)
(ii) Nelson chromatography 2600 system (hardware and software)
Model 3000K data system kit – Model 2600
Software, Model 2T-1444 GPIB controller+cable
Model 761S intelligent interface
(iii) Printer – Epson dot matrix graphics printer

(iv) Paper supply for the printer — 1000 sheets
Printer ribbons — 4
(v) Diskettes — 3 boxes (of 10)

Recurrent costs

(i) Recurrent costs for the chart recorders will be limited to the purchase of fresh chart paper and felt-tip pens. With routine maintenance the recorders should have a life of about 10 years or more.
(ii) Recurrent costs for the chromatography data system will come under two headings. The first is the replacement of paper and printer ribbons for the printer and the other is the purchase of software updates after the initial updating period expires. The purchase of the updates, although not essential, is recommended in order to maintain an awareness of developments.

Thin-layer chromatography

At this level of operation there should be facilities for the use of TLC although the limitation on usage will be the expertise available. It is likely that in most developing countries, commercially manufactured plates will be difficult to obtain and expensive and that the emphasis will be on the use of plates prepared in the laboratory. Suitable commercially prepared plates are detailed below, however, for completeness.

Materials for plate preparation

(i) Plate leveller with capacity for 5×20 cm glass plates
Spreader, adjustable for layer thicknesses between 100 and 2000 µm.
(ii) Glass plates, 20×20 cm — 25
20×10 cm — 25
(iii) Plate carrying rack for 10 plates of 20×20 cm
(iv) Plate storage box for 20 plates of 20×20 cm
(v) Sorbents:
Silica gel G — 2.5 kg
Silica gel G/UV 254 — 2.5 kg

Commercially manufactured plates

(i) Silica gel G, 10×20 cm, 0.25 mm — pack of 50
(ii) Silica gel G UV 254, 10×20 cm, 0.25 mm — pack of 50
(iii) Silica gel C18, 10×10 cm, 0.20 mm — pack of 25

Accessories

(i) UV viewing cabinet (short wave, 254 nm)
(ii) TLC spray cabinet with fume extraction unit (if purchased without fume extraction unit, should be operated in fume cupboard or under a fume hood)
(iii) Separating chambers, with lids, for plates of up to 20×20 cm — 3
(iv) Reagent spray packs (comprising spray reservoir, spray head and propellant canister) — 6 units
Spare propellant canisters — 10
(v) Disposable spotting pipettes:

2 µl, 5 µl, 10 µl — 2 vials of each (100 per vial)

Reagents

The reagent list is not comprehensive and particular analyses may require the purchase of additional reagents

(i)	2,2-oxydiethanol	25 g
(ii)	2,6-dichloro-p-benzoquinone chloroimine	25 g
(iii)	4-(4-nitrobenzyl)pyridine	25 g
(iv)	3,6,9-triazaundecamethylenediamine	25 g
(v)	4-nitrobenzenediazonium tetrafluoroborate	25 g
(vi)	Fluorescein	25 g
(vii)	Silver nitrate	25 g
(viii)	Magnesium chloride	50 g
(ix)	o – toluidine	25 g
(x)	Potassium iodide	50 g
(xi)	p-dimethylaminobenzaldehyde	25 g
(xii)	Indoxyl acetate	25 g
(xiii)	Tris buffer	25 g
(xiv)	Pig liver esterase	

General laboratory equipment

Equipment	No./quantity required
(i) Analytical balance, single pan, electronic, weighing range 0-200 g readable to four decimal places (or similar)	
(ii) Toploading balance, dual range weighing range a) 0-300 g readable to two decimal places and weighing range b) 0-3000 g readable to one or two decimal places (or similar)	
(iii) Laboratory oven, 0°-250 °C, fan convection, capacity 150 l or similar	
(iv) Laboratory hotplate, rectangular, approximate size 23 × 30 cm	
(v) Water distillation unit or deionizer, wall mounted, maximum flow rate 10 l/h	
(vi) Electrothermal heating mantle (for solvent redistillation/purification) 5-l flask capacity with integral energy regulator	
(vii) Refrigerator/freezers	
(a) Refrigerator, spark-proof, capacity 200 l, for storage of reference standard solutions	
(b) Refrigerator, spark-proof, capacity 200 l, for storage of sample extracts	2
(c) Freezer, −10° to −20°C, capacity 500 l, for storage of analytical samples	
(viii) UV/visible spectrophotometer, double beam 1 cm and 4 cm matched absorption cells, set of four of each	
(ix) *Option* If water-generated vacuum is not practicable then a vacuum pump will be required. This must be resistant to water, solvent and corrosive gases (e.g. Genevac CPV 50)	
(x) Laboratory glassware washing machine with suitable baskets and attachments for beakers, flasks, volumetric flasks and pipettes	
Deionizer cartridge, where required	
Detergent	10 packs
Acid rinse solution	10 packs
(xi) Cooled water bath/circulator	2

	No./quantity required
(xii) Laboratory bench-top centrifuge, capacity for 4×200 or 250 ml containers To be supplied with rotor assembly and sealed cups and adaptor for use with tubes of 10 and 50 ml capacity. 6 of the 200/250 ml size containers and 12 of each of the 10 and 25 ml tubes to be included	
(xiii) Electrothermal heating mantle, 500 ml capacity	2

Recurrent costs

The water deionizer will require the purchase of fresh cartridges at intervals and provision should be made for this. Developing country laboratories may experience supply delays necessitating the retention of a large reserve of cartridges. Alternatively, water distillation should be used wherever practicable, including for use with the washing machine, although a large reservoir will be needed together with a line pump to satisfy the washing machine pressure requirements.

None of the other items detailed above has a similar requirement although funds should be available to meet the cost of replacement parts and servicing should faults develop. Longer term provision must also be made for the replacement of equipment with age. It is difficult to forecast the working life of individual items, although for most a life of 10-15 years can be expected barring accidents.

Equipment for sample grinding and extraction

Equipment	No./quantity required
(i) Domestic coffee grinder with two spare blades	2
(ii) Waring blender, spark proof with maceration containers of 1 gallon, 2 pint and 500 ml capacity with relevant adaptor. Spare drive assemblies and blades for each container.	
(iii) Hobart model 84145 (or equivalent) food processor with spare cutting blades	
(iv) Set of good quality knives, stainless steel blade and with wooden handles. Wooden chopping board and sharpener to rehone the knife blades as necessary	
(v) Electrothermal Soxhlet extraction heater, 6-place, 500 ml flask size.	2

Recurrent charges

Recurrent costs will not be incurred as such although there will be the need for funds to be available for the purchase of spare parts and to meet service charges as necessary; the demands of high-speed sample grinding on equipment can be heavy.

Provision must be made for the replacement of the grinding equipment with time. The domestic coffee grinders will have a shorter life span than the Waring blender although it is felt that the advantages of the coffee grinder for the treatment of small samples of, e.g., a cereal, together with its relative cheapness still makes this viable.

Equipment for sample processing

Equipment	No./quantity required
(i) Flask shaker, 6- or 8- place with 60-minute timer	
(ii) Water bath, 1 kW with power controller. Circular with concentric rings and centre cover. Fitted	

	No./quantity required
with constant level device. Diameter 20 cm and depth 13 cm or similar	
(iii) Rotary vacuum evaporator with temperature controlled water bath: Buchi Rotavapor RE 121 or similar	2
with:	
Vacuum controller	3
Spare receiving flasks	4
Vapour ducts, B 24/29 cone	4
Spare gasket seals	8
Clips for securing receiver and distillation flasks	10
Mesh-type material to cover condenser and receiving flask to reduce hazard from glass implosion under vacuum	

Recurrent charges

The above items will incur little in the way of recurrent charges although the rotary evaporator will require periodic replacement of vacuum seals and there is the possibility of damage to the glass parts of the system necessitating repair or replacement. As noted for other items of equipment there will need to be provision for replacement in time.

Glassware and miscellaneous items

A General laboratory glassware	No./quantity required
(i) Volumetric flasks, Grade B (glass stoppered – see (ix))	
5 ml	20
10 ml	50
15 ml	20
20 ml	25
25 ml	50
50 ml	30
100 ml	50
250 ml	10
500 ml	12
1000 ml	6
(ii) Round-bottomed flasks, Quickfit	
50 ml, B24 neck, short	15
250 ml, B24 neck, short	25
500 ml, B24 neck, short	30
1000 ml, B24 neck, short	5
(iii) Conical flasks, Quickfit	
150 ml, B24 neck	15
250 ml, B24 neck	20
500 ml, B24 neck	7
(iv) Measuring cylinders, lipped	
10 ml	5
25 ml	20
50 ml	20
100 ml	15
250 ml	10
500 ml	6
1000 ml	2
(v) Measuring cylinders, stoppered	
10 ml	10
25 ml	25

		No./quantity required
	50 ml	25
	100 ml	20
	250 ml	10
	1000 ml	2
(vi)	Beakers, squat form, lipped	
	50 ml	10
	150 ml	20
	250 ml	20
	500 ml	10
	1000 ml	4
(vii)	Pipettes, Class B	
	Graduated	
	0.5 ml	10
	1 ml	15
	2 ml	15
	5 ml	20
	10 ml	20
	25 ml	4
	Pasteur pipettes	
	14.5 cm, boxes of 1000	2
	23.0 cm, boxes of 1000	2
	Pump pipettes, graduated, 10 ml	10
(viii)	Separating funnels, pear shape, glass stoppered	
	100 ml	20
	250 ml	20
	500 ml	10
	1000 ml	5
(ix)	Glass stoppers	
	(do *not* use plastic stoppers for residue analysis)	
	size C10 (10/13)	50
	C12 (12/14)	60
	C14 (14/15)	60
	C16 (16/16)	20
	B19 (19/17)	30
	B24 (24/20)	40
	B29 (29/32)	15
(x)	Condensers	
	Ether condenser,	
	19/26 socket, 24/29 cone	1
	Allihn condenser,	
	40/38 cone	8
	50/42 cone	8
(xi)	Soxhlet extractors	
	100 ml capacity, 40/38 socket	12
	200 ml capacity, 50/42 socket	12
(xii)	Filtration equipment	
	Filter funnels, Pyrex – 55 mm diameter	6
	125 mm diameter	10
	200 mm diameter	10
	Buchner flasks, Quickfit	
	B24 socket 250 ml	5
	B24 socket 500 ml	5

	No./quantity required
Funnels, Sintaglass, Buchner, sinter porosity 3, 24/29 cone:	
140 ml	4
400 ml	4
Filter funnels, Sintaglass, porosity 3:	
120 ml	15
(xiii) Chromatography columns	
2.2 cm i.d., 60 cm length, B24 socket and with PTFE stopcock	15
1.5 cm i.d., 12.5 cm length, fitted with 100 ml solvent reservoir, B24 socket and with PTFE stopcock	10

B Glassware for solvent re-distillation/recovery	No./quantity required
(i) 5-l round-bottomed flask	2
(ii) Distillation columns, plain, 250 mm	5
(iii) Still head, plain with thermometer pocket	3
(iv) Distillation thermometer, −10° to 250°C	3
(v) Ether condenser, as defined under A(x)	2
(vi) Spiral supports	
(vii) Fenske helices	2 l

C Glassware for sample distillation/evaporation	No./quantity required
(i) Splash heads, sloping, flask cone 24/29, condenser cone 19/26	4
(ii) Receiver adapters, straight, 24/29 socket	4
(iii) Receiver adapters, vertical with vacuum connection	4

D Storage of pesticide stock solutions	No./quantity required
(i) Hypo vials, capacity 125 ml	144 (pack size = 72)
(ii) Caps and liners	288 of each
(iii) Crimper and decapitator	1 of each

E Miscellaneous glassware	No./quantity required
(i) Desiccator, vacuum, O–ring seal, 200 mm with disc	3
(ii) Crystallizing basins:	
121 × 65 mm	5
150 × 75 mm	5
(iii) Basins, porcelain:	
105 mm	3
148 mm	3
(iv) Watch glasses, 90 mm	10
(v) Test tubes, glass stoppered, graduated 0-25 ml	25
(vi) Aspirator with stopcock, 10 l	3
(vii) Adapter, cone/rubber tubing, B24 cone	3
(viii) Weighing funnels:	
85 mm	5
100 mm	5
(ix) Glass jars, plastic screw cap, wide neck	
100 ml	40
250 ml	50
500 ml	50

	No./quantity required
(x) Glass rods, 7 mm	3 m
(xi) Glass tubing, normal wall, borosilicate glass	
7 mm	3 m
9 mm	3 m

F Glassware for dithiocarbamate/head space analysis	No./quantity required
(i) Glass bottles, Pyrex with plastic screw caps capable of being drilled to make a 3 mm hole	
250 ml	6
500 ml	6
Septa to fit screw caps	50
(ii) Round-bottomed flasks, 500 ml, three neck, centre socket B24/29 and side sockets B 19/26	5
(iii) Dropping funnels with B 19/26 cone	5
(iv) Air leak tubes	6
(v) Condenser, Liebig, 400 mm B 24 socket and cone	3
(vi) Gas washbottles, Sintaglass (porosity 1), Dreschel, 125 ml capacity. Inlet/outlet tubes to be fitted with female/male spherical joints (size S13)	5
Spare heads	6 (to be altered by glassblower to fit the tubes listed below, 3 for each)
(vii) Glass tubes, Quickfit, B24/29 socket	
150 mm	6
200 mm	6
(viii) Glass cone, B24/29 to male spherical joint, joint size S13 (*see* (vi) above)	3
(ix) Joint clips for holding spherical joints	6
(x) Gas-tight syringes, Luer lock fitting	
10 ml	2
200 µl	2
50 µl	2
Disposable needles, length 50 mm, gauge 19	10

G Miscellaneous items	No./quantity required
(i) Retort stand bases 250×160 mm	12
Retort stand bases 160×100 mm	10
Retort stand rods 600× 10 mm	10
Retort stand rods 1000× 12 mm	15
(ii) Boss heads	30
Boss heads, single jaw	15
Clamps	30
(iii) Rings, retort stand 70×125 mm	15
Rings, retort stand 100×100 mm	15
Rings, retort stand 130× 90 mm	10
(iv) Scaffolding rods, aluminium alloy, 1800×13 mm	10
(v) Spatulas, mixed sizes	15
Palette knives, assorted sizes	5
Scoops, 25 ml	2
50 ml	2
Forceps, blunt ended	3
Forceps, pointed ends	3

		No./quantity required
	Scissors	3
	Tongs, beaker	2
	Tongs, crucible	2
(vi)	Tubing, vacuum:	
	6 mm i.d.	10 m
	7 mm i.d.	10 m
	Tubing, PVC, colourless:	
	6.5 mm i.d.	10 m
	8 mm i.d.	10 m
(vii)	Pipette filler bulbs	6
	Blow balls	6
	Rubber teats, assorted sizes (1.5, 2.0 and 2.5 ml)	25 of each size
(viii)	Test-tube rack:	
	6-place, 2.5-3.0 cm tube diameter	3
	12-place, 2.5-3.0 cm tube diameter	3
(ix)	Weighing boats, disposable:	
	100 ml capacity	1000
	250 ml capacity	1000
(x)	Aluminium foil:	
	1 m width	100 m
	0.5 m width	100 m
	Cotton wool, absorbent	2.5 kg
	Glass wool	1 kg
	Tissues, boxes of 200	20
	Polythene bags with closures,	
	10×15 cm	250
	20×25 cm	250
	1000×500 cm	150
(xi)	Gloves, acid resistant	1
	Gloves, disposable	3 boxes
	Safety spectacles	4
	Goggles	2
	Ear protectors	3
(xii)	Burner, butane gas	1
	Spare gas cartridges	2
(xiii)	Brushes, soft hair, 15 mm	3
	Brushes, beaker Brushes, bottle assorted sizes Brushes, burette	3 of each type
(xiv)	Bowls, washing-up, polythene	3
	Buckets, polythene	4
(xv)	Plastic containers with lids, capacity approximately 350 ml	30
(xvi)	Tubing clips,	
	10-14 mm	25
	13-17 mm	25
(xvii)	Cork rings,	
	75× 45 cm	20
	115× 85 cm	40
	150×120 cm	10
	240×200 cm	3
(xviii)	Safety screen, 3 – panelled, flexible, on feet	2
(xix)	Flammable solvent store, 710×915×483 mm	2
(xx)	Acid store, 710×915×483 mm	1

Option – If water-generated vacuum is selected there will be a requirement for three metal-bodied water jet filter pumps giving a vacuum of approximately 20 mbar. *Note* that this system will only operate with an adequate water pressure.

Recurrent charges

The rate of consumption of glassware, even with due care, can be quite high and provision should be made for annual replacement at a level of about 15% of the cost of the initial stock. Similarly a number of the items detailed in section F above are consumables and will need replacement at a rate dependent upon use and work patterns.

Reagents and consumables

Filter papers and extraction thimbles		No./quantity required
(i) Filter papers,		
Whatman number 1*	17 cm	2 boxes
	15 cm	2 boxes
	24 cm	1 boxes
Whatman number 4*	7 cm	1 box
	15 cm	1 box
	24 cm	1 box
Whatman number 41*	7 cm	2 boxes
	15 cm	2 boxes
	24 cm	1 boxes

* or equivalents

(ii) Extraction thimbles, cellulose, single thickness – Whatman or equivalent	
33 × 100 mm	25 boxes
41 × 123 mm	25 boxes

General reagents	No./quantity required
(i) Sodium sulphate, granular, anhydrous	25 kg
Sodium sulphate powder, anhydrous	15 kg
(ii) Sodium hydroxide	1 kg
(iii) Sodium chloride	2 kg
(iv) Sodium dichromate	1 kg
(v) Anti-bumping granules	500 g
(vi) Self-indicating silica-gel	2 kg
(vii) Hydrochloric acid	1.0 l
(viii) Sulphuric acid	2.5 l
All reagents must be Analar grade or equivalent.	
(ix) Laboratory glassware detergent	10 l

Adsorbents	No./quantity required
(i) Florisil, PR Grade, 60-80 mesh	10 kg
(ii) Alumina, neutral, activity grade 1	2 kg
(iii) Silicic acid, 100 mesh	200 g
(iv) Bio-beads, S-X3, 200-400 mesh	200 g

Solvents	No./quantity required
All solvents must be glass distilled or pesticide residue grade.	
(i) Acetone	150 l
(ii) Acetonitrile	60 l
(iii) Chloroform	20 l
(iv) Cyclohexane	20 l
(v) Dichloromethane	30 l
(vi) Diethyl ether	50 l
(vii) Ethyl acetate	20 l

	No./quantity required
(viii) Ethyl alcohol, absolute	10 l
(ix) Hexane	200 l
(x) Methyl alcohol	10 l
(xi) Petroleum ether, 40°-60°	30 l

Special reagents

Reagents specified here are in addition to those listed above which are required for basic operations. The list is not meant to be comprehensive but to cover some of the main analyses for which reagents other than basic reagents are required. It is hoped that by offering the information in this way it can provide a guide to requirements on an optional basis without incurring unnecessary costs.

	No./quantity required
(i) Dithiocarbamate analysis by CS_2 evolution and determination spectrophotometrically:	
Carbon disulphide, >99.9% pure	100 ml
Cupric acetate monohydrate, AR	250 g
Diethanolamine	250 g
Stannous chloride, AR	250 g
(ii) Dithiocarbamate analysis by head-space analysis following CS_2 evolution:	
Carbon disulphide, >99.9% pure	100 ml
Stannous chloride, AR	250 g
Thiophen, >99.9% pure	100 ml
(iii) Carbamate residues by GLC:	
1-Fluoro-2,4-dinitrobenzene	25 g
Disodium hydrogen phosphate	500 g
(iv) Inorganic bromide residues by GLC:	
Ethylene oxide	100 ml
Di-iso-propyl ether	2.5 l
Ammonium sulphate	250 g

Rate of consumption

The rate of consumption of materials will obviously depend upon the nature of the analyses performed and also on whether any in-laboratory staff training is being conducted. The quantities of materials recommended above should be sufficient for some 700-750 general analyses although long runs on particular methods will cause an imbalance. The drain on some of the general reagents such as the acids will be slight in most cases.

APPENDIX B3
PESTICIDE RESIDUE LABORATORY MODEL SPECIFICATION: LEVEL 3, 1,500 SAMPLES PER ANNUM

Laboratory capacity

A laboratory established with the equipment, facilities and staffing levels detailed below will have a capacity to analyse up to 1,500 samples per annum (approximately 2,000 analyses with replicates and recovery experiments). The nature and range of the analyses that can be conducted will be unrestricted, although the experience of the staff may be an initial limiting factor. A laboratory of this size (and stature) will commonly be associated with a government department carrying out screening for legislative or public health purposes, or as a regional analytical laboratory in support of local research stations and with a much greater required analytical throughput than that given for the model specification, level 2.

Staffing requirements

The optimal staffing level for the laboratory would be five qualified analytical chemists supported by a similar number of technicians. The staffing level, particularly at the junior level, is generous and would allow for training of these individuals.

Laboratory accommodation

Space requirements

A total minimum floor area of 125 m^2 with 51 m^2 of available bench surface area is required. Laboratory design should optimize the use of the available space and should have regard to the need for supplies of water, electricity and gases for the analytical instruments (*see Laboratory requirements*, p. 5). The accommodation must be sub-divided to separate each of the main activities as suggested below (minimum areas)

	floor area, m^2	available bench area, m^2
Washing-up room	12	5
Sample preparation and storage	23	10
Sample extraction and clean-up	68	27
Instrument room	22	9

A separate weighing room for the analytical balance is recommended within the area designated for sample clean up.

A single fume cupboard is required. Ample under-bench cupboard and drawer storage space must be provided together with shelving for reagents etc. Adequate external storage for solvents and other reagents must be available for stocks above the minimum which can be conveniently and safely kept in the laboratory as a working supply.

The full laboratory suite must be air-conditioned.

Office area requirements have not been specified although these should not be overlooked. Because of the nature of the work, only a minimal amount of written work should be undertaken in the laboratory and office accommodation is essential for the preparation of analytical reports, maintenance of records, etc. The head of the laboratory will normally require office accommodation, although in some cases this could be shared with the other members of staff.

Service requirements and fittings

Water

There is a requirement for good supplies of running water with multiple laboratory outlets.

The washing-up room will require 6 outlets, two outlets to each of two large sinks (60 × 45 × 20 cm or similar) and two wall mounted.

The sample preparation room will require 7 water outlets, three bench-mounted with associated tun dishes and two outlets on each of two bench sinks (40 × 30 × 20 cm or similar)

The main laboratory (sample clean-up) will require 10 outlets, two to each of three bench-mounted sinks, dimensions as for bench sinks above, three bench-mounted with tun dishes and one in the fume cupboard.

Warm-water outlets will be required for the washing-up room only, one outlet per sink.

The instrument room should not have any water outlets.

All bench outlets should be contained within a service spine to preserve bench space.

The washing-up sink in the sample preparation room should have a draining board with ample wall-mounted drying racks for glassware. In addition a heated drying cupboard (150 l capacity or similar) will be required.

Many laboratories rely on scientific glassware washing machines for the bulk of glassware cleaning and a machine is listed in *General laboratory equipment* (x), p. 69. The use of a machine does not obviate the need for the washing-up sink nor for the drying facilities detailed above.

Electricity

A constant, stable power supply is essential. Although some laboratory operations will only require an intermittent supply, some equipment, e.g. refrigerators/freezers and the GLCs will be switched on all the time. It is suggested that the minimum number of power outlets required is:

Washing-up room	6
Sample preparation room	12
Sample clean up room	20
Instrument room	15

The instrument room will require a clean supply, i.e. free from interference, and should also have a voltage stabilizer if power fluctuations are likely.

Gas supplies

There is a requirement for supplies of nitrogen, hydrogen, helium* and compressed air to the Instrument room for the operation of the GLCs (*see Gas supplies*, p. 9) and for one bench outlet each for nitrogen and compressed air in the sample preparation and clean-up rooms. It should be noted that bench supply is practicable from a generated source but the pressure drop can affect the GLCs supplied from this source. The use of a line tank with suitable pressure controls can virtually eliminate this effect, however, if attention is given in the design specification to overall demand, GLC pressure requirements, etc.

Safety

Laboratory safety must be taken into full account when designing the laboratory and its internal layout; national safety regulations and, additionally, 'local'

*Helium is for use with ion trap detector (ITD) detailed on p. 63. If country supplies of helium are poor – both in quality and delivery, or supplies have to be imported, then provision of an ITD should not be considered.

safety policies must be strictly followed. A minimum of safety equipment has been detailed on p. 74 (G (xii)) but this should be extended as necessary to comply with safety policy. Laboratory overalls for members of staff are not included in the list.

Equipment

Gas-liquid chromatography

Four instruments will be required as detailed below.

Instrument one: comprising basic, twin-column oven system with linear, multi-ramp temperature programming and with one heated packed column injector and one capillary injector for split/splitless operation. To be fitted with one electron capture detector and one flame photometric detector and with all associated pneumatic and electrical control systems.

Instrument two: as for the above specification except for the detector configuration which should be for one electron capture detector and one nitrogen phosphorus detector.

Instrument three: as for instrument one but fitted with dual electron capture detectors.

Instrument four: basic oven system with linear multi-ramp temperature programming. To be fitted with a single heated capillary injector and all associated pneumatics. Detection system to be an ITD. To be supplied with associated computer system, printer/plotter and all software including reference library.

Instrument voltage requirements will need to be specified when ordering in all cases.

The purchase of instrument four will depend on the cost, availability and quality of helium supplies. If there is doubt about gas supply, the ITD should not be considered.

Spares, accessories and consumables

(i) One service kit for each of the instruments as recomended by the manufacturer

(ii) Injection septa – 300
Injection liners (if required) — 6 for packed column injectors; 6 for capillary injectors

(iii) Glass GLC columns, empty:

0.5 m	4
1.0 m	9
1.5 m	5

(iv) Column packing materials, commercially prepared, 100 g quantities of each:

10% DC 200 *or* OV 101 Chromosorb HP (80-100 mesh)
1:1 mixture of 10% DC 200 and 15% QF1 each coated on Chromosorb W HP (80-100 mesh)
2% DEGS (stabilized) on Chromosorb W HP (80-100 mesh)
3% OV 225 on Chromosorb W HP (80-100 mesh)
Silianized glass wool, 100 g

Alternatively the individual stationary phases and support materials can be purchased within the cost of the commercially prepared materials and suitable mixtures prepared in the laboratory. This has the advantage, as long as the mixtures can be prepared properly, that a wider range of mixtures can be prepared and in greater quantities for the same initial outlay. Such preparation should only be undertaken by experienced personnel and if doubts exist then the commercially prepared mixtures should be obtained.

(v) Capillary columns for GLC:
25×0.2 mm i.d. fused silica DB 1
25×0.2 mm i.d. fused silica OV 17
25×0.2 mm i.d. fused silica OV 210

(vi) Syringes, hypodermic; needle length will be dependent upon the make and model of GLC instrument.

10 μl	6
2 μl	4
1 μl	3

(vi) A selection of tools including screwdrivers, spanners and allen keys will be required. Note that a limited number of tools are provided with the GLC; check to prevent unnecessary duplication.

Recurrent costs

Recurrent costs will be the continual replacement of materials used from the initial stock. An approximation for the early years, unless the wasteage rate or instrument usage is particularly high, would be a cost of about one-fifth of the cost of the initial stock of consumables and accessories.

As the throughput of the laboratory increases with consequential wear and tear on the instruments then greater recurrent charges can be expected. This is difficult to estimate but the costs could be about 50 % more than in the earlier years; if trouble-free operation is maintained then the costs will be substantially lower.

Provision should also be made where possible for regular servicing of the equipment perhaps by means of a service contract where this is practicable. This can reduce in the long term costly 'call-out' charges.

Future requirements

The future GLC requirements must be carefully considered, costed and provision made. This must include a programme of replacement as equipment ages and although a life span of 10 years can be expected (more in some cases) analytical developments can make an instrument obsolete very quickly and there may be the need to replace a little earlier. The scope for expansion as experience is gained must also be borne in mind.

Gas supplies

Gas supply and purification systems are detailed on pp. 9-12 and those details are not repeated here.

At this scale of operation the use of gas generators is recommended although the helium supply for the ITD will need to be provided by cylinder. The retention of a single cylinder each of nitrogen, hydrogen and compressed air is also recommended as a precaution against instrument 'down time' should a fault develop with any of the generators. Cylinders will also be required for the bench supplies if generated supply is not used. For the GLC systems detailed above, a supply of the following gases will be required:

- nitrogen
- hydrogen
- compressed air
- helium (note earlier comments)

Gas generators

(i) Combined nitrogen and compressed air generator, outputs 2 l/min nitrogen and 4 l/min compressed air or similar.
(ii) Hydrogen, output capacity 300 ml/min or similar

Miscellaneous

(i) Cylinder head gauges, one per gas for back up cylinders and two (one spare) for helium. One each for the air and nitrogen bench supply if cylinder supply adopted.

(ii) Moisture filters for each gas and additionally an oxygen filter for the nitrogen and helium supplies. Five spare moisture filters and three spare oxygen filters. Mounting brackets and fittings.

No filters are required for the bench supply gases.

(iii) Copper tubing, ⅛ inch (3.2 mm) 10 m
¼ inch (6.4 mm) 10 m

(iv) Assorted fittings, ⅛ and ¼ inch (3.2 and 6.4 mm) and calculated to be adequate for the proposed gas distribution network. The following will give a guide as to quantities required:

T-pieces, ¼ inch (6.4 mm)	8
T-pieces, ⅛ inch (3.2 mm)	8
4-way crosses, ¼ inch (6.4 mm)	6
4-way crosses, ⅛ inch (3.2 mm)	6
Unions, ¼ inch (6.4 mm)	8
Unions, ⅛ inch (3.2 mm)	8
¼-⅛ inch reducers (6.4-3.2 mm),	10
¼ inch (6.4 mm) nuts	25
⅛ inch (3.2 mm) nuts	25
¼ inch (6.4 mm) front and back ferrules	30
⅛ inch (3.2 mm) front and back ferrules	30

(v) Tools for cutting tubing, etc. listed on p. 13

Recurrent costs

The generators may require maintenance and there will be the need to renew any associated filters. The hydrogen generator should have provision for the replacement of the palladium catalytic cell after a period depending upon its rate of use (3-6 years).

The on-line gas filters need to be changed or recharged at a rate defined by the indicator; moisture filters may need to be changed more frequently in a humid climate.

Provision should also be made for the purchase of a small amount of tubing and some fittings to take into account any moving of equipment or replumbing.

High-performance liquid chromatography

An HPLC system incorporating:
Programmed solvent gradient elution
Pumping system with programmable flow rate in the range of 0-10 ml/min and an operating pressure range of 0-420 bar
Sample loop injection valve
UV/visible fixed wavelength detector with flow-through cell

Consumables and accessories

(i) An instrument service kit as recommended by the manufacturer

(ii) Columns, cartridge system such as Chrompack Chromsep system or similar

1 10 cm hardware kit
1 20 cm hardware kit

1 spare column compressor
1 spare column housing for 10 cm columns
1 spare column housing for 20 cm columns
5 spare column couplings
10 Teflon rings and screens

10 Valco nuts, 1/16 inch (1.6 mm)
20 Valco ferrules, 1/16 inch (1.6 mm)

6 each of the following Chromsep type columns, 10 cm:
Lichrosorb Si 60, 7μm
Spherisorb ODS, C18, 5 μm
Lichrosorb RP, 7 μm

12 Chromsep guard columns for reversed phase columns
12 Chromsep guard columns for silica columns

(iii) Syringes

25 μl	2
50 μl	2
200 μl	2
1,000 μl	2

Syringes, gas tight, 50 ml 2
Needle length and point design will depend on the valve system fitted to the instrument.

(iv) Injection valve loops:

20 μl	2
50 μl	2
200 μl	1
500 μl	1
1,000 μl	1

Recurrent costs

Recurrent costs will depend upon the rate of use and the wear and tear on system components. There will be a need to replace elements of the service kit as they are used, together with syringes and columns. Used properly the columns will have a life-span of several hundred injections but poor technique can dramatically reduce this. The guard columns will have a relatively short life. An approximation for recurrent costs would be something of the order of 25-30% of the initial cost of the consumable materials for the first 2-3 years with a rise to 50% after that. As the system ages provision must also be made for instrument maintenance charges.

Data handling

The following data handling facilities are recommended to complement the gas– and high-performance liquid chromatographic systems defined on pp. 63 and 65.

Chart recorders

(i) Potentiometric strip chart recorder, input range 1 mv to 1 v, variable chart speed 4
(ii) Chart paper for recorders, 25 rolls
(iii) Pens for recorders: of the two systems available, ink or felt-tip, the felt-tip system is recommended. This incurs a recurrent cost but is felt to be the better system; 25 pens will initially be required.

Integration systems

It is recommended that if access to a more sophisticated form of data handling is required, integration systems, as good as they are, should be rejected in favour of a computerized chromatography data system of which a number are currently available. This may cause problems for laboratories in developing countries and care will need to be taken with power supplies and ambient temperature control. It is not recommended for immediate installation into a new laboratory with relatively untrained staff, but as a later development.

Chromatography data system

A number of systems of varying degrees of sophistication are available commercially. Some are part of a total laboratory information system package but can also be bought separately for use with chromatographic systems – GLC or HPLC. They are expensive, as the requirement is for a computer with the necessary interface between the chromatograph together with the relevant software. The system can be expanded with time and the capacity is large. Data manipulation is possible and with the right software can be a labour-saving device. An advantage is that the computer is available for other purposes whilst not being used for data capture.

Individual system specifications vary and the Nelson 3000 chromatography system is used here as an example:

(i) Computer (minimum requirement): IBM XT (or equivalent), 640K RAM, 10Mb hard disk, single 360K diskette drive. Monochrome display, parallel printer interface, keyboard, DOS 3.0 operating system, Hercules monochrome graphics card (Optional software: word processing and spreadsheet packages)

(ii) Nelson chromatography 2600 system (hardware and software)
Model 3000K data system kit – Model 2600 Software, Model 2T-1444 GPIB controller+cable Model 761S intelligent interface — 3

(iii) Printer – Epson dot matrix graphics printer

(iv) Paper supply for the printer — 3000 sheets
Printer ribbons — 8

(v) Diskettes — 3 boxes (of 10)

Recurrent costs

(i) Recurrent costs for the chart recorders will be limited to the purchase of fresh chart paper and felt-tip pens. With routine maintenance the recorders should have a life of about 10 years.

(ii) Recurrent costs for the chromatography data system will come under two headings. The first is (1) the replacement of paper and printer ribbons for the printer and (2) the purchase of software updates after the initial updating period expires. The purchase of the updates although not essential is recommended in order to maintain an awareness of developments.

Thin-layer chromatography

Provision should be allowed for both the use of commercially manufactured plates and for the use of plates prepared in the laboratory. Commercially prepared plates are likely to be both expensive and difficult to obtain and in practice the emphasis will probably be on the use of self-prepared plates. Suitable commercial plates are detailed below, however, for completeness.

Materials for plate preparation

(i) Plate leveller with capacity for 5×20 cm glass plates,
Spreader, adjustable for layer thicknesses between 100 and 2000 μm.

(ii) Glass plates
20×20 cm — 40
20×10 cm — 40
20× 5 cm — 20

(iii) Plate carrying rack for 10 plates of 20×20 cm — 2

(iv) Plate storage box for 20 plates of 20×20 cm

(v) Sorbents:
Silica gel G — 5 kg
Silica gel G/UV 254 — 5 kg

Commercially manufactured plates

(i) Silica gel G, 10×20 cm, 0.25 mm	2 packs of 50
(ii) Silica gel G UV 254, 10×20 cm, 0.25 mm	2 packs of 50
(iii) Silica gel C18, 10×10 cm, 0.20 mm	2 packs of 25

Accessories

(i) UV viewing cabinet (short wave, 254 nm).	
(ii) TLC spray cabinet with fume extraction unit (if purchased without fume extraction unit, should be operated in fume cupboard or under a fume hood)	
(iii) Separating chambers, with lids, for plates of up to 20×20 cm	5
(iv) Reagent spray packs (comprising spray reservoir, spray head and propellant canister)	8 units
Spare propellant canisters	20
(v) Disposable spotting pipettes:	
2 µl	
5 µl	4 vials of each
10 µl	(100 per vial)

Reagents

The reagent list is not comprehensive and particular analyses may require the purchase of additional reagents.

(i) 2, 2-oxydiethanol	25 g
(ii) 2, 6-Dichloro-p-benzoquinone chloroimine	25 g
(iii) 4- (4-nitrobenzyl) pyridine	25 g
(iv) 3, 6, 9-triazaundecamethylenediamine	25 g
(v) 4-nitrobenzenediazonium tetrafluoroborate	25 g
(vi) Fluorescein	25 g
(vii) Silver nitrate	25 g
(viii) Magnesium chloride	50 g
(ix) o – toluidine	25 g
(x) Potassium iodide	50 g
(xi) p-dimethylaminobenzaldehyde	25 g
(xii) Indoxyl acetate	25 g
(xiii) Tris buffer	25 g
(xiv) Pig liver esterase	

General laboratory equipment

Equipment	No./quantity required
(i) Analytical balance, single pan, electronic, weighing range 0-200 g readable to four decimal places (or similar)	
(ii) Toploading balance (A), dual range, weighing range (a) 0-300 g readable to two decimal places and weighing range (b) 0-3000 g readable to one or two decimal places (or similar) Toploading balance (B), weighing range 0-500 g readable to two decimal places	
(iii) Laboratory oven, 0°-250°C, fan convection, capacity 150 l or similar	
(iv) Muffle furnace, 1100°C. Capacity 7.5-10 l	
(v) Laboratory hotplate, rectangular, approximate size 23×30 cm	
(vi) Water distillation unit or deionizer, wall mounted, maximum flow rate 10 l/h	
(vii) Electrothermal heating mantle (for solvent redistillation/purification), 5-litre flask capacity with integral energy regulator	

		No./quantity required
(viii)	Refrigerator/freezers	
	a) Refrigerator, spark proof, aproximately 200 l capacity for storage of reference standard solutions	
	b) Refrigerator, spark proof, approximately 200 l capacity for storage of sample extracts	2
	c) Freezer, −10°C to −20°C approximately 500 litres capacity, for storage of analytical samples	2
(ix)	UV/visible spectrophotometer, double beam 1 cm and 4 cm matched absorption cells	set of four of each
(x)	Laboratory glassware washing machine with suitable baskets and attachments for beakers, flasks, volumetric flasks and pipettes	
	Deionizer cartridge, where required	
	Detergent	10 packs
	Acid rinse solution	10 packs
(xi)	Cooled water bath/circulator	3
(xii)	*Option* If water generated vacuum is not practicable then a vacuum pump will be required such as the Edwards Speedivac 2 or similar	
(xiii)	Laboratory bench–top centrifuge, capacity for 4 × 200 or 250 ml containers To be supplied with rotor assembly and sealed cups and adapter for use with tubes of 10 and 50 ml capacity. 8 of the 200/250 ml size containers and 12 of each of the 10 and 25 ml tubes to be included	
(xiv)	Electrothermal heating mantle, 500 ml capacity	2

Recurrent costs

The water deionizer will require the purchase of fresh cartridges at intervals and provision should be made for this. None of the other items detailed above has a similar requirement although funds should be available to meet the cost of replacement parts and servicing should faults develop. Longer-term provision must also be made for the replacement of equipment with age. It is difficult to forecast the working life of individual items although for most a life of 10-15 years can be expected barring accidents.

Equipment for sample grinding and extraction

Equipment		No./quantity required
(i)	Domestic coffee grinder with two spare blades	3
(ii)	Waring blender, spark proof with maceration containers of 1 gallon, 2 pint and 500 ml capacity with relevant adapter. Spare drive assemblies and blades for each container	
(iii)	Hobart model 84145 (or equivalent) food processor with spare cutting blades	
(iv)	Set of good quality knives, stainless steel blade and with wooden handles. Wooden chopping board and sharpener to rehone the knife blades as necessary	
(v)	Electrothermal soxhlet extraction heater, six place, 500 ml flask size	2

Recurrent charges

Recurrent costs will not be incurred as such although there will be the need for funds to be available for the purchase of spare parts and to meet service charges as necessary; the demands of high-speed sample grinding on equipment can be heavy.

Provision must be made for the replacement of the grinding equipment with time. The domestic coffee grinders will have a shorter life span than the Waring blender although it is felt that the advantages of the coffee grinder in the treatment of small samples of, e.g. a cereal, together with its relative cheapness still makes this a viable proposition.

Equipment for sample processing

Equipment	No./quantity required
(i) Flask shaker, 6 or 8 place with 60 minute timer	
(ii) Water bath, 1 kW with power controller. Circular with concentric rings and centre cover. Fitted with constant level device. Diameter 20 cm and depth 13 cm or similar	2
(iii) Rotary vacuum evaporator with temperature controlled water bath: Buchi Rotavapor RE 121 or similar	2
with:	
Vacuum controller	3
Spare receiving flasks	3
Vapour ducts, B24/29 cone	4
Spare gasket seals	5
Clips for securing receiver and distillation flasks	5
Mesh covering material for condenser and receiving flasks to reduce hazard from glass implosion under vacuum	

Recurrent charges

The above items will incur little in the way of recurrent charges although the rotary evaporator will require periodic replacement of vacuum seals and there is the possibility of damage to the glass parts of the system necessitating repair or replacement. As noted for other items of equipment there will need to be provision for replacement with time.

Glassware and miscellaneous items

A General laboratory glassware	No./quantity required
(i) Volumetric flasks, Grade B (glass stoppered – see (ix))	
5 ml	30
10 ml	100
15 ml	30
20 ml	40
25 ml	100
50 ml	50
100 ml	100
250 ml	15
500 ml	10
1,000 ml	4
(ii) Round-bottomed flasks, Quickfit	
50 ml, B24 neck, short	25
250 ml, B24 neck, short	50
500 ml, B24 neck, short	50
1,000 ml, B24 neck, short	5

	No./quantity required
(iii) Conical flasks, Quickfit	
150 ml, B24 neck	20
250 ml, B24 neck	30
500 ml, B24 neck	10
(iv) Measuring cylinders, lipped	
10 ml	10
25 ml	30
50 ml	30
100 ml	20
250 ml	15
500 ml	10
1,000 ml	4
(v) Measuring cylinders, stoppered	
10 ml	20
25 ml	50
50 ml	50
100 ml	30
250 ml	15
1,000 ml	4
(vi) Beakers, squat form, lipped	
50 ml	20
150 ml	30
250 ml	40
500 ml	20
1,000 ml	6
(vii) Pipettes, Class B	
Graduated:	
0.5 ml	15
1 ml	20
2 ml	20
5 ml	30
10 ml	30
25 ml	5
Pasteur pipettes	
14.5 cm, boxes of 1,000	5
23.0 cm, boxes of 1,000	5
Pump pipettes, graduated, 10 ml	15
(viii) Separating funnels, pear shape, glass stoppered	
100 ml	25
250 ml	30
500 ml	20
1,000 ml	8
(ix) Glass stoppers	
(do *not* use plastic stoppers for residue analysis)	
size C10 (10/13)	50
C12 (12/14)	100
C14 (14/15)	100
C16 (16/16)	30
B19 (19/17)	50
B24 (24/20)	75
B29 (29/32)	15
(x) Condensers	
Ether condenser, 19/26 socket, 24/29 cone	2
Allihn condenser,	
40/38 cone	8
50/42 cone	8

	No./quantity required
(xi) Soxhlet extractors	
100 ml capacity, 40/38 socket	15
200 ml capacity, 50/42 socket	15
(xii) Filtration equipment	
Filter funnels, Pyrex, 55 mm diameter	10
125 mm diameter	15
200 mm diameter	20
Buchner flasks, Quickfit	
B24 socket 250 ml	6
B24 socket 500 ml	6
Funnels, Sintaglass, Buchner, sinter porosity 3, 24/29 cone:	
140 ml	5
400 ml	5
Filter funnels, Sintaglass, porosity 3:	
120 ml	20
(xiii) Chromatography columns	
2.2 cm i.d., 60 cm in length, B24 socket and with PTFE stopcock	25
1.5 cm i.d., 12.5 cm in length, fitted with 100 ml solvent reservoir, B24 socket and with PTFE stopcock	15

B Glassware for solvent re-distillation/recovery	No./quantity required
(i) 5-litre round-bottomed flask	2
(ii) Distillation columns, plain, 250 mm	5
(iii) Still head, plain with thermometer pocket	3
(iv) Distillation thermometer, −10° to 250°C	3
(v) Ether condenser, as defined under A(x)	2
(vi) Spiral supports	
(vii) Fenske helices	2 litre

C Glassware for sample distillation/evaporation	No./quantity required
(i) Splash heads, sloping, flask cone 24/29, condenser cone 19/26	4
(ii) Receiver adapters, straight, 24/29 socket	4
(iii) Receiver adapters, vertical with vacuum connection	4

D Storage of pesticide stock solutions	No./quantity required
(i) Hypo vials, capacity 125 ml	144 (pack size = 72)
(ii) Caps and liners	288 of each
(iii) Crimper and decapitator	1 of each

E Miscellaneous glassware	No./quantity required
(i) Desiccator, vacuum, 0 – ring seal, 200 mm with disc	5
(ii) Crystallizing basins:	
121 × 65 mm	6
150 × 75 mm	6
(iii) Basins, porcelain:	
105 mm	3
148 mm	4

		No./quantity required
(iv)	Watch glasses,	
	90 mm	10
	150 mm	5
(v)	Test tubes, glass stoppered graduated 0-25 ml	25
(vi)	Aspirator with stopcock, 10 l	3
(vii)	Adapter, cone/rubber tubing, B24 cone	3
(viii)	Weighing funnels:	
	85 mm	5
	100 mm	5
(ix)	Glass jars, plastic screw cap, wide neck	
	100 ml	75
	250 ml	125
	500 ml	125
(x)	Glass rods, 7 mm	3 m
(xi)	Glass tubing, normal wall, borosilicate glass	
	7 mm	5 m
	9 mm	5 m

F Glassware for dithiocarbamate/head space analysis	No./quantity required
(i) Glass bottles, Pyrex with plastic screw caps capable of being drilled to make a 3 mm hole	
250 ml	6
500 ml	6
Septa to fit screw caps	50
(ii) Round-bottomed flasks, 500 ml, three neck, centre socket B24/29 and side sockets B 19/26	5
(iii) Dropping funnels with B 19/26 cone	5
(iv) Air leak tubes	6
(v) Condenser, Liebig, 400 mm B24 socket and cone,	3
(vi) Gas washbottles, Sintaglass (porosity 1), Drechsel, 125 ml capacity. Inlet/outlet tubes to be fitted with female/male spherical joints (size S13)	5
Spare heads	6 (to be altered by glassblower to fit tubes listed below, 3 for each)
(vii) Glass tubes, quickfit, B24/29 socket	
150 mm	6
200 mm	6
(viii) Glass cone, B24/29 to male spherical joint, joint size S13 (see (vi) above)	3
(ix) Joint clips for holding spherical joints	6
(x) Gas-tight syringes, Luer lock fitting	
10 ml	2
200 μl	2
50 μl	2
Disposable needles, length 50 mm, gauge 19	10

G Miscellaneous items	No./quantity required
(i) Retort stand bases 250×160 mm	15
Retort stand bases 160×100 mm	15
Retort stand rods 600×10 mm	15
Retort stand rods 1000×12 mm	15
(ii) Boss heads	40
Boss heads, single jaw	15
Clamps	40

		No./quantity required
(iii)	Rings, retort stand 70×125 mm	20
	Rings, retort stand 100×100 mm	20
	Rings, retort stand 130× 90 mm	15
(iv)	Scaffolding rods, aluminium alloy 1800 mm×13 mm	15
(v)	Spatulas, mixed sizes	15
	Palette knives, assorted sizes	7
	Scoops, 25 ml	5
	50 ml	5
	Forceps, blunt ended	4
	Forceps, pointed ends	4
	Scissors	4
(vi)	Tongs, beaker	2
	Tongs, crucible	2
	Tongs, furnace	1
(vii)	Tubing, vacuum:	
	6 mm i.d.	15 metres
	7 mm i.d.	15 metres
	Tubing, PVC, colourless:	
	6.5 mm i.d.	10 metres
	8 mm i.d.	10 metres
(viii)	Pipette filler bulbs	8
	Blow balls	8
	Rubber teats, assorted sizes (1.5, 2.0 and 2.5 ml)	30 of each size
(ix)	Test tube rack:	
	6 place, 2.5-3.0 cm tube diameter	4
	12 place, 2.5-3.0 cm tube diameter	4
(x)	Weighing boats, disposable:	
	100 ml capacity	2,000
	250 ml capacity	2,000
(xi)	Aluminium foil:	
	1 metre width	100 metres
	0.5 metre width	100 metres
	Cotton wool, absorbent	2.5 kg
	Glass wool	1 kg
	Tissues, boxes of 200	30
	Polythene bags with closures,	
	10×15 cm	250
	20×25 cm	500
	1000×500 cm	150
(xii)	Gloves, heat resistant	1
	Gloves, acid resistant	1
	Gloves, disposable	5 boxes
	Safety spectacles	6
	Goggles	2
	Ear protectors	2
(xiii)	Burner, butane gas	1
	Spare gas cartridges	2
(xiv)	Brushes, soft hair, 15 mm	3
	Brushes, beaker	
	Brushes, bottle assorted sizes	3 of each size
	Brushes, burette	
(xv)	Bowls, washing up, polythene	4
	Buckets, polythene	5
(xvi)	Plastic containers with lids, capacity approximately 350 ml	50

		No./quantity required
(xvii)	Tubing clips,	
	10-14 mm	35
	13-17 mm	35
(xviii)	Cork rings,	
	75×45 cm	30
	115×85 cm	60
	150×120 cm	20
	240×200 cm	5
(xix)	Safety screen, triple panelled, flexible, on feet	3
(xx)	Flammable solvent store, 710×915×483 mm	3
(xxi)	Acid store, 710×915×483 mm	1

Option – If water-generated vacuum is selected there will be a requirement for three metal-bodied water jet filter pumps giving a vacuum of approximately 20 mbar. *Note* that this system will only operate with an adequate water pressure.

Recurrent charges

The rate of consumption of glassware, even with due care, can be quite high and provision should be made for annual replacement at a level of about 15% of the cost of the initial stock. Similarly, a number of the items detailed in section F are consumables and will need replacement at a rate dependent upon use and work patterns.

Reagents and consumables

Filter papers and extraction thimbles		No./quantity required
(i) Filter papers,		
Whatman number 1*	7 cm	2 boxes
	15 cm	3 boxes
	24 cm	2 boxes
Whatman number 4*	7 cm	2 boxes
	15 cm	2 boxes
	24 cm	1 boxes
Whatman number 41*	7 cm	2 boxes
	15 cm	3 boxes
	24 cm	2 boxes

*or equivalent

(ii)	Extraction thimbles, cellulose, single thickness – Whatman or equivalent	
	33×100 mm	50 boxes
	41×123 mm	50 boxes

General reagents		No./quantity required
(i)	Sodium sulphate, granular, anhydrous, AR	40 kg
	Sodium sulphate powder, anhydrous, AR	20 kg
(ii)	Sodium hydroxide, AR	1 kg
(iii)	Sodium chloride, AR	2 kg
(iv)	Sodium dichromate, AR	1 kg
(v)	Anti-bumping granules	500 g
(vi)	Self-indicating silica-gel	2 kg
(vii)	Hydrochloric acid, AR	2.5 litres
(viii)	Sulphuric acid, AR	2.5 litres

Adsorbents	No./quantity required
(i) Florisil, PR Grade, 60-80 mesh	25 kg
(ii) Alumina, neutral, activity grade 1	3 kg
(iii) Silicic acid, 100 mesh	400 g
(iv) Bio-beads, S-X3, 200-400 mesh	200 g

Solvents	No./quantity required
All solvents must be glass distilled or pesticide residue grade	
(i) Acetone	300 litres
(ii) Acetonitrile	80 litres
(iii) Chloroform	30 litres
(iv) Cyclohexane	30 litres
(v) Dichloromethane	30 litres
(vi) Diethyl ether	75 litres
(vii) Ethyl acetate	25 litres
(viii) Ethyl alcohol,absolute	10 litres
(ix) Hexane	400 litres
(x) Methyl alcohol	20 litres
(xi) Petroleum ether, 40°-60°	50 litres

Special reagents

Reagents specified here are additional to those listed above which are required for basic operations. The list is not meant to be comprehensive but to cover some of the main analyses for which reagents other than basic reagents are required. It is hoped that by offering the information in this way it can provide a guide to requirements on an optional basis without incurring unnecessary costs.

	No./quantity required
(i) Dithiocarbamate analysis by CS_2 evolution and determination spectrophotometrically:	
Carbon disulphide, >99.9% pure	100 ml
Cupric acetate monohydrate, AR	250 g
Diethanolamine	250 g
Stannous chloride, AR	250 g
(ii) Dithiocarbomate analysis by headspace analysis following CS_2 evolution:	
Carbon disulphide, >99.9% pure	100 ml
Stannous chloride, AR	250 g
Thiophen, >99.9% pure	100 ml
(iii) Carbamate residues by GLC:	
1 – fluoro-2, 4-dinitrobenzene	25 ml
Disodium hydrogen phosphate	500 g
(iv) Inorganic bromide residues by GLC:	
Ethylene oxide	100 ml
Di-iso-propyl ether	2.5 litres
Ammonium sulphate	100 g

Rate of consumption

The rate of consumption of materials will obviously depend upon the nature of the analysis performed and also on whether any in-laboratory staff training is being conducted. The quantities of materials recommended above should be sufficient for some 2,000-2,200 general analyses, although long runs on particular methods will cause an imbalance. The drain on some of the general reagents such as the acids will be slight in most cases.

APPENDIX C REFERENCES

Listed below in sections 1 and 2 are the major references to analytical procedures for formulation and residue analysis. The procedures detailed in them have been fully evaluated and are accepted as 'referee' methods in cases of dispute.

Section 3 complements the above with a list of periodicals which it is advisable for laboratories to arrange to see through an appropriate library facility where possible. These periodicals update regularly on analytical techniques and procedures. A separate listing of individual papers from periodicals has not been attempted although such reviews are presented regularly in *Analytical Chemistry* and are available from that source.

Section 4 provides references to more general publications concerning, for example, laboratory practices, quality assurance, sampling programmes and interlaboratory study methods.

Finally, sections 5-7 contain references to instrumental techniques and other analytical procedures of use with in-laboratory training programmes for junior staff, safety and some general reading matter.

As many of the books listed are regularly revised and appear in different editions, no attempt has been made to cite specific editions and publication dates.

1 Methodology and procedures in pesticide formulation analysis

(i) *CIPAC Handbook*, Volumes 1, 1A, 1B, 1C, 1D and addenda as published. Collaborative International Pesticides Analytical Council Limited. Printed by Heffer and Sons, Cambridge, United Kingdom

(ii) *US Environmental Protection Agency Manual of Chemical Methods for Pesticides and Residues.* Published and distributed by The Association of Official Analytical Chemists, Arlington, United States.

(iii) *Specifications for Pesticides Used in Public Health.* Published by the World Health Organization, Geneva, Switzerland.
ISBN 92 4154140 7

2 Methodology and procedures for pesticide residue analysis

(i) *Food and Drug Administration Pesticide Analytical Manual.* US Department of Health and Human Services, Washington, DC, United States.
Reproduced by the National Technical Information Service.

(ii) *Analytical Methods for Pesticide Residues in Foods.*
Health and Welfare, Canada. Health Protection Branch, Ottawa, Canada.
ISBN 0–660–12213–8

(iii) *Analytical Methods for Residues of Pesticides.*
Ministry of Welfare, Health and Cultural Affairs, Netherlands.
ISBN 90–12–04672–6

(iv) *Analysis of Pesticide Residues in Human and Environmental Samples.* US Environmental Protection Agency, Environmental Toxicology Division, North Carolina, United States.

(v) *Official Methods of Analysis*. Association of Official Analytical Chemists, Arlington, United States.

(vi) *Recommendations for Methods of Analysis of Pesticide Residues*. Codex Alimentarius Commission, FAO/WHO, Rome, Italy.

3 Library reference material

(i) *Journal of the Association of Official Analytical Chemists*

(ii) *The Analyst*

(iii) *Analytical Chemistry*

(iv) *Journal of Agriculture and Food Chemistry*

(v) *Journal of Chromatography and Chromatographic Abstracts*

(vi) *Journal of Chromatographic Science*

(vii) *Pesticide Abstracts*

(viii) *Pesticide Science*

4 Laboratory practices and procedures

(i) *Guidelines for Sampling and Transporting Samples for Pesticide Residue Analysis*.
Federal Interdepartmental Committee on Pesticides Check Sample Programme, Federal Interdepartmental Pesticide Committee and the Expert Committee on Pesticide Use in Agriculture, Ontario, Canada.

(ii) Samples used for interlaboratory studies of methods for pesticide residue analysis in foodstuffs.
SMART, N. A. (1985) *Residue Reviews*, **96**, Springer Verlag, New York Inc., United States.
This article contains many further useful references.

(iii) Examination of performance in collaborative studies of recommended methods for pesticides residues analysis. SMART, N. A. (1984) *Analyst*, **109**, 781–6.

(iv) *Codex Guidelines on Good Practice in Pesticide Residue Analysis*. Codex Alimentarius Commission, FAO/WHO, Rome, Italy.

(v) *Recommended Method of Sampling for the Determination of Pesticide Residues*. Codex Alimentarius Commission, FAO/WHO, Rome, Italy.

(vi) Quality assurance in the pesticide product laboratory. HILL, D. F. (1985) *Journal of the Association of Official Analytical Chemists*, **68**, 921–4.

(vii) A statistician's approach to repeatability and reproducibility. HAMAKER, H. C. (1986) *Journal of the Association of Official Analytical Chemists*, **69**, 417–28.

(viii) Recommendations for the conduct and interpretation of co-operative trials, Analytical Methods Committee, Royal Society of Chemistry. *Analyst*, 1987, **112**, 679–86.

(ix) Recommendations for the definition, estimation and use of the detection limit, Analytical Methods Committee, Royal Society of Chemistry. *Analyst*, 1987, **112**, 199–204.

5 Instrumental techniques and other procedures

(i) *Handbook of Chemistry and Physics.* CRC Press, Inc. Florida, United States. ISBN 0–8493–0462–8

(ii) *Textbook of Practical Organic Chemistry.* VOGEL, A. I., Longmans, Green and Co., United Kingdom.

(iii) *Textbook of Quantitative Inorganic Chemistry.* VOGEL, A. I., Longmans, Green and Co., United Kingdom.

(iv) *Pesticide Analysis.* DAS, K. G. Marcel Dekker, Inc., New York and Basel. ISBN 0–8247–1087–8

(v) *Quantitative Analysis using Chromatographic Techniques.* KATZ, E., John Wiley and Sons. ISBN 0–471–91406–1

(vi) *Practice of Thin-Layer Chromatography.* TOUCHSTONE, J. C. and DOBBINS, M. F., John Wiley and Sons. ISBN 0–471–09766–7

(vii) *Thin-Layer Chromatography.* STAHL, E., Springer Verlag, Berlin, Germany.

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